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UNIT I

INTRODUCTION TO MATERIALS TESTING

SYLLABUS

Overview of materials, Classification of material testing, Purpose of testing, Selection of material, Development of testing, Testing organizations and its committee, Testing standards, Result Analysis, Advantages of testing.

1.1. OVERVIEW OF MATERIALS

❖ A material is defined as a substance (most often a solid, but other condensed phases can be included) that is intended to be used for certain applications. There are infinite number of materials around us - they can be found in anything from buildings to spacecraft.

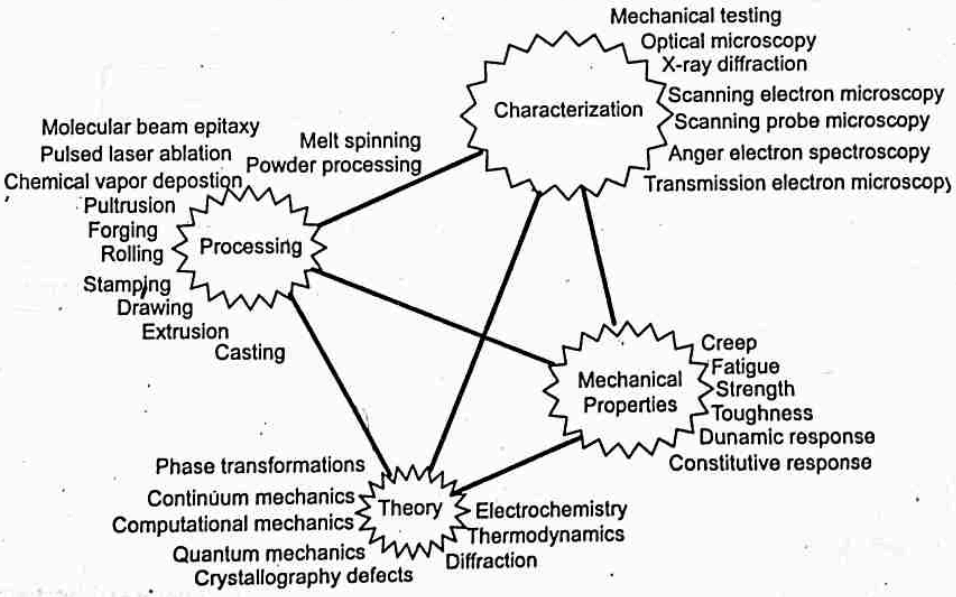


Fig. 1.1. Role of material testing

- Materials can generally be further divided into two classes: crystalline and non-crystalline. The traditional examples of materials are metals, semiconductors, ceramics and polymers. New and advanced materials that are being developed include nanomaterial, biomaterials and energy materials to name a few.
- The basis of materials science involves studying the structure of materials, and relating them to their properties.

1. CLASSIFICATION OF MATERIALS

Solid materials have been conveniently grouped into three basic categories,

- Metals
- Ceramics
- Polymers

This scheme is based primarily on chemical makeup and atomic structure, and most materials fall into one distinct grouping or another.

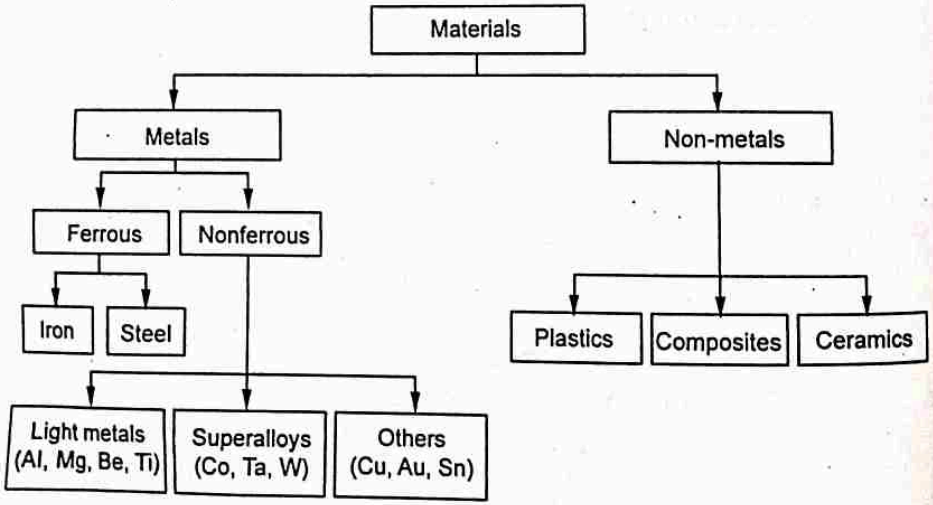
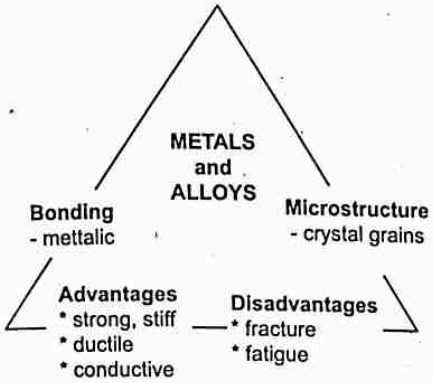


Fig. 1.2. Classification of materials

(i) Metals

- **Metals** are opaque, lustrous elements that are good conductors of heat and electricity. Most metals are malleable and ductile and are, in general, denser than the other elemental substances.

- Metals major classifications are metallic elements (e.g., iron, aluminum, copper, titanium, gold, and nickel), and often also Non-metallic elements (e.g., carbon and oxygen) in relatively small amounts.



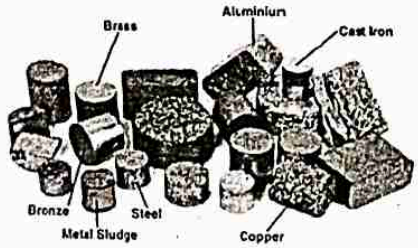


Fig. 1.3. Metal

(ii) Ceramics

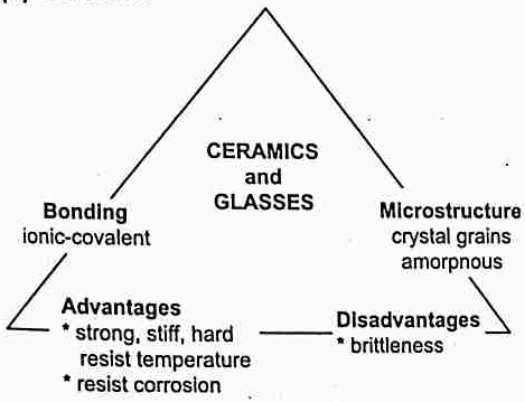


Fig. 1.4. Ceramics

- A **ceramic** is an inorganic non-metallic solid made up of either metal or non-metal compounds that have been shaped and then hardened by heating to high temperatures.
- It is compounds between metallic and nonmetallic elements; they are most frequently oxides, nitrides, and carbides.

(iii) Polymers

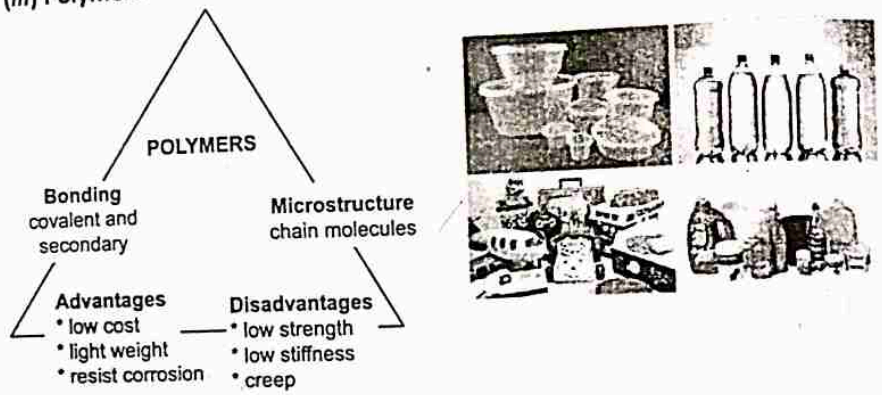


Fig. 1.5. Polymers

- A **polymer** is a large molecule, or macromolecule, composed of many repeated subunits. Due to their broad range of properties, both synthetic and natural polymers play essential and ubiquitous roles in everyday life.
- Many of them are organic compounds that are chemically based on carbon, hydrogen, and other non-metallic elements (i.e., O, N, and Si).

(iv) Composites

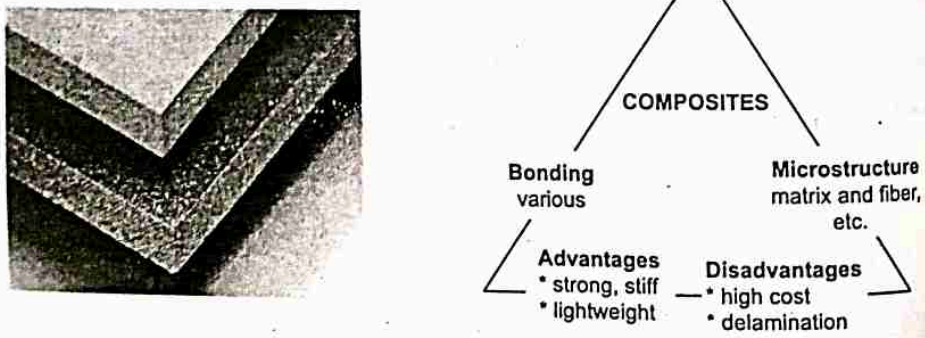


Fig. 1.6. Composites

- A **composites** is a material made from two or more constituent materials with significantly different physical or chemical properties that, when combined, produce a material with characteristics different from the individual components.
- A composite is composed of two (or more) individual materials, which come from the categories previously discussed metals, ceramics, and polymers.

(v) Advanced Materials

- Materials that are utilized in high-technology (or high-tech) applications are some- times termed advanced materials.; examples include electronic equipment (camcorders, CD/DVD players, etc.), computers, fiber-optic systems, spacecraft, aircraft, and military rocketry ,materials that are used for lasers, integrated circuits, magnetic information storage, liquid crystal displays (LCDs), and fiber optics.
- Some of the advanced materials are
  - Semiconductors
  - Biomaterials
  - Smart Materials
  - Nanomaterial

Table 1.1. Comparison between various materials

Property	Metals	Ceramics	Polymers
Bonding	Metallic (Free - Electron Cloud)	Ionic Or Covalent	Covalent
Compressive Strength (Mpa)	100 –1,500	1,000 –5,000	-
Corrosion Resistance	Low To Medium	Superior	Medium
Density (g/cm^3)	From 2 to 20	From 1 to 14	From 1 to 2.5
Ductility or Strain-to Fracture (%)	4 – 40	<1	2 - 4
Electrical Conductivity	High	Low	Low

Property	Metals	Ceramics	Polymers
Fracture Toughness ($MNm^{-3/2}$)	10 – 30	1-10	2-8
Maximum Service Temperature(°C)	1,000	1,800	250
Structure	Mostly Crystalline (Face-Centered Cubic (FCC), Body-Centered Cubic(BCC),Hexagonal Closed Packed(HCP)	Complex Crystalline Structure	Amorphous Or Semi crystalline Polymer
Tensile Strength (Mpa)	100 –1,500	100 – 400	-
Thermal Conductivity	High	Low	Low

2. Bonding in Solids

- The chemical bonds that hold atoms and molecules together in solids. There are two types bonds
  1. Primary bond
  2. Secondary bond

(*a*) Primary bond

- Primary bonds are strong and stiff and do not easily melt with increasing temperature. They are responsible for the bonding of metals and ceramics, and they provide the relatively high elastic modulus (E) in these materials.
- Three types of bonds ionic, covalent, and metallic are collectively termed primary bonds.

(*i*) Ionic bonds

- Ionic bonds occur as a result of strong electrostatic Coulomb attractive forces between positively and negatively charged ions.

(*ii*) Covalent Bonds

- Covalent bonds are often found between atoms with nearly complete outer shells. The atoms typically achieve a more stable electronic structure (lower energy state) by sharing electrons in outer shells to form structures with completely filled outer shells .

(*iii*) Metallic Bonds

- Metallic bonds are the third type of primary bond. The theory behind metallic bonding is often described as the Drude–Lorenz theory (gives relation between thermal conductivity and electrical conductivity).
- Metallic bonds can be understood as the overall effect of multiple electrostatic attractions between positively charged metallic ions.

(*b*) Secondary Bond

- Secondary or physical forces and energies are also found in many solid materials; they are weaker than the primary ones but it influence the physical properties of some materials.
- Van der Waals and hydrogen bonds, which are relatively weak, are called secondary bonds.

(*i*) Van der Waals force

- Small electrostatic attractions may develop between the atoms with slightly higher electron densities and the atoms with slightly lower electron densities. The slight deviation in the electrostatic charges on the atoms are often referred to as temporary dipole attractions or Van der Waals' forces.

(*ii*) Hydrogen Bonds

- Hydrogen bonds are induced as a result of permanent dipole forces.
- Due to the high electronegativity (power to attract electrons) of the oxygen atom, the shared electrons in the water (H_2O) molecule are more strongly attracted to the oxygen atom than to the hydrogen atoms.

3 . Structure in Crystalline Materials

- Metals and ceramics are composed of aggregations of small grains, each of which is an individual crystal. In contrast, glasses have an amorphous or non-crystalline structure.

- For example: Polymers are composed of chainlike molecules, which are sometimes arranged in regular arrays in a crystalline manner.

Basic Crystal Structures

- The arrangement of atoms (or ions) in crystals can be described in terms of the smallest grouping that can be considered to be a building block for a perfect crystal. Such a grouping, called a unit cell, can be classified according to the lengths and angles involved.
- For example: In three-dimensional space with seven major crystallographic unit cells may be better classified into 14 Bravais lattices.

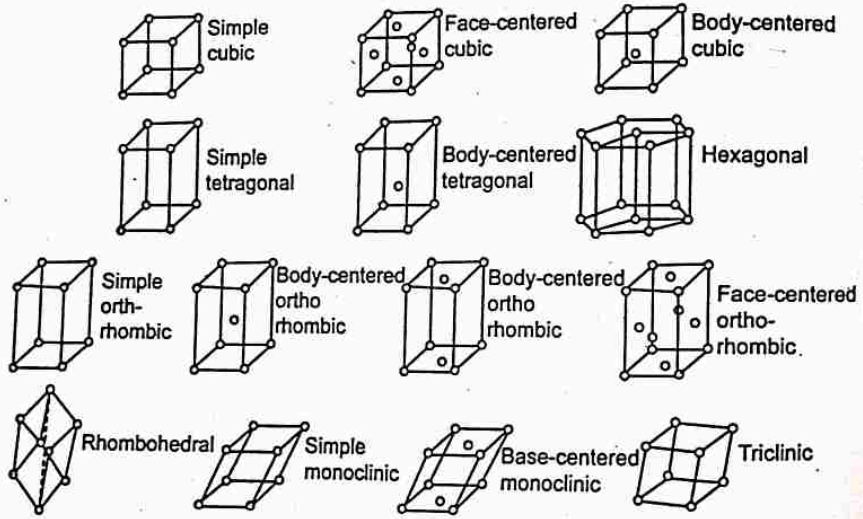


Fig. 1.7. Bravais lattices

4. DEFECTS IN CRYSTALS

- Ceramics and metals in the form used for engineering applications are composed of crystalline grains that are separated by grain boundaries.
- Even within grains, the crystals are not perfect; with defects occurring that can be classed as point defects and line defects or surface defects.
- Line defects are also called as dislocations where the edges of surfaces is relatively displaced lattice planes.
- Line defects is further classified into edge dislocation and screw dislocation.

- Grain boundaries can also be a class of surface defect where the lattice planes change orientation by a large angle.
- In point defects, where an atom is missing (or) is an irregular place in lattice structure.

1.2. MATERIAL TESTING

- Materials testing are established technique which is used to ascertain both the physical and mechanical properties of raw materials and components.
- It also measures the characteristics and behaviour of substances such as metals, ceramics, or plastics under various conditions. Testing of materials is classified for following purposes.
  - Developing standard methods and procedure for testing.
  - Test to establish properties of materials.
  - Test to establish the integrity of the material or component.

CLASSIFICATION OF MATERIAL TESTING

- Test may be classified based on loading condition-static, dynamic or long time tests, based on temperature-normal or room temperature and elevated temperature, based on special requirement on instrument- holdings or gripping, power regulation, bedding the specimen, accuracy , precision, special atmosphere like salt spray, corrosive etc.
- Materials testing classified into three major categories
  - Mechanical testing (or) Destructive testing (DT)
  - Nondestructive testing
  - Material characterization testing

1. DESTRUCTIVE TESTING

- Destructive testing (DT) is a form of object analysis that involves applying a test to break down a particular material to determine its physical properties, such as the mechanical properties of strength, toughness, flexibility, and hardness.
- It is undertaken in order to understand a specimen's performance or material behavior, procedures is carried out to the test specimen's failure.

(a) CLASSIFICATION OF DESTRUCTIVE TESTING

1. Static Testing
2. Impact Testing
3. Cyclic Testing

1. Static Testing

- Where the load is applied gradually in static testing. Different material testing methods available under static testing. They are listed below.
  - Tension Test
  - Compression Test
  - Shear Test(Torsion test)
  - Hardness Test
  - Creep Test
  - Bending test

2. Impact (Dynamic) Testing

- When the given specimen is subjected to shock loads then it is known as the impact load testing or the dynamic load testing.
  - Charpy Test
  - Izod Test
  - Drop Ball
  - Drop Dart
  - Instrumented Puncture Testing

3. Cyclic Testing

- When the load is repeatedly varied in magnitude and the direction then this test is called Impact or dynamic testing.
  - Fatigue test

(b) ADVANTAGES

- Allows a roughly identify the mechanical properties (fracture strength, elongation, modulus of elasticity etc.
- The properties of the bonding can be defined according to the different types of stresses such as tension, compression, shear, peel, dynamic forces of impact.

- The costs of equipment for destructive testing are cheaper compare with the certain equipment used in non - destructive testing.
- Verification of surface preparation, curing conditions and working conditions is minimum.
- Predict and identify the approximate nature of the failure or breakdown that may occur during the lifetime.
- Tests on a relatively cheaper cost.

(c) DISADVANTAGES

- You cannot identify internal defectology (bubbles, delaminating, pores, wrong thickness etc.)
- Need to make large number of specimens simulating the same process (surface preparation, environmental conditions) which cannot be reused once have been tested again.
- Not directly identifies the status of the failure area.

2. NON-DESTRUCTIVE TESTING (NDT)

- Nondestructive Testing (NDT) consists of a variety of non-invasive inspection techniques used to evaluate material properties, components, or entire process units. The techniques can also be utilized to detect, characterize, or measure the presence of damage mechanisms (e.g. corrosion or cracks).
- Many types of NDT techniques are capable of locating defects and determining the features of the defects such as size, shape, and orientation.
- The purpose of NDT is to inspect a component in a safe, reliable, and cost effective manner without causing damage to the equipment or shutting down plant operations. This is in contrast to destructive testing where the part being tested is damaged or destroyed during the inspection process.

Major source of NDT test

- Liquids
- Radiation
- Sound

- Magnetism
- Infrared

(a) CLASSIFICATION OF DESTRUCTIVE TESTING

- Acoustic Emission Testing (AET)
- Infrared Testing (IR)
- Leak Testing (LT)
- Liquid Penetrant Testing (PT)
- Electromagnetic Testing (ET)
- Magnetic Particle Testing (MPT)
- Radiographic Testing (RT)
- Film Radiography (FR)
- Ultrasonic Testing (UT)
- Visual Inspection (VI)

Advanced NDT Techniques

- Advanced methods tend to be understood as emerging technologies, e.g uncertain advantages or limitations, lack of technician qualification criteria, or little to no industry codification.
  - Electromagnetic Testing (ET)
    - Eddy Current Testing (ECT)
    - Magnetic Flux Leakage (MFL)
  - Laser Testing Methods (LM)
    - Holographic Testing
  - Radiographic Testing (RT)
    - Computed Radiography (CR)
    - Computed Tomography (CT)
    - Digital Radiography (DR)
  - Ultrasonic Testing (UT)

- Angle Beam
- Long Range Ultrasonic Testing (LRUT)
- Phased Array Ultrasonic Testing (PAUT)

(b) ADVANTAGES OF NDT TEST

- Most of the equipment in NDT test is portable.
- The obtaining of result is quick and reliable.
- The separate sample preparation is not requires and sample is can be reused.
- To determine the properties of the raw material.
- To check quality at intermediate stages of production processes.
- To ensuring that your infrastructure and vital equipment will provide continued production, undergo minimal degradation and are designed with optimal performance in mind.
- Many properties are found in single specimen.
- Less Waste of specimens.
- Accident Prevention.
- Identify areas of concern before failure.

(c) DISADVANTAGES OF NDT TEST

- Result interpretation is difficult.
- Skilled labor is required.
- Components needing to be cleaned before and after inspection.
- Sensitivity of inspection can sometimes be affected by the finish of a component.
- Sometimes there might be a lack of depth sizing.
- On some non-destructive test methods, only relatively non-porous surfaces can be inspected.
- Some test methods require electricity , affected by variations in magnetic permeability , only effective on materials that are conductive.

COMPARISON BETWEEN DESTRUCTIVE TEST & NON-DESTRUCTIVE TEST

Table 1.2. Destructive Test Vs Non-Destructive Test

DESTRUCTIVE TEST	NON-DESTRUCTIVE TEST
❖ Tests are usually quantitative measurements of load for failure, significant distortion or damage, or life to failure under given loading and environmental conditions.	❖ Tests are usually qualitative and rarely quantitative. They do not usually measure load for failure or life to failure even indirectly.
❖ The correlation of result is directly given by observer.	❖ Skilled judgment and test or service experience are usually required to interpret test indications.
❖ Destructive tests are not usually to apply on parts in working condition.	❖ Non-destructive tests may often be applied to parts in working assemblies without interruption or service beyond normal maintenance or idle periods.
❖ Cumulative change over a period of time cannot readily be measured on a single specimen. ❖ Eg. Concrete cube is tested for 7days, 14days and 28 days.	❖ Non-destructive tests permit repeated checks of a given same specimen over a period of time.
❖ The sample of high cost material or fabrication, the cost of replacing the parts destroyed may be difficult.	❖ Acceptable sample of very high material or fabrication costs are not lost in non-destructive testing.
❖ Tests can be made on only a fraction of the production lot to be used in building.	❖ Tests can be made on every parts to be used in building if economically justified.
❖ A single destructive test may measure only one or a few of the properties that may be critical under service conditions.	❖ Many non-destructive tests, each sensitive to different properties or regions of the material or part, may be applied simultaneously or in sequence.
❖ Tests usually simulate durability conditions. Consequently, they tend to measure serviceability directly and reliably.	❖ Tests usually involve indirect measurements of properties of no direct significance in service.

3. MATERIAL CHARACTERIZATION TESTING

- ❖ Characterization and analytical techniques are methods used to identify, isolate or quantify chemicals or materials, or to characterize their physical properties.
- ❖ Characterization of samples used for external techniques to analysis into the sample's elemental composition, internal structure and thermal, electrical, optical, magnetic properties etc.

TYPES OF MATERIAL CHARACTERIZATION TEST

Table 1.3. Types of characterization test

Major classification	Sub catagory
Microscopy	❖ Optical Microscope ❖ Scanning Electron Microscope (SEM) ❖ Transmission Electron Microscope (TEM)
Spectroscopy	❖ Ultraviolet-visible spectroscopy (UV-vis) ❖ Secondary ion mass spectrometry (SIMS) ❖ Nuclear magnetic resonance spectroscopy (NMR)
Macroscopic testing	❖ Destructive testing ❖ Ultraviolet-visible spectroscopy (UV-vis) ❖ Fourier transform infrared spectroscopy ❖ X-ray diffraction (XRD) ❖ Secondary ion mass spectrometry (SIMS) ❖ Nuclear magnetic resonance spectroscopy (NMR) ❖ Differential thermal analysis (DTA) ❖ Dielectric thermal analysis (DEA) ❖ Thermogravimetric analysis (TGA) ❖ Differential scanning calorimetry (DSC) ❖ Impulse excitation technique (IET)

(a) ADVANTAGES

- Less time is required for testing.
- Complex structures can be analyzed easily.
- Multiple properties is observed in single test.
- High resolution image processing techniques.

(b) DISADVANTAGES

- Only few of the equipment is portable.
- Cost of installation is high.
- Most of the test requires specimen preparation.
- Vacuum is needed for many tests.
- Sample size are prepared under some restrictions.
- Most of the testing methods are uncommon.
- Single method is unfit for testing all materials.

1.3. PURPOSE OF TESTING

- To maintain the quality and consistency of the finished product.
- To avoid mistakes in the first stage of the manufacturing process.
- To obtain compliance certification by following guidelines and regulations of testing and by obtaining standard limit of materials properties.
- To ensure that the materials are suitable for production and usage.
- To determine the reason behind product failure during manufacture or while in use.
- To prevent failure in usage.
- Used as a quality control check in the material manufacturing or processing. Destructive test is used to check the durability, specific requirement and non-destructive test used for finding defect.
- For the acceptance of material, the component performance would fulfill the requirement of testing.
- To check the components prior the final assemble.

- To check the component in service without damage and deterioration
- Used for research and development of existing and new materials.

1.4. SELECTION OF MATERIAL

- Material selection is one of the foremost functions of effective engineering design as it determines the reliability of the design in terms of industrial and economical aspects.
- Generally, it is in Iterative nature. There is a strong element of trial and error where an initial design is done and then analyzed, tested, and subjected to trial production. Changes may be made at any stage of the process to satisfy requirements not previously considered or problems just discovered.

(a) STEPS TO BE CONSIDERED FOR SELECTION OF MATERIALS

Step 1: Identify the design requirements.

- Each step involves a synthesis process in which all of the various concerns and requirements are considered together. Compromises between conflicting requirements are usually necessary and continual effort.
- The design requirements include the following items
  - Performance requirements
  - Simplicity and practicability
  - Reliability requirements
  - Size, shape, and mass requirements
  - Cost requirements
  - Manufacturing and assembly requirements
  - Industry standards
  - Sustainability requirements
- Identifying as many of the requirements as possible is critical. For many products, some of these requirements are not applicable, making the information gathering will make the process easier.

Step 2: Identify materials selection criteria

- The materials selection criteria are specific materials properties derived from the requirements identified during pervious step.

- For example, for a component that must support a specific load, the minimum yield stress that is required for the component's material can be determined. This will be one of the material selection criteria.

Step 3: Identify candidate materials

- Use the materials selection criteria to rule out materials that will not satisfy all the materials selection criteria. When evaluating whether a material might be appropriate for the application, be sure to consider the materials range of values for the properties of interest.

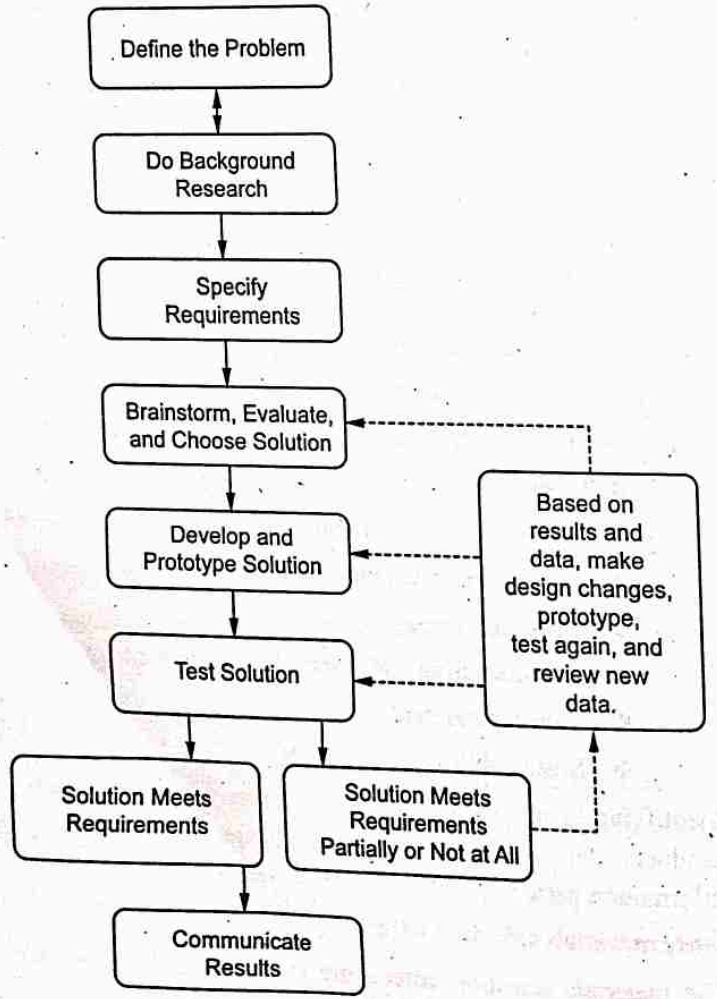


Fig. 1.8. Failure analysis

Step 4: Evaluate candidate materials

- There may be candidate materials for which there insufficient data available to indicate whether the materials satisfy certain selection criteria. These materials will have to be analyzed and tested to determine whether they do meet the selection criteria by creating a prototype.

Step 5: Select materials

- Select the materials that satisfy all the materials selection criteria at the lowest cost. Remember, cost includes the cost of the material and the cost to fabricate a component or form a joint between components.

Step 6: Failure Analysis

- The selection of materials is finalized with help of failure analysis mode which is given in Fig. 1.8.

Step 7: Service Experience

- Design changes may also be made as a result of experience with a limited production run of a new product. Purchasers of the product may also use it in a way not anticipated by the designer, resulting in failure.
- The design process often continues even after a product is established and widely distributed.

(b) FACTORS AFFECTING SELECTION OF MATERIALS

1. Performance

- This characteristic refers to those properties that are required for the product to satisfy its functional requirements.
- Materials typically perform one or more functions in a product such as carrying loads, providing heat conduction or thermal insulation, providing electrical conduction or insulation, or containing fluids.

(i) Mechanical properties

- The material must possess a certain strength and stiffness. Selected materials are examined for strength and stiffness values, and then potential materials are further inspected for other desired properties.

(ii) Wear of materials

- Wear is a problem when the materials are contacting each other in a product. So it must be ensured that the selected materials have sufficient wear resistance.

(iii) Corrosion

- It is an important engineering design criterion for designs open to the environment for a longer period of time. Some materials are very likely to be corroded in the service depending on the service environment.
- Metals like iron are heavily prone to corrosion if it not prepared to resist corrosion. Painting or any other surface coating method, cathodic protection, etc. are possible ways to minimize the effect and increase the service life.

(iv) Ability to manufacture

- Although the material is well capable of using for the design, it may be difficult to manufacture.
- If this selection criteria is neglected the manufacture process might be very costly making it unprofitable as a commercial product.

(v) Cost

- Cost is a critical fact to consider when selecting materials for a certain design for most products because they are facing a severe competition in the market.
- The cost factor can be neglected when performance is given the top priority. When estimating costs, all the associated cost factors must be considered to get a more reasonable value. It may involve the transportation, processing costs, etc.

2. Reliability and Environmental Resistance

- This characteristic relates to the durability of a material, which is its ability to resist deterioration in the environment in which it will be used. It includes such properties as fatigue resistance and resistance to radiation, chemical solvents, and corrosive agents.
- Critical characteristics that are needed to satisfy the functional requirements and their constraints.

3. Reducibility

- Material selection cannot be made independently of the selection of the manufacturing process, since the manufacturing process will affect the performance properties of the material.
- Furthermore, the selection of the manufacturing process will depend on certain properties of the materials. Material properties that can dictate the choice of a manufacturing process include ductility, toughness, formability, and castability. In addition, one must take into account the geometric attributes of the production.

1.5. DEVELOPMENT OF TESTING

- **Materials testing**, measurement of the characteristics and behavior of such substances as metals, ceramics, or plastics under various conditions by a full- or small-scale model of a proposed machine or structure may be tested.
- Alternatively, investigators may construct mathematical models that predict capabilities of the structure.
- Standard test methods have been established by such national and international bodies as the International Organization for Standardization (ISO).

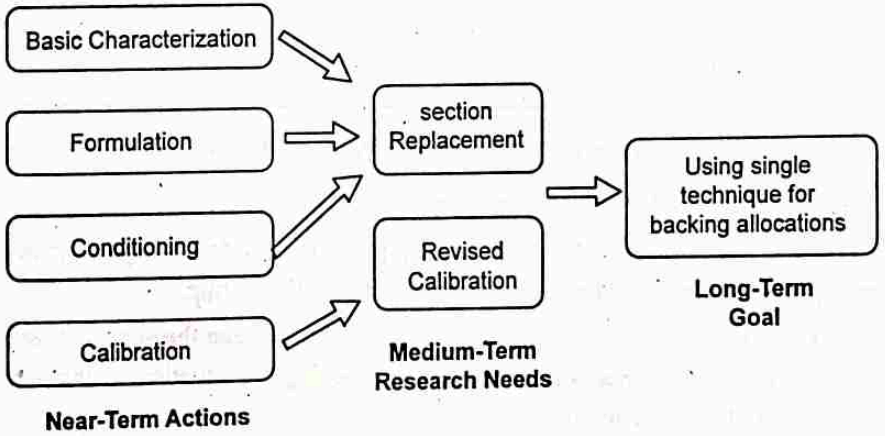


Fig. 1.9. Formulation in test development

(a) STAGES IN DEVELOPMENT OF TESTING

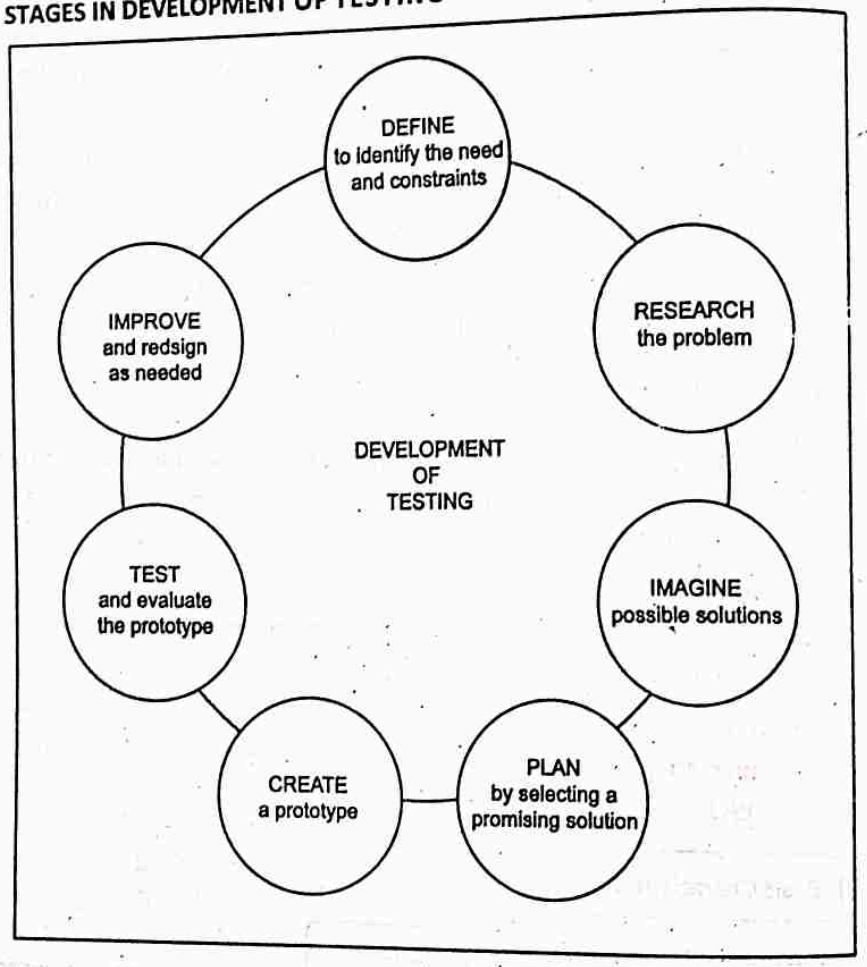


Fig. 1.10. Stages in development of testing

(a) Identify the Need & Define the Problem

The first step is to identify and define the problem. The following major problem, which needs to consider during development of material testing.

- A problem can be regarded as a difference between the actual situation and the desired situation. It involves diagnosing the situation so that the focus on the real problem.
- Development of varies time, cost, sample and labor minimizing test techniques.

- Development of a new manufacturing / processing line or making changes to an existing one, needs an improvement of testing methods under different conditions and in different applications.
- Improvement of Troubleshooting, to determine what is causing issues during processing.
- Scale-up of a testing technology.
- Increase fundamental understanding of materials.
- Improvement of the process/product performance relative to the needs and demands of customers.
- Reduction of existing process spread, which leads to poor capability.

(b) Research the Problem

- Some possible ways to identify potential process by using knowledge of the process, historical data, cause-and-effect analysis and brainstorming, etc.,
- The research of problem may concern of a condition to be improved, a difficulty to be eliminated, or a troubling question that exists in literature or testing techniques , specific issue, contradiction or gap between present and future testing techniques in that need of meaningful understanding and deliberate investigation.
- If important factors are left out during development of testing experiment, then the results of the experiment will not be accurate which must be take care.

(c) Develop possible testing methods

- The size of the testing is dependent on the number of factors or interactions to be studied, the number of levels of each factor, budget and resources allocated for carrying out the experiment, etc.
- The development testing plan methods is done using various techniques of graphical presentation, such as Auto cad, simulation techniques methods, etc.
- During the design stage, it is quite important to consider the confounding structure and resolution of the design.

- The material testing code book gives the basics of testing development standards, which is based on environment, material specification and result analysis methods etc.

(d) Evaluate the Alternatives & Select Most Promising methods

- The various possible method is developed and stimulated in softwares to ensure the theoretical acceptance.
- Pre presenting the information of testing methods are deciding criteria of effective method.
- The testing methods need to satisfy the basic criteria like cost and time.
- The combination of different testing methods is also selected for the effectiveness.

(e) Initial Design

- The initial design is often made on the basis of avoiding stresses that exceed the yield strength of the material. Then the design is checked by more refined analysis, and changes are made as necessary to avoid more subtle modes of material failure, such as fatigue, brittle fracture, and creep.
- In making design decisions that involve safety and durability, the concept of a safety factor is often used. The safety factor in stress is the ratio of the stress that causes failure to the stress expected to occur in the actual service of the component. That is,

$$X1 = \text{stress causing failure/stress in service}$$

(f) Construct a prototype

- The materials that will be used in final testing methods may be expensive or difficult to fabricate, so prototypes may be made from different materials than the final product. In some cases, the final production materials may still be undergoing development themselves.
- A prototype, or trial model, is often made and subjected to simulated service testing to demonstrate whether it is functions properly.
- Prototypes are generally made with much closer individual inspection and the assumption that some adjustment or rework will be part of the fabrication process.

- Prototypes may also be exempted from some requirements that will apply to the final product.

(g) Test and Evaluate the Prototype

- It is important to test and evaluate your prototype along the way for functionality, usefulness, and safety. The final product may be subject to a number of quality assurance tests to verify conformance with drawings or specifications.
- These tests may involve custom inspection fixtures, statistical sampling methods, and other techniques appropriate for ongoing production of a large quantity of the final product.

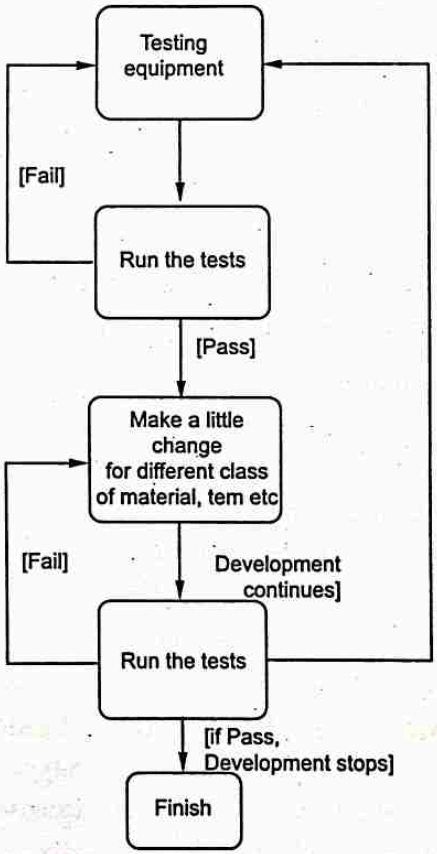


Fig. 1.11. Evaluation of prototype

- The failure of prototype leads to choosing alternatives and redesign the section.
- The early estimate of loads may have been quite uncertain. A prototype may also be subjected to simulated service testing until either a mechanical failure occurs, perhaps by fatigue, creep, wear, or corrosion, or the design is proven to be reliable. This is called durability testing.
- For very large items, it may be impractical or uneconomical to test a prototype of the entire item. A part of the item, that is, a component, may then be tested.

(h) Communicate the Design

- Communication design is a mixed discipline between design and information-development which is concerned with such as printed, crafted, electronic media or presentations to communicate with people for overcoming some unreliable problems.

(i) Redesign

- The redesign is approached existing testing techniques is outdated for the present materials and to minimizing the calibration.

Example for development of testing

- **DEVELOPMENT OF MECHANICAL TESTING**-Structures and machines, or their components, fail because of fracture or excessive deformation. In attempting to prevent such failure, the designer estimates how much stress (load per unit area) can be anticipated, and specifies materials that can withstand expected stresses.
- Test machine grips are designed to transfer load smoothly into the test piece without producing local stress concentrations.
- **DEVELOPMENT OF STATIC COMPRESSION** - Tests determine a material's response to crushing, or support-type loading (such as in the beams of a house). Testing machines and extensometers for compression tests resemble those used for tension tests.
- **DEVELOPMENT OF STATIC SHEAR AND BENDING TESTS** - In plane shear tests indicate the deformation response of a material to forces

applied tangentially. Shear strength of rivets and other fasteners also can be measured.

- **DEVELOPMENT OF MEASURES OF DUCTILITY**- Ductility is the capacity of a material to deform permanently in response to stress. Ductility can be expressed as strain, reduction in area, or toughness. Reduction in area (change in area per unit area) may be measured, for example, in the test section of a steel bar that necks when stressed.
- **DEVELOPMENT OF HARDNESS TESTING**-Based on the idea that a material's response to a load placed at one small point is related to its ability to deform permanently (yield), the hardness test is performed by pressing a hardened steel ball (Brinell test) or a steel or diamond cone (Rockwell test) into the surface of the test piece.
- **DEVELOPMENT OF IMPACT TEST**-Many materials, sensitive to the presence of flaws, cracks, and notches, fail suddenly under impact.
- **DEVELOPMENT OF FRACTURE TOUGHNESS TESTS**-The criterion for failure became sudden propagation of a crack rather than fracture. Tests have shown that cracks occur by opening, when two pieces of material part in vertical plane, one piece going up, the other down; by edge sliding, where the material splits in horizontal plane, one piece moving left, the other right; and by tearing, where the material splits with one piece moving diagonally upward to the left, the other moving diagonally downward to the right.
- **DEVELOPMENT OF CREEP TEST**-Creep is the slow change in the dimensions of a material due to prolonged stress; most common metals exhibit creep behavior. In the creep test, loads below those necessary to cause instantaneous fracture are applied to the material, and the deformation over a period of time (creep strain) under constant load is measured, usually with an extensometer or strain gauge.
- **DEVELOPMENT OF FATIGUE TEST**- Materials that survive a single application of stress frequently fail when stressed repeatedly. This phenomenon, known as fatigue, is measured by mechanical tests that involve repeated application of different stresses varying in a regular cycle from maximum to minimum value.

1.6. TESTING ORGANIZATIONS AND ITS COMMITTEE

(a) INTERNATIONAL ORGANIZATION FOR STANDARDIZATION (ISO)

- The International Organization for Standardization (ISO) is an international standard-setting body composed of representatives from various national standards organizations.
- ISO is a voluntary organization whose members are recognized authorities on standards, each one representing one country. Members meet annually at a General Assembly to discuss the strategic objectives of ISO. The organization is coordinated by a central secretariat based in Geneva.

(b) ASTM INTERNATIONAL

- ASTM International, formerly known as American Society for Testing and Materials, is an international standards organization that develops and publishes voluntary consensus technical standards for a wide range of materials, products, systems, and services.

(c) BUREAU OF INDIAN STANDARDS (BIS)

- BIS is the National Standard Body of India established under the BIS Act 2016 for the harmonious development of the activities of standardization, marking and quality certification of goods and for matters connected therewith or incidental there to.
- BIS has been providing traceability and tangibility benefits to the national economy in a number of ways
  - Providing safe reliable quality goods
  - Minimizing health hazards to consumers
  - Promoting exports and imports substitute
  - Control over proliferation of varieties etc. Through standardization, certification and testing

(i) Organization OF BIS

- The organization of BIS consists of following members,
  - Governing Council Members
  - Executive Committee
  - Administrative Structure

(ii) Organization chart

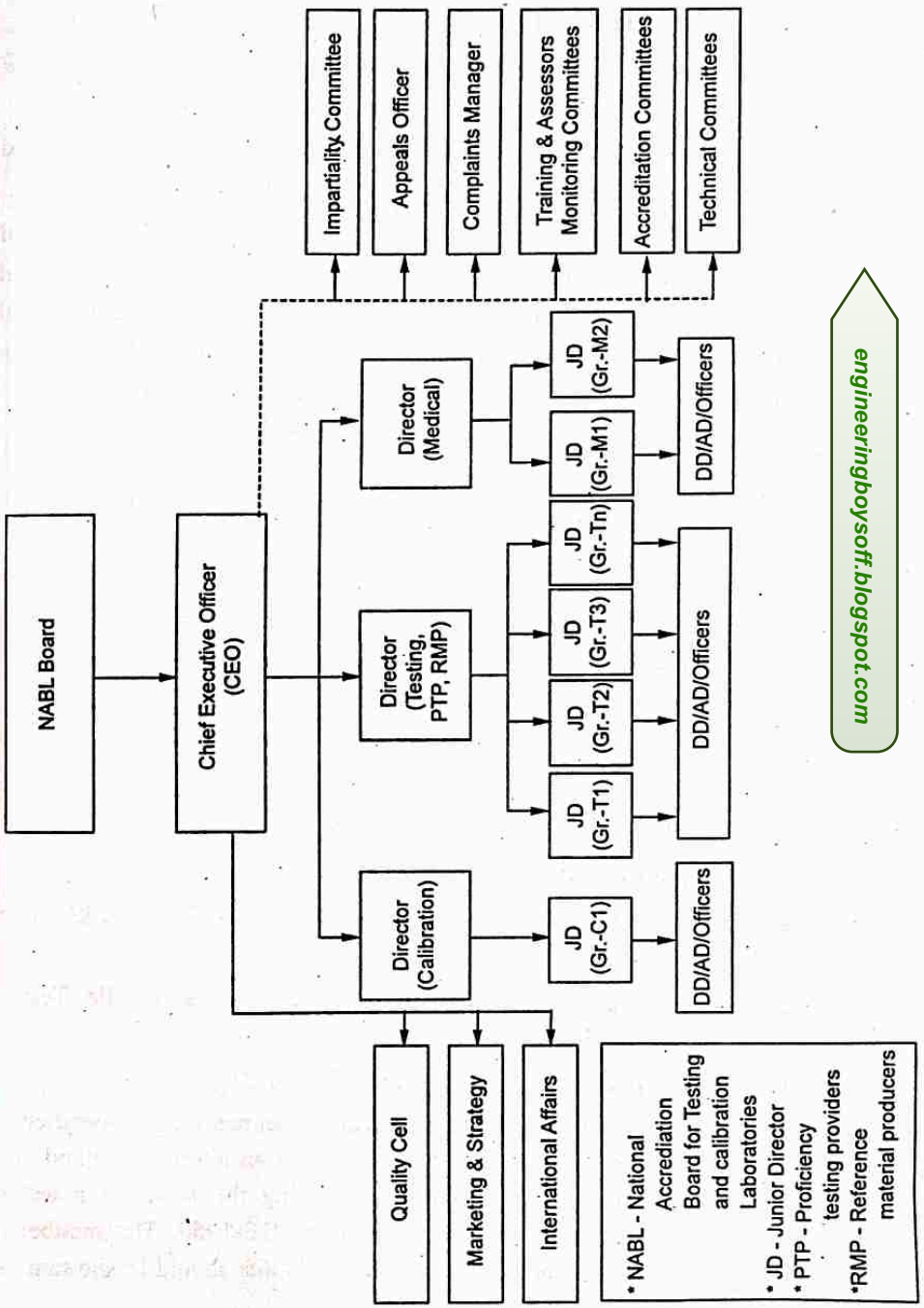


Fig. 1.12. Organization chart

(iii) OTHER TESTING ORGANIZATIONS

- The National Metrological Institutes (NMIs) namely National Physical Laboratory (NPL) and Bhabha Atomic Research Centre (BARC).
- The Standards Bodies namely Bureau of Indian Standards (BIS) and Standardization, Testing and Quality Certification (STQC).
- Council for Industrial and Scientific Research (CSIR), the other Boards of Quality Council of India (QCI), the other organizations under nodal department of QCI i.e. Department for Promotion of Industry and Internal Trade, the other Departments / organizations under nodal Ministry i.e. Ministry of Industry and Commerce are the bodies related to NABL.

(iv) Codes for TEST procedure

- Some of the testing standards codes followed in India
  1. Rockwell hardness test (IS: 1586-2000)
  2. Brinell and Vickers hardness tests (IS: 2281-2005 RA-2011)
  3. Impact tests (Charpy V-Notch and Izod tests) (IS: 1757-1988 and IS: 1598-1977 RA-2009))
  4. Tensile Test (IS: 1608-2005 RA-2011)
  5. Compression Test (IS: 1608-2005 / ISO 4506-1979)
  6. Bend test for metal products(IS:1599-1985 RA-2011)
  7. Shear Test (IS: 5242-1979 RA-2006)
  8. Beam or flexural bending test (IS: 16-1959)
  9. Torsion test and Fatigue test (IS:5074-1969 RA-2001 and IS: 5075-1985 RA-2001)
  10. Indian Standard Mechanical Testing of Metals Tensile Testing (IS:1608-1995)
  11. Metallic materials - Bend test (IS 1599 - 2012)
- For all the tests described in this section, the method as specified in relevant ISO standard may also be followed as an alternate method. The final value, observed or calculated, expressing the result of a test or analysis, is rounded off in accordance with IS:2-1960. The number of significant places retained in the rounded off value should be the same as that of the specified value in the code.

1.7. BENEFITS OF TESTING

(i) Safety issues can be identified

- The tests are carried out to ensure product safety, and also to make sure the person carrying out the work on any machinery or components is safe too. In mechanical testing, the equipment testing area is covered with glass plate to present from shattering of test piece out of equipment.
- Most non-destructive tests are harmless to humans, although tests involving radiographic must be carried out under strict settings. All tests must ensure that products are left completely undamaged.
- Its main aim, when used properly, and the results of the tests accurately acted upon is to identify and resolve problems that could otherwise be disastrous.

(ii) It provides reliability

- If workers in industry want reliable and accurate results, all material testing will offer stability.
- The testing technique are accurate way of inspection since it is repeatable and used to correlate results.
- Any given piece of equipment or machinery can undergo a range of non-destructive tests which will remove the risk of any inaccuracy of result, or oversight for long range. The testing equipment needs calibration for better result.

(iii) It is cost effective

- These types of tests can also give insights that can result in the effective replacement or repair of components or equipment before a real malfunction or breakdown occurs, which will save more money in the long term.

(iv) It offers reassurance

- Reassurance is such a simple thing, but it can sometimes be the most important advantage to testing methods.
- The operation of testing equipment being harmless and it also help to prevent injury (or) fatalities by structures, machinery (or) components etc.

- When workers know they are safe, they feel more secure and this is something that can benefit productivity and output, overall.

1.8. PRESENTATION OF RESULT

- It is very important by sharing the knowledge of result or development with others which leads to the various development of test result by other scientist or researchers.
- The steps to be followed for description of test report
  - Statement of the problems
  - Materials, methods and procedure used during testing
  - Result analysis
  - Summary, conclusion and discussion
  - Appendices to support findings

(*i*) Statement of the problems

- Statement of the problems describes the objectives of testing which intends about problem.

(*ii*) Materials, methods and procedure used during testing

- Materials, methods and procedure used during testing section includes the material to be tested, the conditions of testing specimen, important apparatus used for testing and the major procedure followed by testing which is referenced from the Indian standard code books.

(*iii*) Data presentation and Result analysis

- The result data presented by plotting it in various methods with proper units assigned or listed in clear and meaningful manner. In every method of result presentation, the statement of result is summarized with the significance of materials.
- The result analysis is done by various methods,
  - Charts
  - Graphs
  - Tabulation
  - Statement
  - Analytic Software

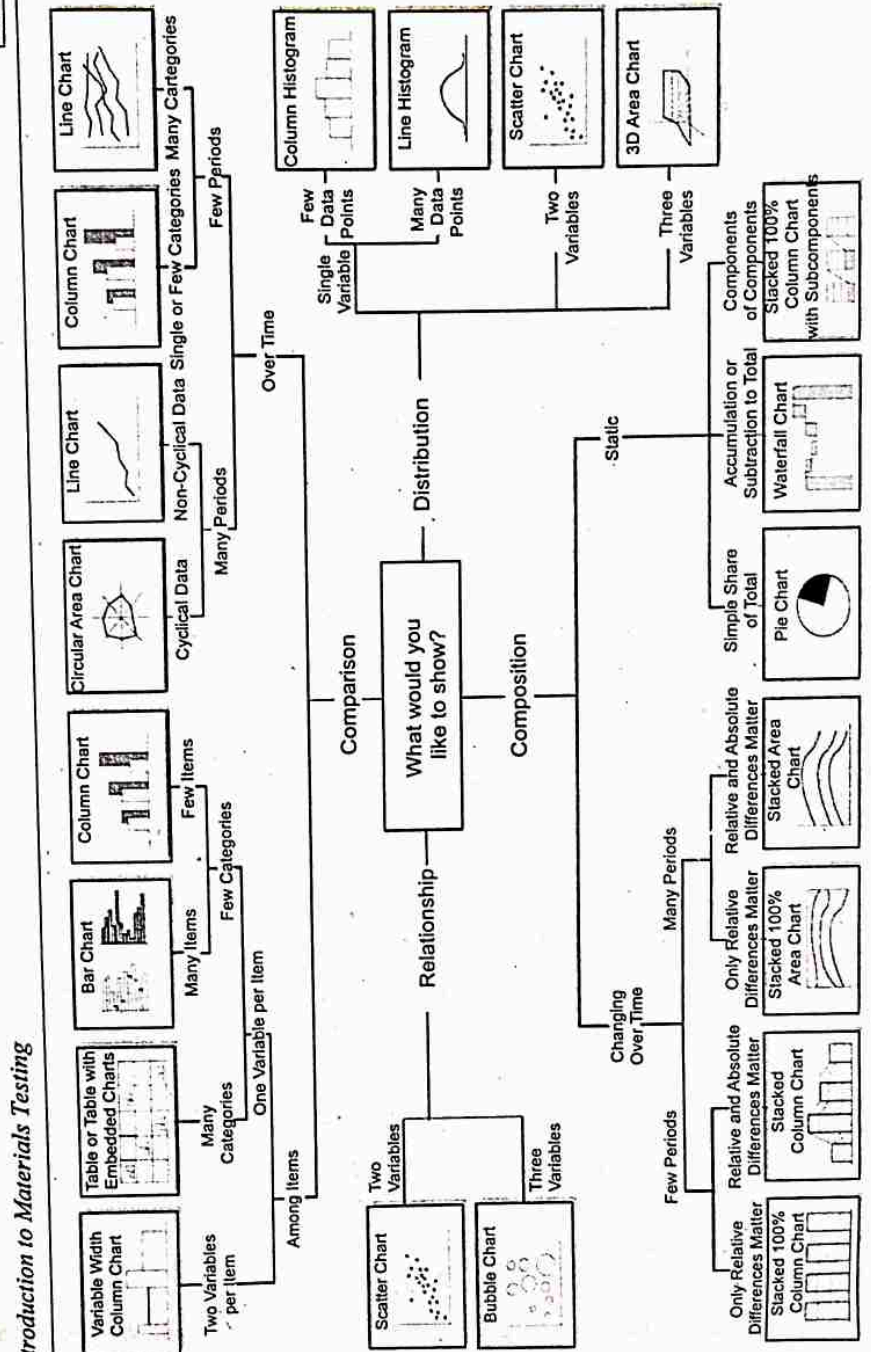


Fig. 1.13. Types of graphs and charts

(a) CHARTS & GRAPHS

- A chart displays schematic processes based on the outcome, validity or answer to a previous variable. Graphs, used to display comparisons between 2 variables. For example: line graphs involve an x-axis horizontally and a y-axis vertically on a grid

(b) TABULATION

- Tabulation is a systematic & logical presentation of numeric data in rows and columns, to facilitate comparison and statistical analysis accompanying with summarizing result.

Major Objectives of Tabulation

- To Simplify the Complex Data
- To Bring Out Essential Features of the Data
- To Facilitate Comparison
- To Facilitate Statistical Analysis
- Saving of Space

(c) STATEMENT

- Statement statistics is a form of mathematical analysis that uses quantified models, representations and synopses for a given set of experimental data or real-life studies. Statistics statement studies methodology to gather, review, analyze and draw conclusions from data.
- **Example:** The result of 28 days strength of silicon mixed cube is 25% greater than the conventional concrete.

(d) ANALYTIC SOFTWARE

- Software analysis is the analytics specific to the domain of software systems taking into account source code, static and dynamic characteristics (e.g., software metrics) as well as related processes of their development and evolution.

Example:

- ***BIOVIA* MATERIALS STUDIO-** Materials Studio allows you to easily build, modify, visualize and simulate a wide range of materials.

- **LAS X MATERIALS SCIENCE MODULES-** LAS X can be enhanced with a range of advanced modules and applications to form a powerful microscopy imaging environment.
- **MATLAB** - Computation and plotting.
- **AUTO CAD** - Designing of outline element, 2Dand 3D element.
- **STADD PRO** - Designing of structures.
- **ABACUS** - Finite element analysis.
- **ANSYS ELECTRONICS** – It is the premier solution for electromagnetic field, circuit, systems and multi physics simulation and analysis for electronic design.

(iv) SUMMARY, CONCLUSION AND DISCUSSION

- It describes about the general findings of test or experiment and summarizes the important point. Also gives the view about the various error or difficulties occurred during testing. It gives new view and opinion about material, projected view and acceptability for use in market and environment.

(v) APPENDICES TO SUPPORT FINDINGS

- It gives supporting data for testing the materials like code books, past material testing history and data for better clarity for testing.

1.9. TESTING VS INSPECTION

- Testing is the physical performance of an operation series aimed at providing quantitate data regarding the properties of a material. It provides the information about the quality of material.
- Inspection is the observance of a material to determine the presence or absence of a desired one. It aimed about the controlling the quality of materials by establishing criteria of acceptance or rejection.

1.10. PRECISION VS ACCURACY

- The **accuracy** of an experiment, object, or value is a measurement of how closely results agree with accepted value. The degree of conformity and

correctness of something when compared to a true or absolute value. Single factor or measurement.

- The **precision** of an experiment, object, or value is a measure of the reliability and consistency. A state of strict exactness - how often something is strictly exact. Multiple measurements or factors are needed.

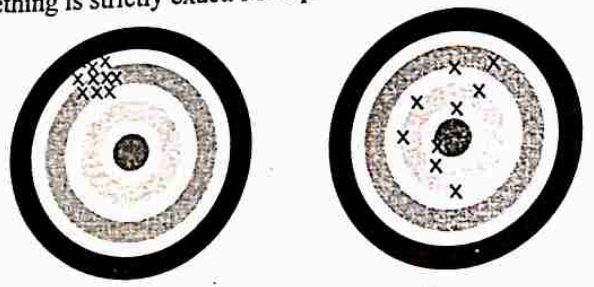


Fig. 1.14. Precision vs. Accuracy

TWO MARK QUESTIONS WITH ANSWERS

1. ***What are the major types of materials?***

Solid materials have been conveniently grouped into three basic categories,

- Metals
- Ceramics
- Polymers

2. ***Define line defects.***

- Line defects are called dislocations and are the edges of surfaces where there is a relative displacement of lattice planes. One type is an edge dislocation, and the other is a screw dislocation.

3. ***What are the benefits of testing?***

- Safety issues can be identified
- It provides reliability
- It is cost effective
- It offers reassurance

4. ***What are types of material testing?***

- Materials testing classified into three major categories
  - Mechanical testing (or) Destructive testing (DT)
  - Nondestructive testing.
  - Material characterization testing

5. ***Define NDT.***

- Nondestructive Testing (NDT) consists of a variety of non-invasive inspection techniques used to evaluate material properties, components, or entire process units. The techniques can also be utilized to detect, characterize, or measure the presence of damage mechanisms (e.g. corrosion or cracks).

6. ***Write contrast between NDT and destructive testing.***

DESTRUCTIVE TEST	NON-DESTRUCTIVE TEST
❖ Tests are usually quantitative measurements of load for failure, significant distortion or damage, or life to failure under given loading and environmental conditions.	❖ Tests are usually quantitative and rarely quantitative. They do not usually measure load for failure or life to failure even indirectly.
❖ The correlation of result is directly given by observer.	❖ Skilled judgment and test or service experience are usually required to interpret test indications.
❖ Destructive tests are not usually to apply on parts in working condition.	❖ Non-destructive tests may often be applied to parts in working assemblies without interruption or service beyond normal maintenance or idle periods.

7. ***What is the test used to test metals?***

- Major test used for testing metals are destructive one i.e., Test, Shear(Torsion test),Test, Creep Test, Bending test etc

8. ***Why more concentration is needed for selection of materials?***

- Material selection is one of the foremost functions of effective engineering design as it determines the reliability of the design in terms of industrial and economical aspects.
- It is Iterative in nature, there is a strong element of trial and error where an initial design is done and then analyzed, tested, and subjected to trial production. Changes may be made at any stage of the process to satisfy requirements not previously considered or problems just discovered.

9. ***What are factors to be considered during selection materials?***

- Performance
- Mechanical properties
- Wear of materials
- Corrosion
- Ability to manufacture
- Cost
- Reliability and Environmental Resistance
- Reducibility

10. ***What are stages in development of testing?***

- Identify the Need & Define the Problem
- Research the Problem
- Develop possible testing methods
- Evaluate the Alternatives & Select Most Promising methods
- Initial Design
- Construct a prototype
- Test and Evaluate the Prototype
- Communicate the Design
- Redesign

11. ***Define prototype.***

- A prototype, or trial model, is often made and subjected to simulated service testing to demonstrate whether or not a machine or vehicle functions properly.

12. ***Differentiate precision and accuracy.***

S.No.	ACCURACY	PRECISION
1.	The accuracy of an experiment, object, or value is a measurement of how closely results agree with accepted value.	The precision of an experiment, object, or value is a measure of the reliability and consistency.
2.	The degree of conformity and correctness of something when compared to a true or absolute value. Single factor or measurement.	A state of strict exactness — how often something is strictly exact. Multiple measurements or factors are needed.

13. ***Why development of testing is necessary?***

- A problem can be regarded as a difference between the actual situation and the desired situation. It involves diagnosing the situation so that the focus on the real problem.
- Development of various time, cost, sample and labor minimizing testing techniques.
- The destruction of material reduction technique.
- Scale-up of a testing technology.
- Increase fundamental understanding of materials.
- Improvement of the process/product performance relative to the needs and demands of customers.
- Reduction of existing process spread, which leads to poor capability.

14. ***Define ISO.***

- The International Organization for Standardization (ISO) is an international standard-setting body composed of representatives from various national standards organizations.
- ISO is a voluntary organization whose members are recognized authorities on standards, each one representing one country. Members meet annually

at a General Assembly to discuss the strategic objectives of ISO. The organization is coordinated by a central secretariat based in Geneva.

15. ***What is the testing standard organization followed in India?***

- ❖ BIS is the National Standard Body of India established under the BIS Act 2016 for the harmonious development of the activities of standardization, marking and quality certification of goods and for matters connected therewith or incidental there to.

REVIEW QUESTIONS

1. Write a review on types of materials.

   Ans: Section No. 1.1 **Page No:** 1.1

2. What are aspects that you understand from testing of materials?

   Ans: Section No. 1.2 **Page No:** 1.9

3. Write the classification of various material testing.

   Ans: Section No. 1.2 **Page No:** 1.9

4. What are the advantages and disadvantages encountered by various material testing?

   Ans: Section No. 1.2 **Page No:** 1.10

5. Differentiate between NDT and Destructive testing.

   Ans: Section No. 1.2 **Page No:** 1.14

6. Why testing of materials are important?

   Ans: Section No. 1.3 **Page No:** 1.16

7. What are steps to be followed during selection of materials?

   Ans: Section No. 1.4 **Page No:** 1.17

8. What are criteria that affect the selection of materials?

   Ans: Section No. 1.4 **Page No:** 1.19

9. Explain various stages in development of testing in detail.

   Ans: Section No. 1.5 **Page No:** 1.22

10. What are the purpose of developing a test? Explain with few examples.

    Ans: Section No. 1.5 **Page No:** 1.26

11. What is BIS? Explain its organization.

    Ans: Section No. 1.6 **Page No:** 1.28

12. Explain various benefits of testing.

    Ans: Section No. 1.7 **Page No:** 1.31

13. How will you represent the result analysis of testing?

    Ans: Section No. 1.8 **Page No:** 1.32

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UNIT II

MECHANICAL TESTING

SYLLABUS

Introduction to mechanical testing, Hardness test (Vickers, Brinell, Rockwell), Tensile test, Impact test (Izod, Charpy) - Principles, Techniques, Methods, Advantages and Limitations, Applications. Bend test, Shear test, Creep and Fatigue test - Principles, Techniques, Methods, Advantages and Limitations, Applications.

2.1. INTRODUCTION TO MECHANICAL TESTING

- Mechanical testing reveals the properties of a material under dynamic or static force which is also known as destructive testing.
- Designed to ensure that materials are suitable for their intended applications, mechanical testing includes methods such as tensile strength, compression strength, impact resistance, fracture toughness and fatigue.
- Materials testing study reveals the behavior of materials under different loads. In particular, the relationship between the acting forces and the resulting deformation is found, in which the limit stresses that lead to failure of components are considered.
- To assure performance, safety, and durability, it is necessary to avoid excess deformation i.e. bending, twisting, or stretching of the components (parts) of the machine, vehicle, or structure. In addition, cracking in components must be avoided entirely, or strictly limited, so that it does not progress to the point of complete fracture.
- **The study of deformation and fracture in materials is called mechanical behavior of materials.** Knowledge of this area provides the basis for avoiding these types of failure in engineering applications. One aspect of the subject is the physical testing of samples of materials by applying.

1. MECHANICAL PROPERTIES OF MATERIAL

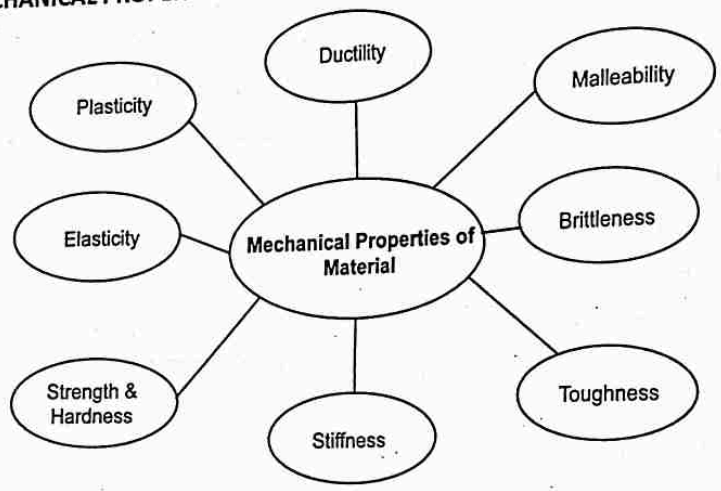


Fig. 2.1. Mechanical properties

1. Strength & Hardness

- Strength is the ability of a material to resist the externally applied forces without breaking or yielding. The internal resistance offered by a part to an externally applied force is called stress.
- Hardness is a very important property of the metals and it also embraces many different properties such as resistance to wear, scratching, deformation and machinability etc.

2. Stiffness

- Stiffness is the ability of a material to resist deformation under stress. The modulus of elasticity is the measure of stiffness.

3. Elasticity

- It is the property of a material to regain its original shape after deformation when the external forces are removed. This property is desirable for materials used in tools and machines than rubber.

4. Plasticity

- Plasticity is a property of a material which retains the deformation produced under load permanently. This property of the material is necessary for forgings, in stamping images on coins and in ornamental work.

5. Ductility

- Ductility is the property of a material enabling it to be drawn into a wire with the application of a tensile force. A ductile material must be both strong and plastic. The ductility is usually measured by the terms, percentage elongation and percentage reduction in area. The ductile material commonly used in engineering practice are mild steel, copper, aluminum, nickel, zinc, tin and lead.

5. Brittleness

- It is the property of breaking of a material with little permanent distortion. Brittleness of a material is opposite to ductility property.
- Brittle materials are withstanding compression load. When subjected to tensile loads snap off without giving any sensible elongation. Cast iron is a brittle material.

6. Malleability

- It is a special case of ductility which permits materials to be rolled or hammered into thin sheets, making wire. A malleable material should be plastic in nature but it is not essential to be so strong. The malleable materials commonly used in engineering practice are lead, soft steel, wrought iron, copper, and aluminum.

7. Toughness

- Toughness is the property of a material to resist fracture due to high impact. It is measured by the amount of energy that a unit volume of the material has absorbed after being stressed up to the point of fracture.
- This property is desirable in parts subjected to shock and impact loads.

8. Resilience

- It is the property of a material to absorb energy and to resist shock and impact loads. It is measured by the amount of energy absorbed per unit volume within elastic limit. This property is essential for designing the spring materials.

2. PROPERTY BASED TESTING METHODS

- The characteristic values obtained from the testing process are used for materials development, designing components and in quality assurance.

- There is a range of standardized testing methods to characterize the mechanical properties of materials as precisely as possible

Table 2.1. Mechanical Property vs. test

Mechanical Property	Destructive Testing Method
❖ Elasticity, Plasticity	❖ Tensile Test, Compression Test, Bending Test, Torsion Test
❖ Stiffness, Material Behaviour Under Static Load	
❖ Creep Behaviour	❖ Creep Rupture Test
❖ Hardness	❖ Brinell, Rockwell, Vickers
❖ Toughness	❖ Impact Test
❖ Fatigue Behaviour, Fatigue Strength	❖ Wöhler Fatigue Test

3. MATERIAL FAILURE

- **Material failure** is the loss of load carrying capacity of a **material** unit. This definition introduces to the fact that **material failure** can be examined in different scales. The material failure happens due to two major phenomena,
  - Deformation failure
  - Fracture failure

(a) DEFORMATION FAILURE

- A deformation failure is a change in the physical dimensions or shape of a component that is sufficient for its function to be lost or impaired.
- **Elastic deformation & Plastic deformation** - Deformation that appears quickly upon loading can be classed as either elastic deformation or plastic deformation. Elastic deformation is recovered immediately upon unloading, whereas plastic deformation is permanent.
- **Creep-**It is deformation that accumulates with time. Depending on the magnitude of the applied stress and its duration, when deformation may become so large that a component can no longer perform its function.

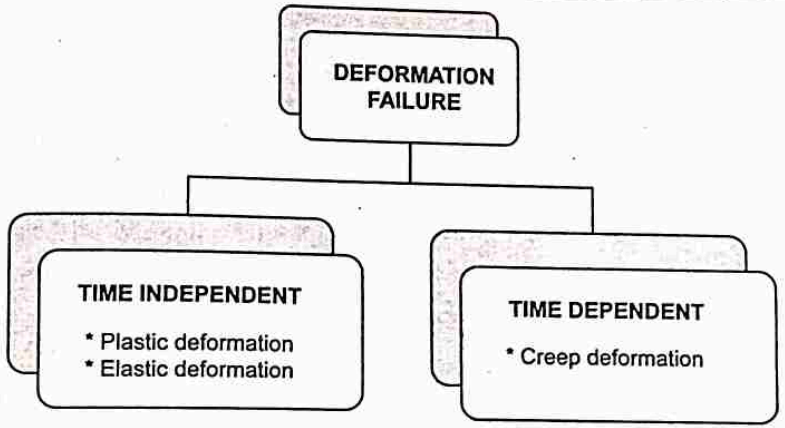


Fig. 2.2. Types of demoformartion failure

(b) FRACTURE FAILURE

- Cracking to the extent that a component is separated into two or more pieces is termed fracture.

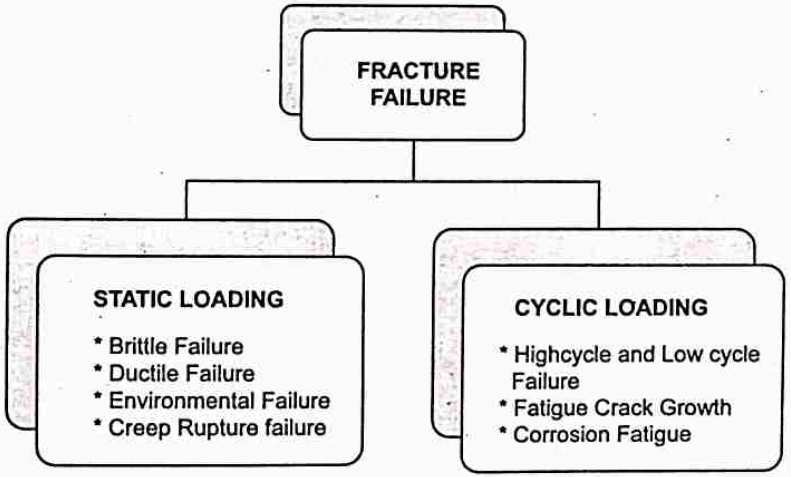


Fig. 2.3. Fracture failure

- **Brittle or Ductile fracture-**The possible growth of the cracks by fatigue, as this can lead to a brittle or ductile fracture once the cracks are sufficiently large. Such situations are identified by the term fatigue crack growth and may also be analyzed.

- **Environmental cracking failure**-Fracture may occur as a result of a combination of stress and chemical effects, and this is called environmental cracking failure.
- **Corrosion fatigue**-It is the combination of cyclic loading and corrosion. It is often a problem in cyclically loaded
- **Fatigue crack growth** -Creep deformation may proceed to the point that separation into two pieces occurs. This is called creep rupture and is similar to ductile fracture, except that the process is time dependent.
- **Creep rupture** -The common cause of fracture is fatigue, which is failure due to repeated loading. In general, one or more tiny cracks start in the material, and these grow until complete failure occurs.
- **High-cycle fatigue & low-cycle fatigue** -If the number of repetitions (cycles) of the load is large, say millions then the situation is termed high-cycle fatigue. Conversely, low-cycle fatigue is caused by a relatively small number of cycles; say tens, hundreds, or thousands. Low-cycle fatigue is generally accompanied by significant amounts of plastic deformation whereas high-cycle fatigue is associated with relatively small deformations that are primarily elastic.

2.2. HARDNESS TEST

1. HARDNESS

- The term 'hardness' is a structure-sensitive mechanical property of materials, primarily associated with the surface. If a material is uniform in composition and structure, the hardness measured on the surface layer will represent the hardness of the bulk of the material.
- The hardness is defined as the resistance of a material to permanent or plastic deformation of its surface, usually by indentation, under static or dynamic load

2. CLASSIFICATION OF HARDNESS

- Depending on the manner in which the hardness test is conducted, hardness may be classified as follows
  - Indentation hardness
  - Rebound hardness
  - Scratch hardness
  - Wear or abrasion hardness
  - Cutting hardness

(a) Indentation hardness

- It is the resistance of a material to permanent indentation under static or dynamic load. The types of indentation hardness test is given below,

  (*i*) Brinell; (*ii*) Meyer; (*iii*) Vickers (macro- and micro-hardness); (*iv*) Rockwell (regular and superficial); (*v*) Knoop (micro hardness); (*vi*) Nano hardness (mostly by Vickers and Berkovich indenters) etc.
- Classification based on scale of indenter used which is describe in the table with testing methods.

Table 2.2. Types of indenter hardness test

Major group	Testing methods	Force applied
Macro-Hardness Tests (Rapid routine hardness measurements)	Rockwell test Brinell test Vickers test	50N To 30000N
Micro-Hardness Tests (Hardness of coatings, surface hardness, or hardness of different phases in the multi-phase material is measured)	Micro-Vickers test Knoop test	10 To 1000gf.
Nano-Hardness Test	-	1 Nano-Newton

(b) Rebound hardness

- It is the resistance offered by a material to strike and absorb energy for plastic deformation under impact loads, causing the hammer to rebound.
- Most common example is the 'Shore scleroscope hardness test' which measures the hardness in terms of the rebound height of the indenter. It is virtually an indentation test.

(c) Scratch hardness

- It is the resistance of a material to scratch by another material, for example Mohs scale of hardness which is discussed after.

(d) Wear or abrasion hardness

- It is the resistance of a material to abrasion and wear, when subjected to rotational or sliding motion, for example file hardness test.

(e) Cutting hardness

- It is the resistance of a material to various cutting or drilling operations. This hardness is a measure of machinability of a material.
- 'Bauer drill test' is one of the various tests employed to determine the cutting hardness or machinability of materials

4. MOHS SCALE

- Mohs scale of hardness is widely used in the field of mineralogy but rarely applied for the testing of metals and alloys; it is a qualitative ordinal scale characterizing scratch resistance of various minerals through the ability of harder material to scratch softer material.

Table 2.3. Mohs scale of hardness

Mohs scale	Material
1	Talc
2	Gypsum
3	Calcite
4	Fluorite
5	Apatite
6	Orthoclase feldspar
7	Quartz
8	Topaz
9	Corundum
10	Diamond

5. INDENTER

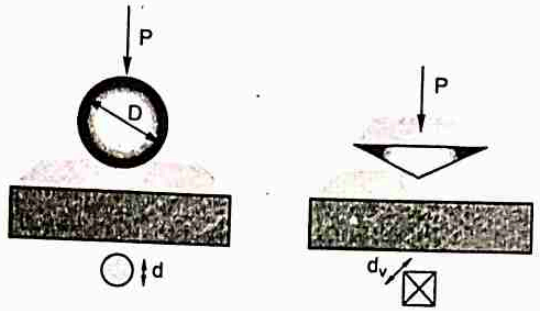


Fig. 2.4. Steel ball & pyramid indenter

- Indenter is the tool of material which causes deformation or indentation on the surface of the specimen to be tested which must be harder than the test piece.
- When the indenter is forced into the test piece, the indenter will be subjected to varying amount of elastic deformation depending on the magnitude of the applied load and the hardness of the test piece.
- The deformation mark or impression on the surface of the test piece is called indentation.

Table 2.4. Types of indenter

Indenter Type	Test
Hard Metal Ball	Brinell Hardness Test
Right Pyramid with a Square Base	Vickers Hardness Test
Diamond Or Ball Type	Rockwell Hardness Test

6. SELECTION CRITERIA OF HARDNESS TESTER

Main elements to consider before choosing a hardness tester

(a) Test load

- This is determined by the hardness of the material. Metals such as steel or alloys, for example, require test loads of up to 3,000 kgf, while soft metals require only 500 kgf. The higher the load, the higher the accuracy. It is important to note that the impression should not exceed 1/10 of the thickness of the sample.

(b) Hardness range

- It determines the material of the indenter. Over 650 HB/30 hardness, you should favor a diamond indenter. Below this value, steel or hard metal indenters are suitable.

(c) Accuracy level

- It depends on the surface to be measured (cleanliness, flat surface, static or dynamic system, etc.).

(d) Adaptability of the device

- It is importance varies according to the shape and size of the samples to be tested.

6. BENEFITS OF HARDNESS TEST

- Easy
- Inexpensive
- Quick
- Non-destructive
- May be applied to the samples of various dimensions and shapes
- May be performed in-situ

2.3. BRINELL HARDNESS TEST

- The standardized method for quantitative determination of indentation hardness, which was the first widely accepted indentation hardness test known as 'Brinell hardness' test
- **The Brinell hardness test consists of forming an indentation by forcing a standard spherical ball indenter into the surface of the material.**
- In this test a hardened steel ball of 2.5, 5 or 10 mm in diameter is used as indenter.
- The loading force is in the range of 300N to 30000N (300N for testing lead alloys, 5000N for testing aluminum alloys, 10000N for copper alloys, 30000N for testing steels).

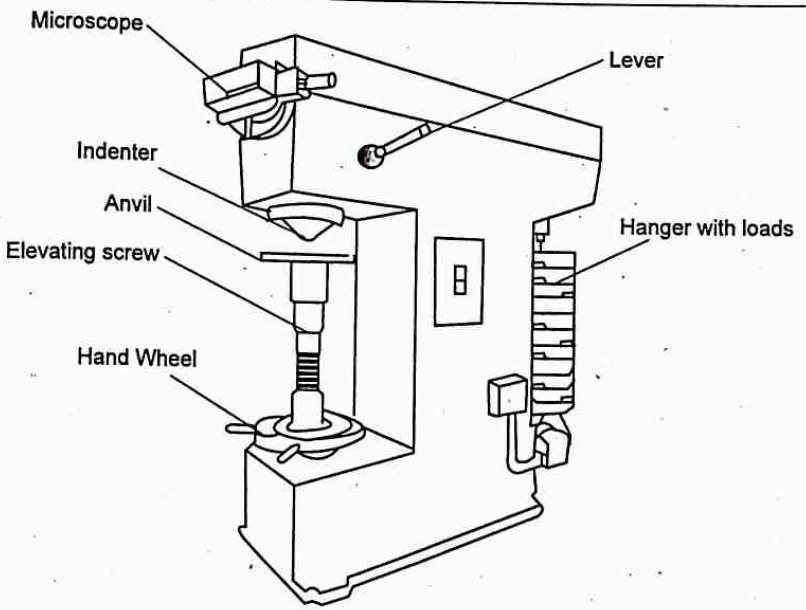


Fig. 2.5. Brinell tester with components

1. PRINCIPLE

- An indenter (hard metal ball with diameter) is forced into the surface of a test piece and the diameter of indentation, '*d*' left in the surface after removal of the surface, 'F' is measured under a definite static load applied for a standard period of time.
- The standard Brinell hardness tester operates usually under hydraulic pressure that applies force.

2. MAJOR COMPONENTS

- Brinell hardness tester
- Brinell microscope
- Indenters and Plunger
- Anvil

3. INDENTERS

- The diameters of spherical steel ball indenters used in the standard Brinell hardness test are either 5 or 10 mm. The ball indenter normally used is

made from heat treated hard high carbon steel, known as 'Hultgren ball' (made from tungsten carbide).

4. LOAD APPLICATION

- In Brinell hardness tester, load application and time duration is based the materials tested.

Table 2.5. Load application of metal

Load application	Diameter of ball	Duration	Metals
3000 kg	10-mm	10 Seconds	Iron, Steel and Alloys Having Hardness Similar to Steel
750 kg	5-mm		
500 kg	10-mm	30 Seconds	Copper, Annealed Brass and Magnesium Alloys etc.,
1000 kg	10-mm	15 Seconds	Gun Metal/Bronze and Cold-Worked Brass, etc

4. WORKING

- The surface of the test specimen must be either machined, ground, lapped or polished.
- The specimen is placed on the anvil of the testing machine, and the anvil is raised by rotating the hand wheel so that the specimen surface is brought in tight contact with the apex of the indenter
- Place the specimen on the test table and, apply a minor load to bringing both the pointers on the dial gauge to the 'set' positions.
- Apply the major load (remaining part of the test load) on the specimen by turning the loading lever backward.
- Maintain the load on the specimen exactly for the specified dwell time (15 seconds) and then release it by turning the loading lever forwards.
- Take out the specimen and measure the diameter of the indentation formed on it by using the Brinell Microscope.

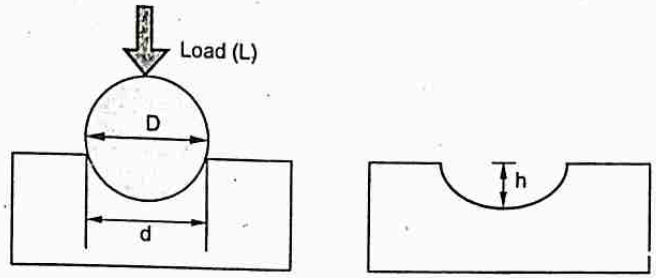


Fig. 2.6. Indentator impression

5. BRINELL HARDNESS NUMBER (BHN)

- The Brinell hardness number (BHN), expressed in units of kilograms per square millimeter , is defined as the ratio of the applied load (F in kilograms) to the curved surface area of the elastically recovered indentation(Area in square millimeters).

Brinhell hardness number (BHN) H_b = F/Area

$$\text{Area} = \frac{\pi D}{2}\left[D - \sqrt{(D^2 - d^2)}\right]$$

$$H_B = \frac{2F}{\pi D\left[D - \sqrt{D^2 - d^2}\right]}$$

$$h = \frac{1}{2}\left[\frac{D}{D^2 - d^2}\right]$$

H_b = Brinell hardness number (BHN)

h = Depth of Indentation (h)

D = Diameter of Ball in mm

L = Applied load in kg

d = Diameter of indentation in mm.

Table 2.6. Brinell hardness number for varies materials

Recommended Materials	Brinell Hardness Number (BHN)
Steel and similarly hard ferrous and other alloys	140–600
Harder Non-ferrous metals and alloys like gun metal/bronze, cold-worked brass	50–200

Recommended Materials	Brinell Hardness Number (BHN)
Non-ferrous metals and alloys like copper, annealed brass, magnesium alloys	25 – 100
Softer Non-ferrous metals and alloys like aluminum, lead, tin and their alloys	10 – 50

6. BEHAVIOUR OF DEFORMATION

Two types of anomalous behaviour, as illustrated schematically in cross-sections of Brinell indentation

- ❖ 'Ridging' or 'Piling up' which there is a formation of a lip or a raised ridge of material around the periphery of the indentation. This condition is observed with cold-worked materials having little ability to strain-harden.
- ❖ 'Sinking in' in which a depressed surface of the material is formed around the periphery of the indentation. This type of behaviour is frequently observed with annealed materials having a high rate of strain hardening.

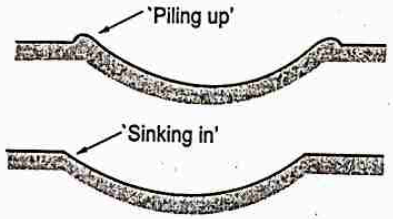


Fig. 2.7. deformation behaviour

7. ERROR IN READING

- ❖ Errors in reading the diameter of the indentation may arise from the following two causes ,
  - (*i*) Error in reading from the microscope
  - (*ii*) The boundary of the indentation is not distinct.

8. ADVANTAGES

- ❖ Simple, robust and low-cost indenters.
- ❖ The Brinell method can be used for testing non-homogeneous materials.
- ❖ A choice can be made between a large numbers of test forces.
- ❖ The influence of surface scratches and roughness will be less in the Brinell test than other hardness tests.
- ❖ The specimen surface can be rough.
- ❖ Suitable for hardness tests on large blanks such as forged pieces, castings and hot-rolled etc
- ❖ Measurement is usually not affected by movement of the specimen.

9. DISADVANTAGES

- ❖ Restriction of application range to a maximum Brinell hardness of 650 HBW.
- ❖ Restriction when testing small and thin-walled specimens.
- ❖ The test location must be prepared.
- ❖ High risk of deforming the material to be in test.
- ❖ Good illumination of the test indent is important for ensuring correct evaluation of the test indent.
- ❖ Limitation in applying the method on thin specimens of very hard materials.
- ❖ The process is slow (by comparison with the Rockwell method).
- ❖ Due to the larger size of the indentation, the application of Brinell hardness test is not possible on small jobs or critically stressed portions which would crack during the indentation.

2.4. ROCKWELL HARDNESS TEST

- ❖ In the Rockwell test the depth of the indenter penetration into the specimen surface is measured. Each time a test is performed two loads are applied to the sample being tested.

1. PRINCIPLE

- ❖ Rockwell hardness test is to determine the hardness of a metal by 'differential depth' measurement test. This hardness testing method

involved the measurement of the increment of depth of an indenter force into the metal by a primary and a secondary load.

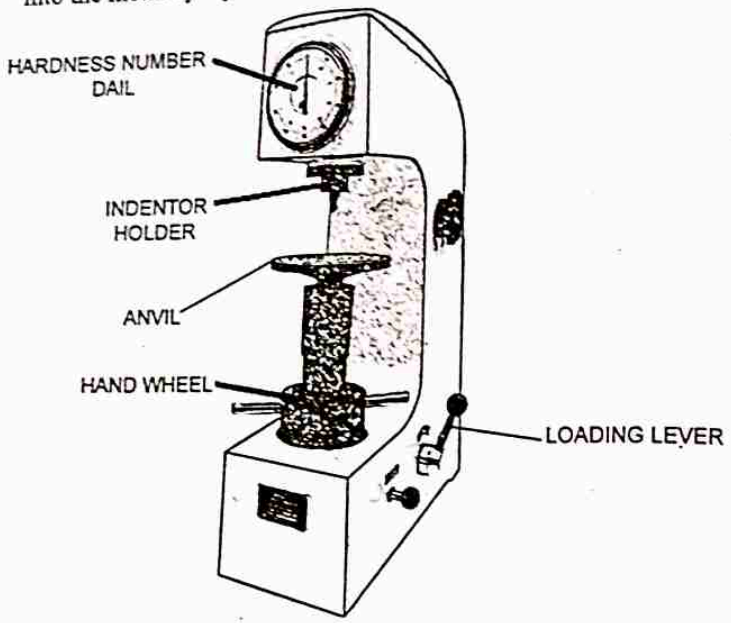


Fig. 2.8. Rockwell hardness tester

2. COMPONENTS

- Rockwell hardness tester
- Indenter

3. INDENTER

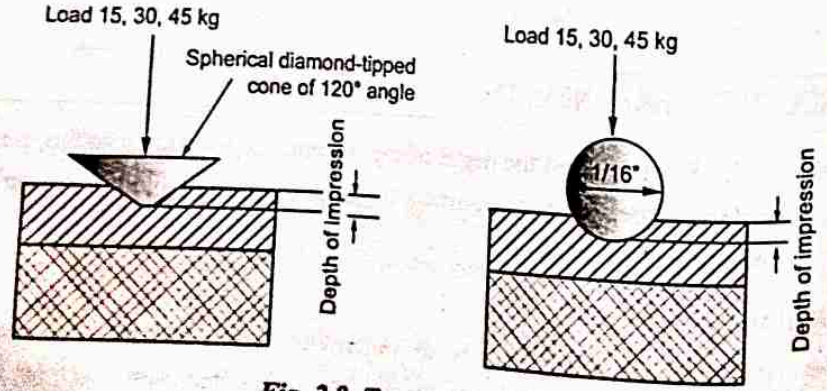


Fig. 2.9. Types of indenter

- The indenter or 'penetrator' is either made of hardened steel with shape of a spherical ball or made of diamond with shape of a cone having a spherical tip called the 'Brale'.
- The indenter may be either a diameter 1/16", 1/8" or a spherical diamond cone of 120° angle.

4. LOADING CONDITION

- Loads during testing are applied in two stages
  - Minor static load
  - Major static load

(a) Minor Static Load

- Minor static load of 10 kg is applied to form a very shallow indentation on the surface of the specimen through compression of a calibrated coiled spring placed within the machine between the indenter shaft and the dial.
- The purpose of applying minor load is as follows:
  - To eliminate the error that may arise due to variable contacts between the indenter and the surface of the specimen.
  - To set the indenter on the specimen and hold it in position.
  - To eliminate the error that may arise due to slight surface imperfections, i.e. to minimize the surface preparation of the specimen.
  - To reduce the tendency for 'ridging' or 'sinking in' caused by the indenter.

(b) Major Static Load

- Major static load of either 50, 90 or 140 kg is applied on the surface of the specimen through a system of weights and levers by means of an operating handle in the machine that enlarges the initially formed indentation under minor load.
- The total static load applied for indentation on the test piece is the summation of the minor load and the major load, which is equal to either 60 or 100 or 150 kg.

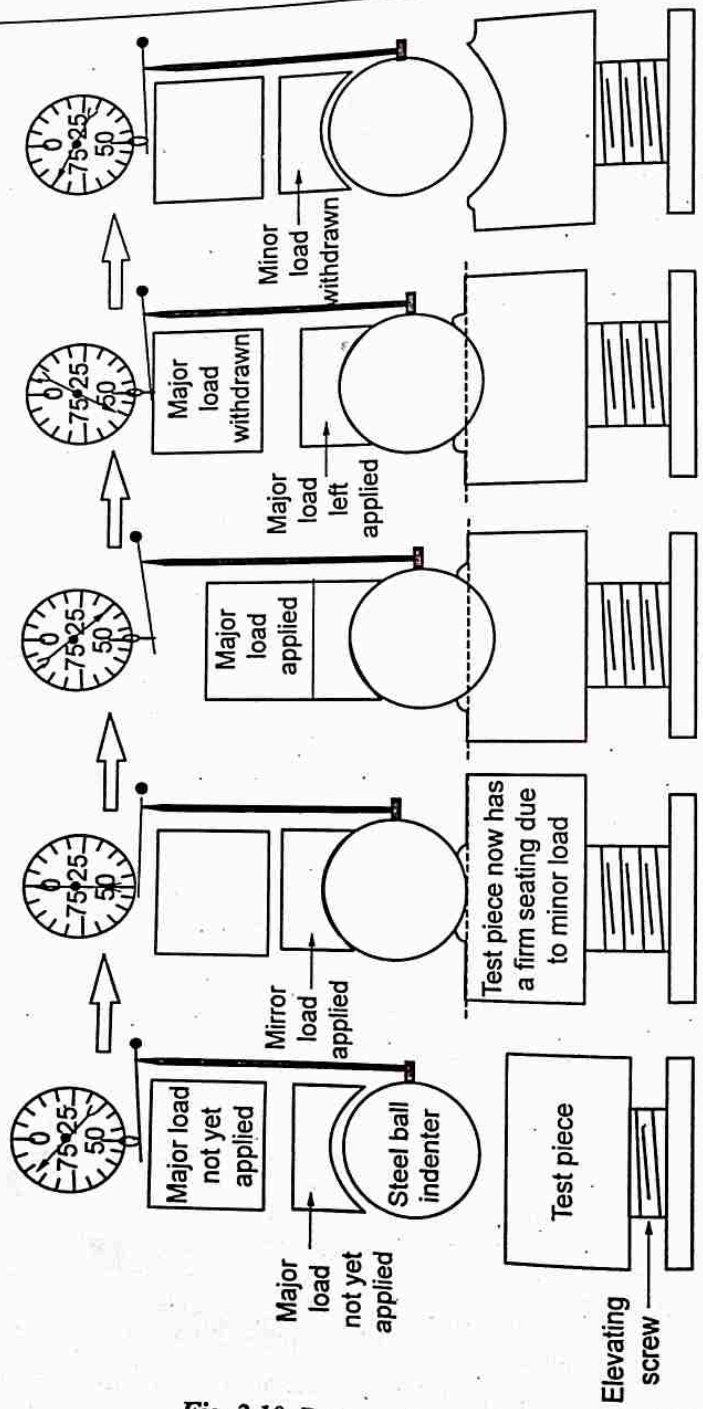


Fig. 2.10. Process of loading

5. WORKING

- The specimen to be tested is made flat by grinding and then roughly polished because any surface irregularities will be taken care of by the minor load.
- The application of the minor load becomes effective when the surface of the specimen kept on the anvil is brought in contact with the indenter by rotating the anvil elevating wheel.
- First, the indenter is forced into the test material under a preliminary minor load and this depth is recorded.
- With the minor load still applied an additional load is introduced known as the major load which increases the depth of penetration on the sample.
- The major load is then removed, and the force on the sample is returned to the minor load.
- The increase in the depth of penetration that results from applying and removing the major load is used to calculate the Rockwell hardness value.

6. ROCKWELL HARDNESS SCALE

- The Rockwell hardness value obtained for materials of different hardness with different combinations of total or major load and indenter are employed during the tests.

Table 2.7. Rockwell hardness scale

Rockwell Hardness Scale				
Scale	**Indenter**	**Load (kg)**	**Dial number**	**Typical material**
A	Diamond cone	60	Black	Cemented carbide, case hardened surface, thin steel
B	1.5 mm ball	100	Red	Copper, aluminium, brass, cast iron
C	Diamond cone	150	Black	Hard cost iron, hardened steel
D	Diamond cone	100	Black	Thin steel specimens

E	3 mm ball	100	Red	Soft aluminium and alloys, magnesium alloy, bearing metals
F	1.5 mm ball	60	Red	Bearing alloy, anneled copper and alloy

7. APPLICATIONS

- It is widely applied in the industry of Cemented carbides, Copper alloys Thin steel and medium case hardened steel, Cast iron, aluminum etc due to the rapidity and simplicity

8. ADVANTAGES

- High accuracy is achieved.
- Relatively low procurement costs for the testing machine because no optical measuring device is necessary.
- Relatively short test time because the hardness value is automatically displayed immediately following the indentation process.
- Relatively short time needed to train operator.
- Only small size of the impressions produced and lot of trails is followed in same specimen.
- It is generally used for testing of larger samples.
- It can be used for advanced tests.
- There was no special surface preparation.
- No measurements of indentation profiles as with other hardness tests.

9. DISADVANTAGES

- The main limitations are due to the fact that between maximum and minimum load there is only a 10:1 ratio.
- There has a possibility of errors due to the shifting of samples under test loads during the test cycle.
- The quality of the indenter and the surface has a strong influence on the test results.

- Possibility of measurement errors due to movement of the test piece and poorly seated or worn machine components during the application of the test forces.
- Sensitivity of the diamond indenter is high. If any damage in diamond indenter, thus producing a risk of incorrect measurements.
- Relatively low sensitivity on the difference in hardness.

10. ADVANTAGES OVER VICKER AND BRINELL HARDNESS TEST

- Its advantage over the Brinell test is that it can measure the hardness of harder materials that is beyond the scope of the Brinell test.
- It is faster because arbitrary hardness values can be read directly from the dial of the machine.
- It differs from the Brinell test in that the indentation and the loads are smaller, and hence, the resulting indentation is smaller and shallower, which is less objectionable in the finished parts.
- Due to application of minor load, the surface preparation of the specimen is minimized in comparison to the Brinell as well as Vickers hardness tests. Only rough grinding of specimen surface may be adequate for Rockwell hardness test.

2.5. VICKER HARDNESS TEST

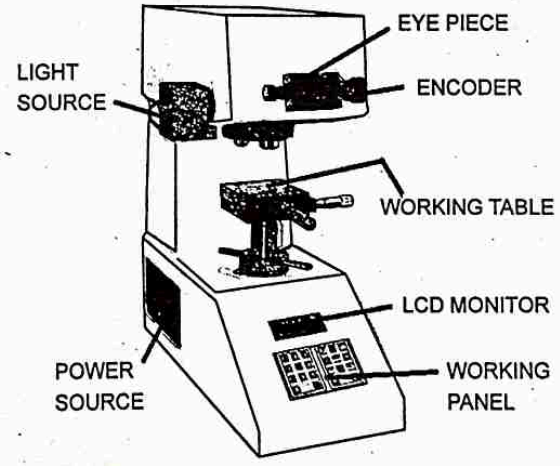


Fig. 2.11. Vickers hardness tester

❖ The Vickers hardness test, used to determine quantitatively the indentation hardness of material under the application of a constant static load, is a widely accepted method for research work because it is capable of measuring hardness from very soft materials to extremely hard materials without changing the load or indenter.

1. PRINCIPLE

❖ A diamond indenter in the form of a right pyramid with a square base and with a specified angle between opposite faces at the vertex is forced into the surface of a test piece followed by measurement of the diagonal length of the indentation left in the surface after removal of the test force F.

❖ The Vickers hardness test is a static hardness test method, used for both macro and micro hardness testing. It is an optical method of testing where the size of the indentation left by the indenter is measured to determine the hardness value of a test specimen.

2. COMPONENTS

❖ Vickers hardness tester

❖ Intender

3. INTENDER

❖ It is made of diamond in the form of a square-based pyramid with an included angle of 136° between opposite faces.

4. LOADING CONDITION

❖ The loads that can be applied to the indenter in Vickers hardness tester are 1, 2.5, 5, 10, 20, 30, 50, 100 and 120 kg through appropriate selection of weights.

5. WORKING

❖ Place the specimen carefully on the testing table.

❖ Turn the hand wheel slowly in the clockwise direction so that the specimen gets focused on the front screen sharply.

❖ Now bring the inventor to the "set" position and turn on the loading, dwell-unloading cycle.

❖ The indentation is now projected on the front focusing screen.

❖ Measure the diagonals along both the axis of the impression and record them.

6. VICKERS HARDNESS NUMBER

❖ Vickers hardness number is frequently called as the diamond pyramid hardness number.

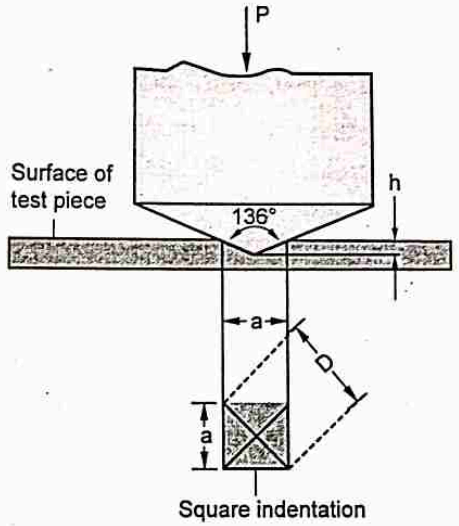


Fig. 2.12. Vickers diamond indenter

❖ The Vickers hardness number (VHN) is defined as the ratio of the applied load, P (kilogram) to the surface area of the elastically recovered pyramidal indentation (A_s in square millimeters).

$$VHN = \frac{P}{A_s} \text{ kg/mm}^2$$

$$VHN = \frac{P}{D^2 / 1.854}$$

$$= 1.854 \times \frac{P}{D^2}$$

P = Applied load in (kg)

A_s = Lateral area of elastically recovered pyramidal indentation (mm^2)

D = Average diagonal length of square indentation in (mm)

7. ADVANTAGES

- The Vickers method can be used with any & all materials and test specimens, from soft to hard, as it covers the entire hardness range.
- Testing thin sheets, small test pieces or test surfaces, thin-walled tubes, thin, hard and plated coatings is possible.
- There is only one type of indenter, which can be used for all Vickers methods.
- Non-destructive testing is possible, so the test specimen can be used for other purposes
- The small indentation has no influence on the function or appearance of tested materials or products.
- Useful for finding stress values.

8. DISADVANTAGES

- The test location must be prepared (ground and polished), otherwise precise evaluation is difficult.
- The process is rather slow (compared with the Rockwell method). The test cycle takes somewhere between 30 and 60 seconds, not including the time taken to prepare the specimen.
- Relatively long test time due to the measurement of the diagonal lengths.
- Due to the need to conduct optical indent evaluation, Vickers hardness testers must be equipped with an optical system, which makes them more expensive to purchase than Rockwell testers.
- Sensitivity of the diamond indenter to damage.
- Very sensitive to effects of vibration, especially in the micro hardness range.

2.6. KNOOP HARDNESS TEST

- The quantitative determination of hardness on materials over a very small area under the application of a constant static load, the diamond indenter known as the 'Knoop' indenter and hardness test is called Knoop hardness test (Micro hardness).

2.7. MONOTRON HARDNESS TEST

- The Monotron hardness test also operates on the depth of the indentation is fixed or predetermined under the application of variable static loads during hardness measurements of different materials, whereas different sizes of the indentations are formed under an applied constant load in other hardness tests.

2.8. NANO HARDNESS TESTS

- Nano hardness tests or Nano indentation tests, in which the magnitudes of applied forces are usually in the milli-newton range, may be as low as 0.1 mN. Majority of nano indentation tests aim to obtain Young's modulus along with measurement of hardness of the specimen material from the load–displacement data obtained in tests.

2.9. COMPARISON BETWEEN ROCKWELL TEST, BRINELL TEST AND VICKERS TEST

Properties	Brinell	Rockwell	Vicker
Indenters	Hard metal	Steel ball or diamond cone	Square-based pyramid diamond indenter with a 136° included angle
Load	Typically 1kg to 3000kg	30-100 kg	Typically 10g to 1,000g
Duration	15- 30sec	10-15 sec	30-60 sec
Advantages	Simple surface preparation, easy measurement	Higher speed, immediate reading, shallow imprint.	The test specimen can be used for other purposes.
Disadvantages	Impression is large with visible trace	Possibility of cone breakage	Surface preparation is needed

2.10. TENSILE TEST

❖ Tensile test is a measurement of the ability of a material to withstand forces that tend to pull it apart and to determine to what extent the material stretches before breaking. Tensile modulus, an indication of the relative stiffness of a material, can be determined from a stress–strain diagram.

❖ The tension test is the most common method for determining the mechanical properties of materials, such as strength, ductility, toughness, elastic modulus, and strain- hardening capability.

1. PRINCIPLE

❖ A standardized specimen with a known cross section is loaded uniformly with relatively low increasing force in the longitudinal direction.

❖ A uniaxial stress state prevails in the specimen until contraction commences. The ratio of stress to strain can be shown from the plotted load-extension diagram.

2. EQUIPMENT

❖ Universal Testing Machine (UTM), Extensometer, Scale Vernier Calipers, Punching tools.

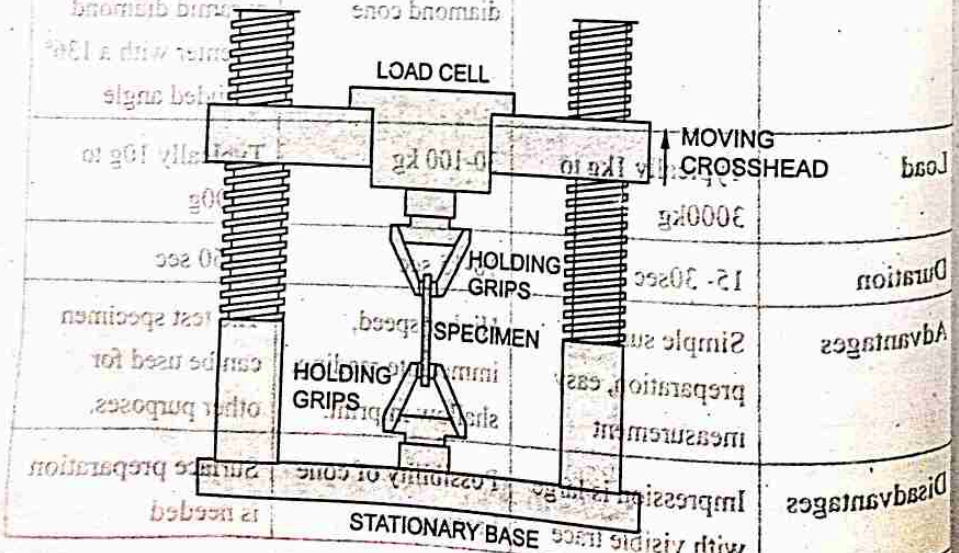


Fig. 2.13. Tensile testing apparatus

3. UNIVERSAL TESTING MACHINE

❖ Universal testing machine has two crossheads with one is adjusted for the length of the specimen and the other is hydraulic powered driven to apply tension and compression to the test specimen.

❖ It is capable of force capacity from 500N to 1MN.The strain measurements are measured with an extensometer.

4. EXTENSOMETER

❖ The accurate measurement of dimensional change achieved by attaching the sensitive measurement device to test piece. The devices used to measure longitudinal strain are termed as extensometer.

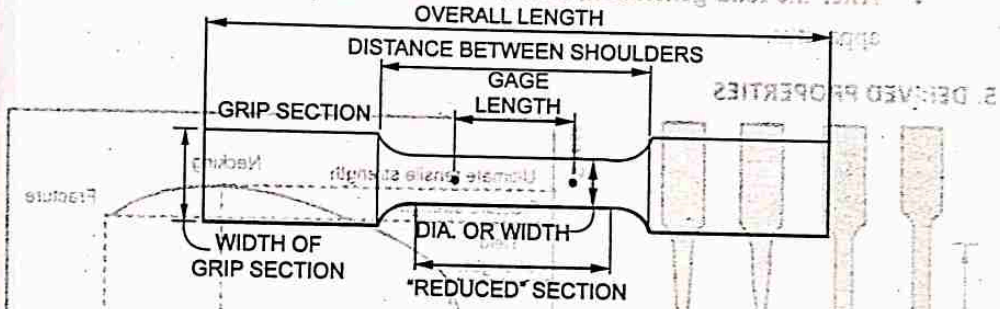


Fig. 2.14. Sample Tensile Specimen

4. WORKING

❖ **Preparation of Specimen:** Initially, the steel rod specimen is cleaned and gauge length is marked on it. The ultimate load range to be fixed.

❖ **Placing the Specimen:** Once the specimen is placed, the jaws are locked.

❖ **Placing Extensometer:** Fix the extensometer on the specimen and set the reading to zero.

❖ **Load Application:** When the specimen is under load, slowly unclamp the locking handle. Note the extension at a convenient load increment. Extensometer must be removed before reaching the yield point.

❖ **Important Load Points:** With the increase in load at some point, the load pointer remains stationary this indicates the yield point. With further increase in load, the pointer goes backward and specimen breaks. The load

before this breaking is the ultimate load. The load at the breaking of the specimen is called as the breaking load.

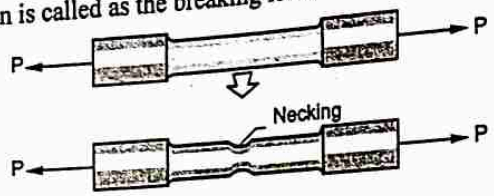


Fig. 2.15. neck formation

- Necking is a large reduction caused in the cross-sectional area of the steel rod. Measure the diameter of the specimen at the neck.
- After the load generation, load is reversed and specimen is removed from apparatus.

5. DERIVED PROPERTIES

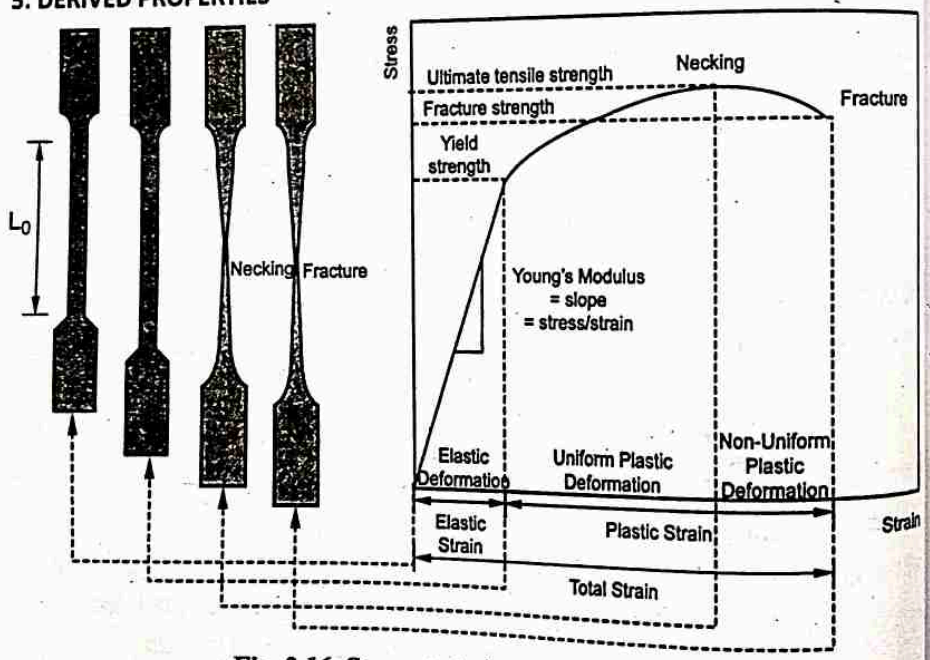


Fig. 2.16. Stress strain curve of mild steel

- **Proportional Limit:** The material is capable of sustaining the applied load without any deviation, it is defined as proportionality of stress to strain within elastic limit (Hooke's Law).

- **Elastic Limit** - The lowest stress at which permanent deformation can be measured.
- **Engineering Stress:** Stress (nominal stress) is defined as the ratio of the applied load to the original cross-sectional area of the specimen

$$\text{Stress } (\sigma) = \frac{\text{Applied load}}{\text{Original cross-sectional area}}$$

- **Engineering Strain:** Strain is defined as change in length to original length

$$\text{Strain } (e) = \frac{\text{Change in length}}{\text{Original length}}$$

$$\text{Percentage of elongation} = \frac{[\text{Final length(at fracture)} - \text{Original length}]}{\text{Original length}}$$

$$\text{Percentage of Reduction in area} = \frac{[\text{Original area} - \text{Area at fracture}]}{\text{Original area}}$$

- **Tensile strength:** Tensile strength is stress obtained at highest applied force is the tensile strength which is the maximum stress on curve.

$$\text{Tensile strength} = \frac{\text{Force (load)}}{\text{Cross-section area}}$$

- **Ultimate tensile strength:** The ultimate tensile strength at yield and at break is calculated.

$$\text{Tensile strength at yield} = \frac{\text{Maximum load}}{\text{Cross-section area}}$$

$$\text{Tensile strength at break} = \frac{\text{Break load}}{\text{Cross-section area}}$$

- **Tensile Modulus (Modulus of elasticity or Young's modulus):** Tensile Modulus and Elongation are derived from a stress strain curve. The extensometer is magnifies the actual stretch of the specimen.

$$\text{Tensile modulus} = \frac{\text{Difference in stress}}{\text{Difference in corresponding strain}}$$

- **Yield strength:** The yield strength (YS) is the stress required to produce a small specified amount of plastic deformation.

$$\text{Yield point} = \frac{\text{Load at sharp discontinuity}}{\text{Original cross sectiona area}}$$

❖ **Upper Yield Point and Lower Yield Point**

(*a*) Upper Yield Point is beyond elastic limit a ductile material has plastic properties. Upper yield point is the point at which maximum external load or stress is required to initiate plastic deformation inside the material.

(*b*) Lower Yield Point is material length will increase with a very small increase in external load (stress). In other words it is the point at which minimum load is required to maintain the plastic behavior of the material

❖ **Ductility:** Ductility is the extent of plastic deformation that the material undergoes before fracture.

❖ **Elasticity:** Elasticity is the property of the material which enables the material to return to its original form after the external force is removed.

❖ **Necking:** In stress strain curve when the specimen reaches its ultimate stress then diameter of portion starts decreasing because of local instability. This phenomenon is called necking.

❖ **Plasticity:** It is a property that allows the material to remain deformed without fracture even after the force is removed.

❖ **Strain hardening:** The increase in the tensile strength of the material is due to strain hardening which is due to the increased dislocations interactions during the deformation of the tensile test. This is called Strain-hardening.

❖ **Proof Stress:** The stress that causes a percentage increase in gauge length. It can be found by drawing a line parallel to the straight part of the graph. A value can be taken from the vertical axis.

6. FACTORS AFFECTING TENSILE TESTING

❖ Specimen Preparation and Specimen Size
❖ Rate of Straining
❖ Temperature
❖ Hydrostatic Pressure Effects
❖ Radiation Effects

7. VARIES FORMS OF TENSILE TEST

1. Tensile adhesion
2. Tensile shear
3. Tensile grab
4. Tensile pulling
5. Tension fatigue
6. Tensile creep.

8. ADVANTAGES

❖ The main advantages of this test are to check yield strength, tensile strength and ductile property of material.
❖ Used for selecting materials for an application based.
❖ It provides safety and integrity of materials.
❖ It determines batch quality.

9. DISADVANTAGES

❖ It does not provide information about the material at different temperatures.
❖ It does not identify the strength of the material at differing strain rates.
❖ It does not identify any possible asymmetry in the material strength.
❖ It provides no information about the strength of the material in different environments.
❖ It provides no information about changes in the material strength due to the process of forming the material. (A casting will have different properties than a forging or a sintered metal part.
❖ Since it is destructive testing, the material gets wasted every time. The test is mainly restricted to ductile materials.
❖ Tensile test is a destructive testing, where sample is made in Standard size.
❖ It is done under constant strain rate and constant temperature.

2.11. IMPACT TEST

- Impact testing is a very popular and fast method for evaluating the fracture toughness of materials. One of the purposes of this method is to evaluate the energy absorbed by a standard specimen during tests at a very high strain rates.
- The impact test is a dynamic test, carried out with notched specimen and known as notched-bar impact test.

1. METHODS OF IMPACT TESTING

- Impact blow may be applied by means of dropping weight, a swinging pendulum and a rotating flywheel.
- Specimen is ruptured in impact test by a single blow, repeated blows of constant magnitude and repeated blows of increasing magnitude.
- Impact tests may be carried out with different types of loading, Flexural loading, Tensile loading, Compressive loading and Torsional loading.
- Based on the loading condition, impact blow pattern and rupturing blow, the impact test is classified as follow

1. Single-Blow Pendulum Impact Test
   - (*i*) Charpy Notched-Bar Impact Test
   - (*ii*) Izod Notched-Bar Impact Test
2. Drop Weight Test (DWT)
3. Robertson Crack-Arrest Test
4. Dynamic Tear (DT) Test
5. Instrumented Puncture Testing
6. Tensile Impact test.

2. PURPOSE OF IMPACT TESTING

- The purpose of an impact test is to determine the ability of the material to absorb energy during a collision.

- This energy may be used to determine the
  - Toughness
  - Impact Strength
  - Fracture Resistance
  - Impact resistance or fracture resistance of the material

2.12. THE IZOD AND CHARPY TEST

- **The Charpy V- notch** impact test is the most common fracture toughness test. A notched specimen is broken by a swinging pendulum and the amount of energy required to break the specimen is recorded.
- **The Izod impact** strength test is a standard method of determining the impact resistance of materials. A pivoting arm is raised to a specific height (constant potential energy) and then released. The arm swings down hitting a notched sample, breaking the specimen.

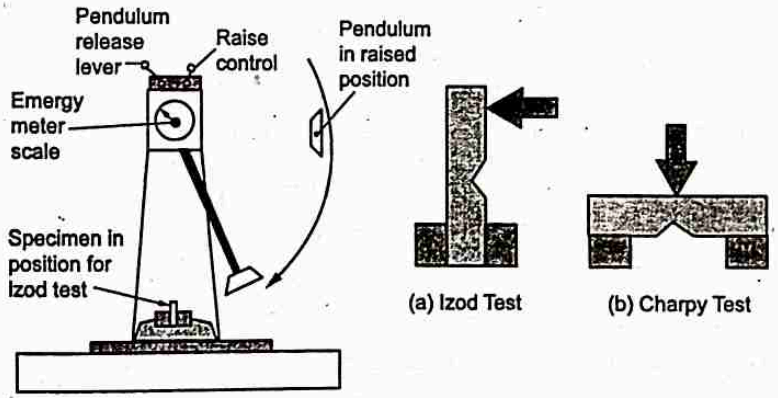


Fig. 2.17. Pendulam impact machine and notch position

1. PRINCIPLE OF IMPACT TESTING

- The sample is placed into a holding fixture with the geometry and orientation determined by the shape of a pendulum is released from a known height so that it collides with the specimen with a sudden force.

- This collision between the weight and specimen generally results in the destruction of the specimen but the transfer of energy between the two is used to determine the fracture mechanics of the material.

2. WORKING

- The test piece is held in the anvil, prior to the release of the pendulum by are lease mechanisms from its fixed starting point.
- A Charpy V-notch specimen is placed across parallel jaws in the impact testing machine and for Izod test specimen is placed perpendicular to jaws.
- The pointer is set up to its maximum value (300 J).
- Initially before release, the pendulum will have a potential energy that depends on the weight of the pendulum and its height off all. Upon release, the pendulum strikes and breaks the test piece by a single blow, which consumes part of the energy of the pendulum depending on the toughness of the test material.
- The energy consumed to break the specimen is measured, usually in joule, from the position of a pointer on a dial attached to the impact tester.
- Observations of the energy absorbed are recorded.

3. APPLICATIONS OF IMPACT TEST

- The impact test also indicates the notch sensitivity of a material resulting from the presence of internal stress raisers such as grain boundary inclusions, internal cracks, and second phases.
- The impact test is often used to assess the transition temperature from the ductile to brittle state which occurs as the temperature is lowered.

4. ADVANTAGES

- The chief merit is that comparatively in expensive small-sized test specimens are used in these tests, which are also comparatively simple to carry out.
- The higher the impact value of a material is, the higher the toughness or tenacity of the material.

- Tests can easily be performed over a wide range of sub ambient temperatures.
- Tests can be applied to study and compare the effects of heat treatments and alloy additions on the notch tough-ness of a material.
- Tests are well suited for quality control and material acceptance purposes.
- The impact value can be used for determining the load bearing capacity of a material against momentary stress from impact strength and fracture energy.

5. DISADVANTAGES

- Impact tests are of little value for most wrought nonferrous metals such as aluminum, copper, and their alloys since there is no transition to brittle behavior.
- The main problem is that the results obtained from these tests cannot be readily used in design.
- The notched-bar impact properties are remarkably influenced by the size, shape and sharpness of the flaw.
- Variation in result if improper placement of the test piece in the impact tester.

2.13. COMPARISON BETWEEN IZOD IMPACT TEST AND CHARPY IMPACT TEST

Parameter	Izod impact test	Charphy impact test
Specimen position	Specimen held at vertical	Specimen held at horizontal
Point of strike	At Upper tip of specimen	At Point of notch but in opposite direction
Types of notch	V-notch	V-notch and U-notch
Type of hammer	Farming hammer	Ball pin hammer
Specimen dimension	75 × 10 × 10 mm	55 × 10 × 10mm

Parameter	Izod impact test	Charphy impact test
Notch face	Facing the striker, fastened in pendulum	Face is positioned away from the striker
Materials used	Plastics and metals	Metals
Holding	It imitates cantilever beam	It imitates simply supported beam
Temperature	It is largely affected by temperature changes	It shows minimum error to temperature changes
Calculation	The Izod impact value (J/m, kJ/m^2) is calculated by dividing the fracture energy by the width of the specimen.	The Charpy impact value (kJ/m^2) is calculated by dividing the fracture energy by the cross-section area of the specimen.
Energy	The fracture energy is determined from the swing-up angle of the hammer and its swing-down angle	The fracture energy is determined from the swing-up angle of the hammer and its swing-down angle.

2.14. BEND TEST

- Bending tests is standard test method for material of smooth bars like flat metal spring, concrete, natural stone, wood, plastics, glass and ceramics.
- It also called as flexural test (particularly to evaluate tensile strength of brittle material which is difficult to under estimate in uniaxial tension test).

1. PRINCIPLE

- Bending tests are conducted by placing a length of material across a span and pushing down along the span to bend the material causing a concave surface or a bend to form without the occurrence of fracture and are typically performed to determine the ductility or resistance to fracture of

that material, the elastic modulus of bending, flexural stress, and flexural strain of a material.

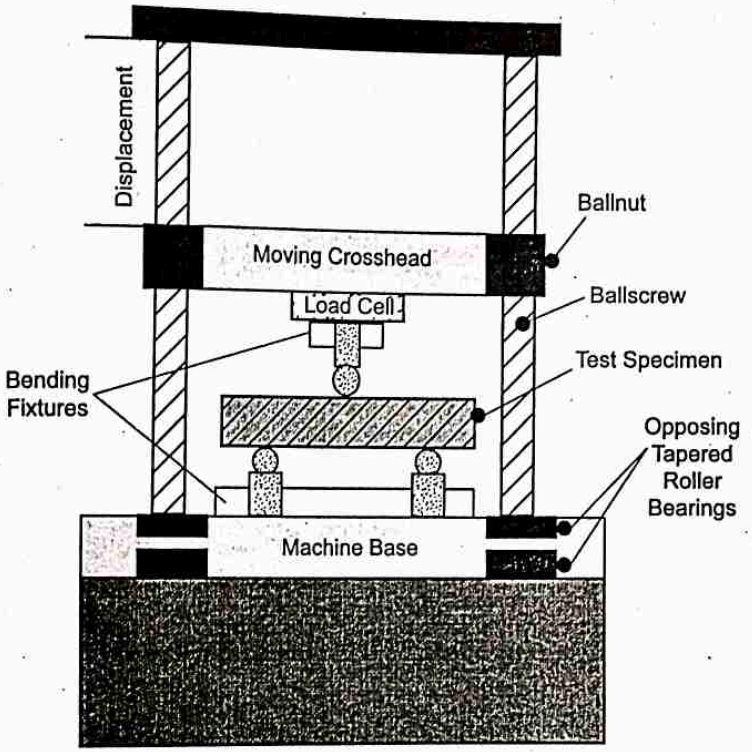


Fig. 2.18. Bending test equipment

METHODS OF BEND TEST BASED ON LOAD POSITION

- **Single-point loading at the free end of a cantilever beam** - A cantilevered beam is fixed at one end and the other end is free. In cantilever beam tests, a load is applied to the free end of the beam until failure occurs
- **Centre point loading (or) Three-point bending test** - Three-point bend fixtures configuration of flexural strength testing, where a specimen is loaded at a location midway between two supports bearings
- **Four-point bending test**-Four-point bend fixtures configuration of flexural strength testing where a specimen is symmetrically loaded at two locations

that are situated one quarter of the overall span, away from the outer two support bearings.

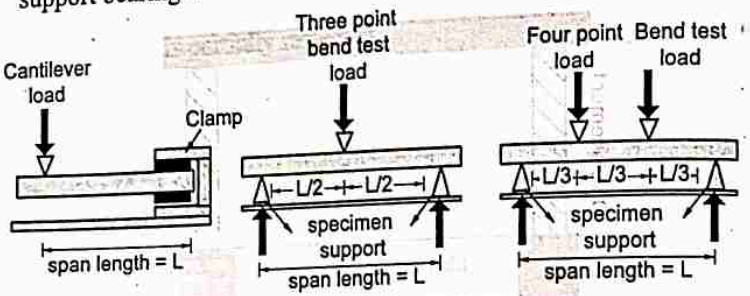


Fig. 2.19. Cantilever, 3-point and 4point Load position

Bend Testing Universal Testing Systems

- Material testing systems accurately and reliably measure the flexural properties of metals, concrete, plastics, medical devices and other products and components. The machines can calculate flexural modulus, flexural strength, and yield point at maximum capacities.

Bend Fixtures

- Bend fixtures are used to determine the flexural properties of rigid and semi-rigid materials. They are available in a variety of capacities, spans, and support diameters and widths. It consists of default adjustable load pointer based on loading position.

3. WORKING

- The bending fixture is supported on the platform of hydraulic cylinder of the UTM.
- A loading beam that rests on two rollers on the top of beam to be tested is used to apply the loads. Accurate spacing of the supports and loading points is necessary.
- A load is applied to the loading beam accurately at the mid-point between its two supporting rollers for three-point loading (or) four-point loading.
- The supports are generally knife-edge or convex. The load applicator is a rounded knife-edge with an included angle of 60°, applied either at mid span (for three-point testing) or symmetrically placed from the supports (for four-point testing).

- These rollers in turn must be spaced accurately at equal distances from the supporting rollers for the beam to be tested.
- If the distance between the supporting rollers of the test-beam is L; the supporting rollers of the loading beam are often located at L/3 or L/4 distances from the test-beam supports, although any equal location distances can be used.
- Load and either deflection or strain are usually recorded in the test.
- Using this method, a beam mounted on supports is studied under a applied force to the beam.
- The bending test demonstrates the relationship between the load of a bending beam and its elastic deformation.
- The loading is held in the middle cross head.
- At a particular load the deflection at the center of the beam is determined by using a dial gauge.

4. DERIVED PROPERTIES

(*a*) Flexural strength, also known as modulus of rupture or bend strength or transverse rupture strength is a material property, defined as the stress in a material just before it yields in a flexure test.

1. For three point bending test (rectangular cross section)

$$\sigma_f = \frac{3FL}{2bd^2}$$

2. For four point bending test where the loading span is 1/2 of the support span (rectangular cross section)

$$\sigma_f = \frac{FL}{bd^2}$$

3. For four point bending test where the loading span is 1/3 of the support span (rectangular cross section)

$$\sigma_f = \frac{3FL}{4bd^2}$$

σ_f = Stress in outer fibers at midpoint, (MPa)

= load at a given point on the load deflection curve, (N)

L = Support span, (mm)

b = Width of test beam, (mm)

d = Depth or thickness of tested beam, (mm)

(b) Deflection is the degree to which a element is displaced under a flexural load (due to its deformation).Deflection for three point bending test,

$$\delta_C = \frac{FL^3}{48EI}$$

E = Modulus of Elasticity (or) Young's modulus

I = Area moment of inertia of cross section

4. FACTORS AFFECTING THE MODULUS OF RUPTURE

1. Types of loading
2. Length of span
3. Shape of the cross-section of a beam
4. Cross-sectional dimensions of a beam
5. Rate of loading, i.e. speed of testing

5. ADVANTAGES

- Simpler sample geometries.
- Minimum sample machining is required.
- Simple test fixture.
- Possibility to use as-fabricated materials
- The bend test is a simple and inexpensive qualitative test that can be used to evaluate both the ductility and soundness of a material.
- It is often used as a quality control test for butt-welded joints, having the advantage of simplicity of both test piece and equipment.
- The main advantage of a three point flexural test is the ease of the specimen preparation and testing.

6. DISADVANTAGES

- The results of the testing method are sensitive to specimen and loading geometry and strain rate.
- More complex stress distributions through the sample.

2.15. SHEAR TEST

- In Shear test, the shear force is the load that causes two contiguous parts of the body to slide relative to each other in a direction parallel to their plane of contact.

1. PRINCIPLE

- Shear strength measures a material's ability to resist forces that cause the material to slide against it. The Specimen is loaded in shear fixtures, load in applied perpendicular to specimen through plunger. The phenomenon of shear applies through the shear fixtures (coupling device) is known as shear test.

2. TYPES OF SHEAR TEST

1. Single shear test
2. Double shear test

3. COMPONENTS

- Universal testing machine
- Vernier caliper
- Shear fixtures

4. SHEAR FIXTURES

- Two coupling braces which is used for both single or double shear connection. Both are at similar position with certain distance apart. For single shear, specimen is routed to single brace and for double shear, specimen is routed fully.

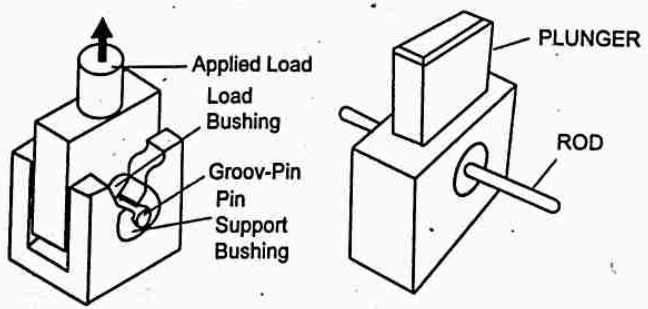


Fig. 2.20. Layout of shear fixtures

5. SINGLE & DOUBLE SHEAR TEST

- In the double-shear method, the specimen is sheared off at two cross sections. In the single-shear process, the specimen only shears away at one cross section. Calculating the shear strength in the two processes differs in the cross-sectional area to be applied. The shear strength determined in the shear test is important in the design of bolts, rivets and pins, as well as for calculating the force required for shears and presses.

6. WORKING

- The diameter is measured using the vernier caliper
- Mount the shear fixtures on UTM and load the specimen in shear fixture accordance to need of shear test. Operate (push) buttons for driving the motor to drive the pump.

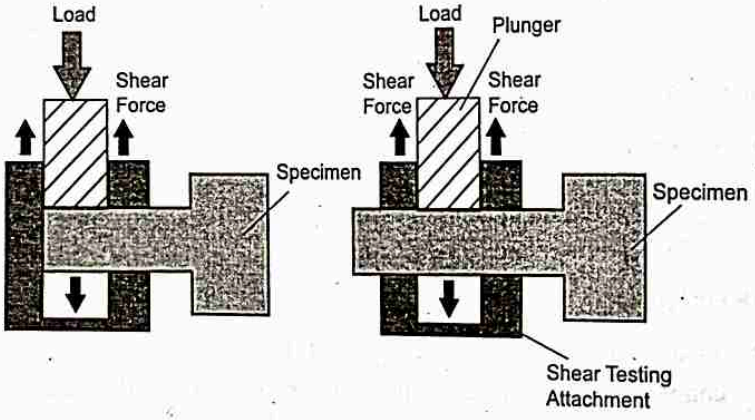


Fig. 2.21. Single and double shear loading

- Gradually move the head control level direction till the specimen shears. Take the load at which the specimen shears.

$$\text{Shear Stress } (\tau) = \frac{F}{2A}$$

where, F = Force at breaking shear

A = Shearing Surface

7. ADVANTAGES

- The time of testing is small

- The adhesion capacity also can be found.
- Result evaluations is direct manner
- No skilled labor is requires

8. LIMITATIONS

- Due to limitation in diameter of hole, size of testing rod is limited.
- If Error in measurement of the diameter of the specimen gives large variation in results.
- For sheet metal, thickness is restricted.

2.16. CREEP

- The material is stressed with static load at increased temperature. The material fails before yield point and without an increase in load lead to a slow but steady irreversible plastic deformation, also known as creep. After a sufficiently long, even load time, this leads to fracture of the specimen.
- The creep is viscoelasticity process.
- Stress relaxation is closely related to creep. In stress relaxation, the stresses resulting from loading of a structural component decrease in magnitude over a period, even though the dimensions of the component remain constant.

1. CREEP TEST

- In the creep rupture test, a specimen is subjected to load at constant stress and constant temperature.
- This experiment is performed multiple times with different temperature, but always at static loading. The plastic deformations are measured in continuous intervals.
- All measured values can then be transferred to a creep diagram. The measured elongation shows a characteristic curve, which is known as the creep curve.
- The creep rupture test determines the characteristic values for the creep strength and the various strain values

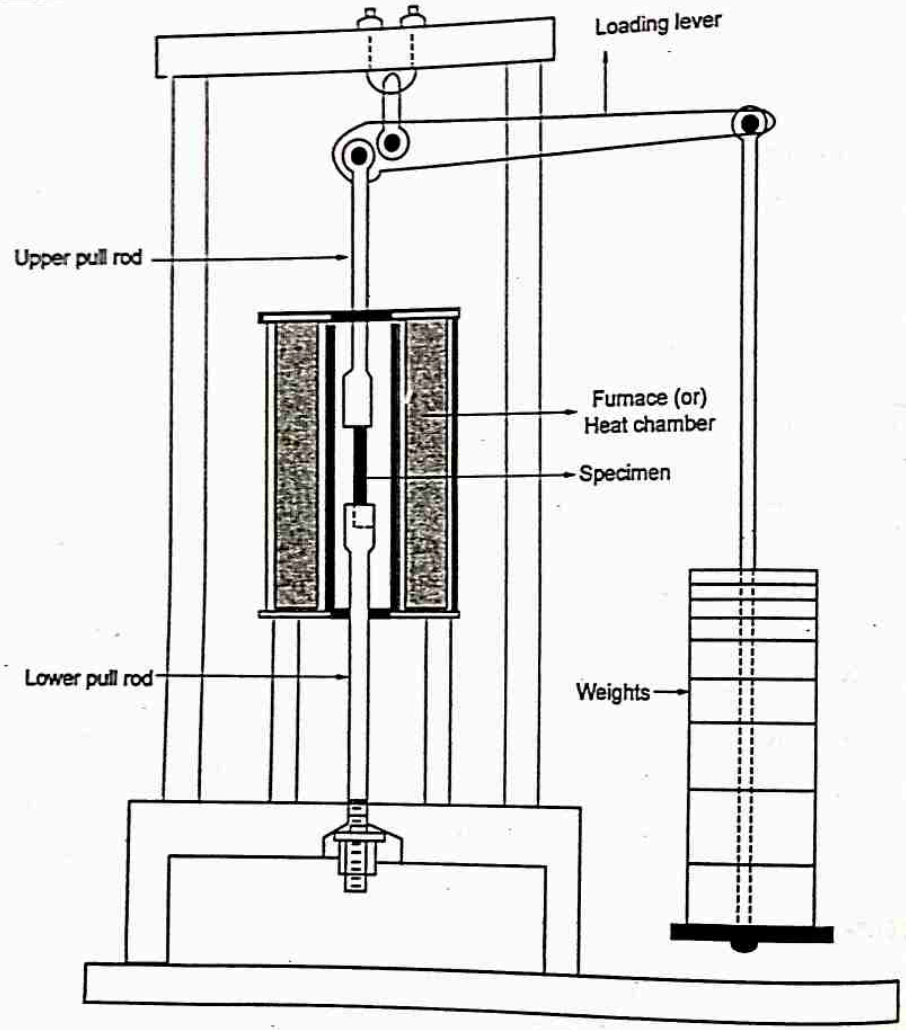


Fig. 2.22. Constant loading creep

2. Procedure

- Mark the sample for the reduced gauge length (uniform width), **Heating Chamber** is what surrounds the object and maintain the temperature that the object is subjected to gradually elevated temperature.
- Measure the dimensions (gauge length and width by Vernier caliper and thickness by screw gauge) of the given lead sample.
- Fix the ends of the sample up to mark in the jaws of the machine

- Adjust the extensometer position on the load such that needle on dial is at '0' position
- The value of change dimension with respect to time for increased temperature with static loading is noted until the specimen fails.

The final result will be done in following steps,

- Calculation of stress
- Plot strain vs. time
- Calculate the creep rate as a function of time and identify the various stages of creep
- Finding the minimum creep rate at each stage

3. Stages of Creep

(a) Primary Creep

- Primary creep or transient creep the initial creep stage where the slope is rising rapidly at first in a short amount of time.
- After a certain amount of time has elapsed, the slope will begin to slowly decrease from its initial rise.

(b) Secondary creep

- Steady-state creep or secondary creep after the primary creep, the creep rate reaches essentially a steady state, in which the creep rate changes little with time. This region of approximately constant creep rate.
- During this stage, the steady state is achieved because of an approximate balance between two opposing factors: the strain hardening that tends to reduce the creep rate and the softening or recovery process that tends to increase it.
- The creep rate is constant so the line on the curve shows a straight line that is a steady rate.

(c) Tertiary Creep

- The last stage of creep when the object that is being subjected to pressure is going to reach its breaking point.

- In this stage, the object's creep continuously increases until the object breaks. The slope of this stage is very steep for most materials. During this stage, high stresses or/and at high temperatures.
- The creep rate is greater and increases continuously till the material undergoes fracture.
- Tertiary creep occurs when the effective cross-sectional area of the specimen is reduced remarkably either due to localized necking or internal void formation.

(d) Ultimate ductile failure

- It which takes place when the crack becomes sufficiently long so that the remaining cross-section can no longer sustain the applied load.

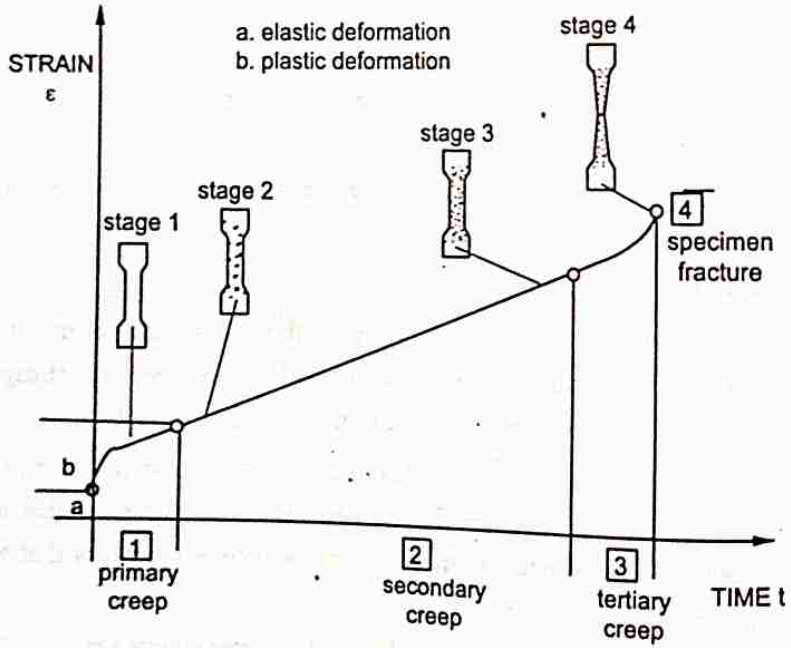


Fig. 2.23. Stages of creep

4. MECHANISM OF DEFORMATION

- **Dislocation creep:** At high stresses (relative to the shear modulus), creep is controlled by the movement of dislocations. That involves dislocation glide and climb.

- **Diffusional creep :** That involves stress-assisted diffusional flow of atoms and vacancies
- **Grain-boundary sliding:** It is a shear process occurring in the direction of grain boundary, causing the movement of grains relative to each other in polycrystals.

5. METHODS TO REDUCE CREEP

- Solid solution strengthening
- Particle dispersion strengthening
- Precipitation hardening
- Increasing grain size

6. ADVANTAGES

- To determine the stability of a material and its behaviour when it is put through ordinary stresses like crcep test.
- The understanding of their properties and advantages of one material's use over another.

7. LIMITATION

- Intermediate stopping of instrument cause error in result
- The size must be precise or else it cause error
- As costliness and long testing times.
- It also demands large sample material out take which often involve weld repair.

2.17. FATIGUE TEST

- When the component is subjected to repeated cyclic stress (due to rotation, bending or vibration) leads to failure even though the stress below the yield strength of material.
- This progressive failure of the material at a stress much lower than that required to cause fracture on a single application of load is called a fatigue failure.

1. PRINCIPLE

- A fatigue test is used for the determination of the maximum load that a sample can withstand for a specified number of cycles. Cyclic fatigue tests produce repeated loading and unloading in tension, compression, bending, torsion or combinations of these stresses.
- These tests are used to generate fatigue life and crack growth data, identify critical locations or demonstrate the safety of a structure that may be susceptible to fatigue.

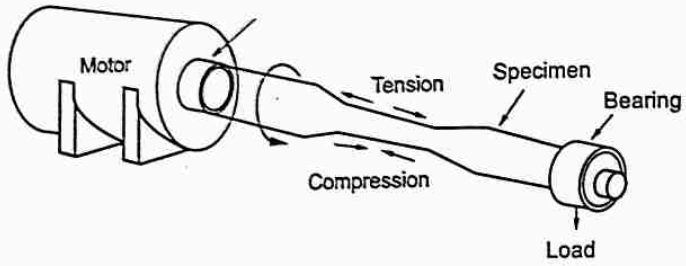


Fig. 2.24. The cantilever type fatigue testing

2. METHODS TO DETERMINE FATIGUE LIFE

Following methods to determine the fatigue life of a material

(a) The stress-life method: A mechanical part is often exposed to a complex, often random of sequence of loads values of large and small range.

Types of stress life method

- Rainflow analysis
- Fatigue damage spectrum
- S-N curve
- Miner's rule.

(b) The strain-life method: When strains are no longer elastic, such as in the presence of stress concentrations, the total strain can be used instead of stress as a similitude parameter. This is known as the strain-life method

(i) The crack growth method: An estimate of the fatigue life of a component can be made using a crack growth equation by summing up the width of each increment of crack growth for each loading cycle

(ii) Probabilistic methods: It is based on either life or crack growth methods.

WORKING

- In the fatigue strength test, a rotating, cantilever-mounted specimen is subjected to a bending moment.
- In the cylindrical specimen, this creates an alternating stress due to rotary bending. After a certain number of load cycles, the specimen fractures because of material fatigue.
- Polish the sample surface as smooth as possible and observe for any surface defects and deep scratch/machining marks. Reject the sample if you find any defects.
- Measure dimensions of the given specimen of mild steel.
- The equipment is mounted with motor driven chuck. The load is suspended in the opposite end of motor.
- Fit the specimen is in the sample holder such that it passes through the opening provided in the rod on which the loads are seated.
- After fitting the sample, keep the desired load on the seat .
- At the top end the tension is applied and bottom end compression is provided with rotating of specimen.
- Switch on the instrument to conduct the fatigue test by rotating specimen at 90° with compression and tension and rotate to 180° with same cycle loads. Repeat the cycle of stress upto of on a rotating shaft in four-point bending.
- Record the time for the failure, when it occurs.
- Note the appearance of the fractured surface in each case.

4. STAGES IN FATIGUE FAILURE

(a) Stage 1-Crack initiation

- Crack initiates at which are pre-existing flaws or generated during the cyclic straining process.
- The minor crack initiates at the surface of material which occurs after loading for a while. The crack develops by grain to grain of material. The growth of crack is slow in this stage.

(b) Stage 2-Crack propagation

- Slip-band crack growth, where the initial crack grows along slip planes, i.e. planes of high shear stress.
- The minor crack is gradually propagates to major one as cyclic stress to continues. This Crack propagation in a specimen is also determined by the grain size. The larger the grains are, the rougher the crack surfaces will be. In case of unloading, these stresses cause an opposite plastic deformation and close the crack even at the crack tip.

(c) Stage 3-Sudden fracture

- Finally the sudden fracture occurs in material due to stress that is not supported by the materials section planes of high tensile stress, where a well-defined crack propagates in a direction normal to the maximum applied tensile stress.

5. S-N CURVE

- Fatigue test involve testing the specimens under various cycles of stress, usually in a combination of tension, bending and rotation.
- The test is conducted with variation of stress amplitudes (S); the number of cycles (N), the point of total failure in the specimen is recorded.
- S-N curves are derived from tests on samples of the material to be characterized (often called coupons) where a regular sinusoidal stress is applied by a testing machine which also counts the number of cycles to failure. This process is sometimes known as coupon testing.
- Stress amplitude is defined as the maximum stress, in tension and compression, to which the specimen is subjected.
- The resulting diagram is called a stress-cycle (or S-N) diagram (sometimes also stress-life or Wöhler diagram)
- To determine an S–N curve, a group of fatigue specimens is tested at different stress levels and at each of the several stress levels the loaded specimen is rotated until it fractures.
- The sudden bend of curve indicates the endurance limit. The maximum endurance limit for S-N curve is nearly 108 cycles. S-N curve calculated in semi log graph

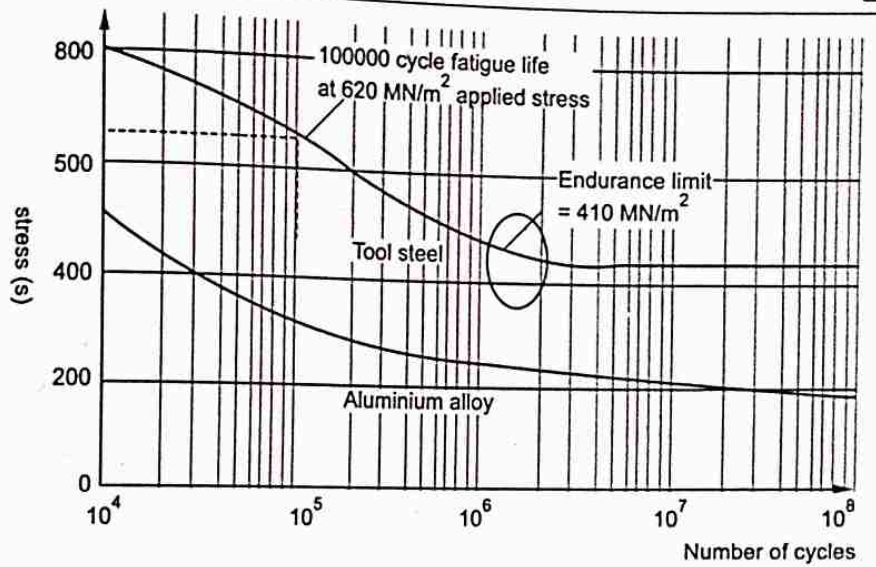


Fig. 2.25. S-N curve for steel and aluminum alloy

6. DERIVED PROPERTIES

- **Maximum stress** is calculated by

$$\sigma = \frac{10.18\, lF}{d^3}$$

σ = Maximum fatigue stress
l = Length of specimen
F = Fatigue failure load
d = Diameter of rod

- **Endurance limit (fatigue limit)** is the maximum fatigue stress applied to material without failure. It is 50% of fatigue failure load which is preferred for design criteria
- **Fatigue life** is the survival of component on particular stress.
- The fatigue strength defines the load limit up to which a material that is loaded dynamically withstands without breaking.
- **Endurance ratio** is ratio of Endurance limit and Tensile strength. It is nearly half of tensile strength. It allows calculating the fatigue strength from the tensile strength. For most materials it is in the range of 0.4-0.5.

- **Stress amplitude** (σ_a) is half the difference between the maximum stress (σ_{max}) and minimum stress (σ_{min}).

$$\sigma_a = \frac{\sigma_{max} - \sigma_{min}}{2}$$

- **Mean Stress** (σ_m) is half the sum between the maximum stress (σ_{max}) and minimum stress (σ_{min}).

$$\sigma_m = \frac{\sigma_{max} + \sigma_{min}}{2}$$

- **Stress ratio R** is the defined as ratio of minimum stress (σ_{min}) and maximum stress (σ_{max}).

$$R = \frac{\sigma_{min}}{\sigma_{max}}$$

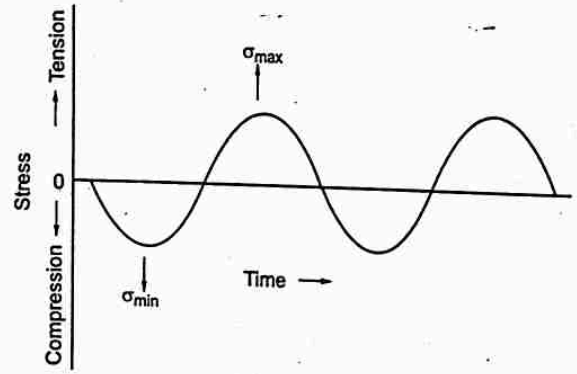


Fig. 2.26. Compression and tension flow cycle

7. TYPES OF FATIGUE CYCLE

(a) High-cycle fatigue

- When fatigue tests are conducted with a fixed cycle of load or stress limits, it is called a stress-controlled fatigue. It is a high-cycle fatigue (often simply termed as fatigue) because fatigue failure takes place at high numbers of stress cycles, usually more than 10^4 cycles (about 10^4 to 10^8 cycles). Since majority of the fatigue failures in service occurs at $N > 10^4$ cycles, the fatigue in the high-cycle region (stress-controlled fatigue) has received an engineering importance.

- A load-controlled servo-hydraulic test rig is commonly used in these tests, with frequencies of around 20–50 Hz. Other sorts of machines - like resonant magnetic machines - can also be used, to achieve frequencies up to 250 Hz.

6. Low-cycle fatigue

- When fatigue tests are conducted with a fixed cycle of elastic plus plastic strain limits, it is called a strain-controlled fatigue or a low-cycle fatigue because fatigue failure takes place when the number of cycles necessary to cause fatigue failure, $N < 10^3$ cycles. Testing is conducted with constant strain amplitudes typically at 0.01 – 5 Hz.

8. FACTORS INFLUENCE THE FATIGUE LIMIT

- Mean stress
- Size of specimen
- Surface condition
- Stress ratio
- Corrosion
- strain range
- Surface finish and quality
- Surface treatments
- Load sequence and overload
- Temperature

9. METHODS TO REDUCE FATIGUE

- Changes in the materials
- Peening
- Deep cryogenic treatment
- Re-profiling.

10. ADVANTAGES

- Demonstrate the safety of a structure that may be susceptible to fatigue.
- Generate fatigue data.

- Identify critical locations.
- Fatigue tests can also be used to determine the extent that widespread fatigue damage may be a problem.

11. LIMITATIONS

- It fails to recognize the probabilistic nature of fatigue and there is no simple way to relate life predicted by the rule with the characteristics of a probability distribution.
- It does not consider the effect of an overload or high stress which may result in a compressive residual stress that may retard crack growth.

TWO MARK QUESTIONS WITH ANSWERS

1. ***What is advantage of charpy test on izod test?***
   - More suitable for low-temperature tests which must be completed within a few seconds from the time of removal of the test piece from the coolant. This is due to easier placement of the Charpy specimen in the tester compared to the Izod.
   - Free from compressive stresses around the notch, while gripping of the Izod specimen inside the clamp device produces the compressive stresses around the notch.
2. ***Define true stress–strain and engineering stress–strain.***
   - True Stress is Stress value obtained by dividing the instantaneous area into applied load
   - True Strain is Provides a more realistic assessment of "instantaneous" elongation per unit length

$$\varepsilon = \int_{L_0}^{L} \frac{dL}{L} = ln\frac{L}{L_0}$$

   - **Engineering Stress:** Stress (nominal stress) is defined as the ratio of the applied load to the original cross-sectional area of the specimen

Stress (σ) = applied load / original cross-sectional area

- **Engineering Strain**: Strain is defined as change in length to original length

Strain (e) = change in length / original length

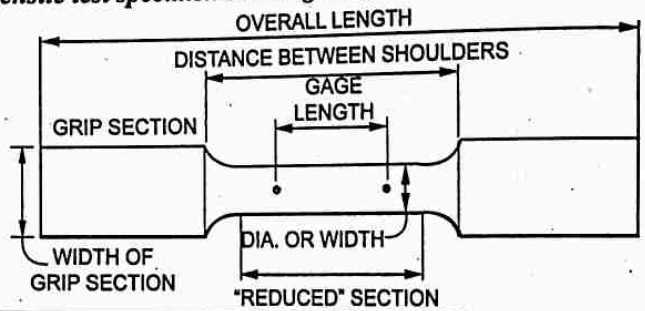
3. ***What are the major mechanical properties of material and its test to determine it?***

Mechanical Property	Destructive Testing Method
❖ Elasticity, Plasticity	❖ Tensile Test, Compression Test, Bending Test, Torsion Test
❖ Stiffness, Material Behaviour Under Static Load	
❖ Creep Behaviour	❖ Creep Rupture Test
❖ Hardness	❖ Brinell, Rockwell, Vickers
❖ Toughness	❖ Impact Test
❖ Fatigue Behaviour, Fatigue Strength	❖ Wöhler Fatigue Test

4. ***What are various failure modes of materials?***
   - Material failure is the loss of load carrying capacity of a material unit. The material failure happens due to two major phenomena,
     - Deformation failure
     - Fracture failure
5. ***How the hardness influence the material properties?***
   - 'Hardness' is a structure-sensitive mechanical property of materials, primarily associated with the surface. It is the resistance of a material to permanent or plastic deformation of its surface.
6. ***List out the different types of Hardness testing machines.***
   - (*i*) Brinell; (*ii*) Meyer; (*iii*) Vickers (macro - and micro-hardness); (*iv*) Rockwell (regular and superficial); (*v*) Knoop (micro hardness); (*vi*) Nano hardness (mostly by Vickers and Berkovich indenters)
7. ***What property of metal does the impact test measure? Give its significance.***
   - The purpose of an impact test is to determine the ability of the material to absorb energy during a collision.

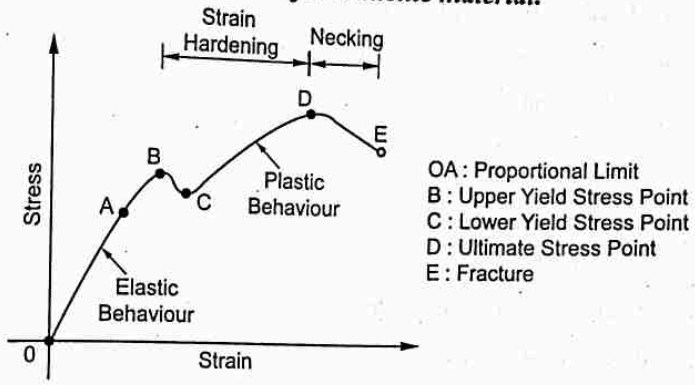
- This energy may be used to determine the
  - Toughness
  - Impact Strength
  - Fracture Resistance
  - Impact resistance or fracture resistance of the material

8. *Sketch a tensile test specimen showing all dimensions in inch.*



All values in inches	Plate type (1.5 in. wide)	Sheet type (0.5 in. wide)	Sub-size specimen (0.25 in. wide)
Gauge length	8.00 ± 0.01	2.00 ± 0.005	1.000 ± 0.003
Width	1.5 + 0.125 – 0.25	0.500 ± 0.010	0.250 ± 0.005
Thickness	0.188 ≤ T	0.005 ≤ T ≤ 0.75	0.005 ≤ T ≤ 0.25
Fillet radius (min.)	1	0.25	0.25
Overall length (min.)	18	8	4
Length of reduced section (min.)	9	2.25	1.25
Length of grip section (min.)	3	2	1.25
Width of grip section (approx.)	2	0.75	3/8

9. *Neatly draw stress–strain curve for a ductile material.*



10. *Give the dimensions of Charpy and Izod impact test samples.*

Parameter	Izod impact test	Charphy impact test
Specimen dimension	Length-75 mm Width-10 mm Thickness-10 mm	Length-55 mm Width-10 mm Thickness-10 mm

11. *What are advantages made the choice of Brinell hardness test?*

- A choice can be made between a large numbers of test forces.
- The influence of surface scratches and roughness will be less in the Brinell test than other hardness tests.
- The specimen surface can be rough.
- Suitable for hardness tests on large blanks such as forged pieces, castings and hot-rolled etc
- Measurement is usually not affected by movement of the specimen

12. *What kind of indenter suitable for Vickers hardness?*

- It is made of diamond in the form of a square-based pyramid with an included angle of 136° between opposite faces.

13. *What are properties can be determined from tensile testing?*

- The tension test is the most common method for determining the mechanical properties of materials, such as strength, ductility, toughness, elastic modulus, and strain- hardening capability.

14. ***Define strain hardening and proof stress.***

- **Strain hardening**: This increase in the tensile strength of the material is due to strain hardening which is due to the increased dislocations interactions during the deformation of the tensile test. This is called Strain - hardening.
- **Proof Stress:** The stress that causes a percentage increase in gauge length. It can be found by drawing a line parallel to the straight part of the graph. A value can be taken from the vertical axis.

15. ***Write the classification of impact test based on load application.***

- Impact test classified based on load application applied by means of dropping weight, a swinging pendulum and a rotating flywheel.

16. ***What is the basic principle involved in Charpy and Izod impact test?***

- The Charpy V- notch impact test is the most common fracture toughness test. A notched specimen is broken by a swinging pendulum and the amount of energy required to break the specimen is recorded.
- The Izod impact strength test is a standard method of determining the impact resistance of materials. A pivoting arm is raised to a specific height (constant potential energy) and then released. The arm swings down hitting a notched sample, breaking the specimen.

17. ***Compare Charpy and Izod impact test based on specimen position & point of strike?***

Parameter	Izod impact test	Charphy impact test
Specimen position	Specimen held at vertical	Specimen held at horizontal
Point of strike	At Upper tip of specimen	At Point of notch but in opposite direction

18. ***Define deflection.***

- **Deflection** is the degree to which a element is displaced under a flexural load (due to its deformation).Deflection for three point bending test,

$$\delta_C = \frac{FL^3}{48EI}$$

E = Modulus of Elasticity (or) Young's modulus

I = Area moment of inertia of cross section

19. ***What is modulus of rupture in bending nature?***

- Flexural strength, also known as modulus of rupture or bend strength or transverse rupture strength is a material property, defined as the stress in a material just before it yields in a flexure test.
- For three point bending test (rectangular cross section)

$$\sigma_f = \frac{3FL}{2bd^2}$$

- For four point bending test where the loading span is 1/2 of the support span (rectangular cross section)

$$\sigma_f = \frac{FL}{bd^2}$$

20. ***What is mean by shear relaxation in creep mechanism?***

- Stress relaxation is closely related to creep. In stress relaxation, the stresses resulting from loading of a structural component decrease in magnitude over a period, even though the dimensions of the component remain constant.

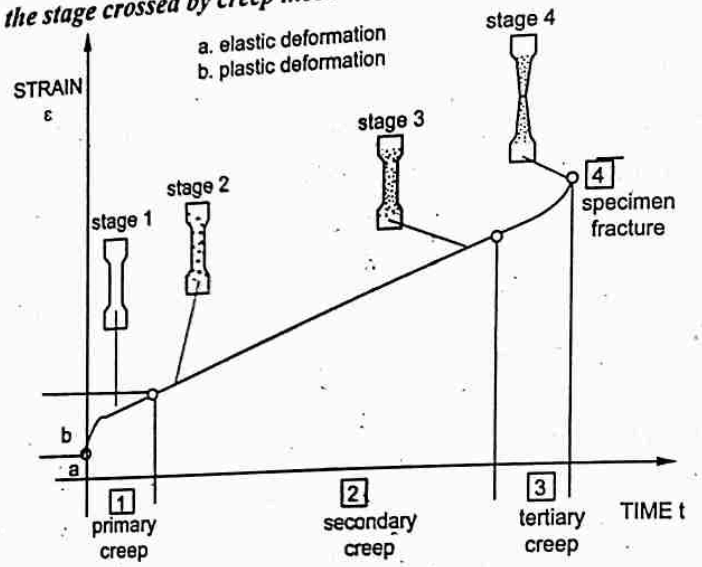
21. ***How the steady state creep mechanism will works?***

- Steady-state creep or secondary creep after the primary creep, the creep rate reaches essentially a steady state, in which the creep rate changes little with time. This region of approximately constant creep rate. The steady state is achieved because of an approximate balance between two opposing factors: the strain hardening that tends to reduce the creep rate and the softening or recovery process that tends to increase it.

22. ***How will control the creep?***

- Solid solution strengthening
- Particle dispersion strengthening
- Precipitation hardening
- Increasing grain size

23. *Sketch the stage crossed by creep mechanism.*



24. *Define the property of fatigue.*

- When the component is subjected to repeated cyclic stress (due to rotation, bending or vibration) leads to failure even though the stress below the yield strength of material.

25. *What is principle involved in fatigue testing?*

- A fatigue test is used for the determination of the maximum load that a sample can withstand for a specified number of cycles. Cyclic fatigue tests produce repeated loading and unloading in tension, compression, bending, torsion or combinations of these stresses.

26. *What are the methods available to calculate the fatigue life?*

- The stress-life method
- The strain-life method
- The crack growth method
- Probabilistic methods

27. *What is role of SN curve in fatigue mechanism?*

- S-N curves are derived from tests on samples of the material to be characterized, where a regular sinusoidal stress is applied by a testing machine which also counts the number of cycles to failure.

28. *Define Endurance limit.*

- Endurance limit (fatigue limit) is the maximum fatigue stress applied to material without failure. It is 50% of fatigue failure load which is preferred for design criteria

REVIEW QUESTIONS

1. State the working principle of the machine used for tension test. What care should be taken while performing a test on UTM?

   Ans: Refer Section No. 2.10 **Page No.** 2.26

2. Explain the various mode load application in the Rockwell hardness test.

   Ans: Refer Section No. 2.4 **Page No.** 2.15

3. Explain the working principles of machines used to conduct Charpy and Izod impact test. How specimens are put–up in both the tests? Why?

   Ans: Refer Section No. 2.12 **Page No.** 2.33

4. Discuss the factors considered for selection of hardness testing machine. What care must be taken while selecting specimens for hardness test?

   Ans: Refer Section No. 2.2 **Page No.** 2.19

5. Sketch the various types of fatigue cycles. How is S–N curve constructed? Explain the significance of endurance limit.

   Ans: Refer Section No. 2.17 **Page No.** 2.47

6. With a neat sketch, explain the various stages of creep curve.

   Ans: Refer Section No. 2.16 **Page No.** 2.43

7. What are properties arrived from the bending test? How do you relate with failure of section?

   Ans: Refer Section No. 2.14 **Page No.** 2.36

8. What are the test available for testing following material and explain with working procedure

   - Plastic

- ❖ Wood
- ❖ Steel Plate

Ans: Refer Section No. 2.14, 2.15 **Page No.** 2.36, 2.41

9. What are the various destructive tests available and which is more suitable to the hardness of material?

Ans: Refer Section No. 2.2 **Page No.** 2.4, 2.6

10. What are major contrasts of nature between izod and charpy test?

Ans: Refer Section No. 2.11 **Page No.** 2.35

11. How could you determine the fatigue life of material with the cyclic stresses?

Ans: Refer Section No. 2.17 **Page No.** 2.47

12. In the steel industry, iron rod is manufactured. Now, the iron rod is need to quality check. What is the quickest test available for testing various properties?

Ans: Refer Section No. 2.10 **Page No.** 2.26

13. In the construction site of steel cell phone tower, the quality engineer need to the check bold quality used for connection purpose. What is better test used for checking bold that used in connection? Explain with experimental procedure with advantages and limitation.

Ans: Refer Section No. 2.15 **Page No.** 2.41

□□

UNIT III

NON DESTRUCTIVE TESTING

SYLLABUS

Visual inspection, Liquid penetrant test, Magnetic particle test, Thermography test – Principles, Techniques, Advantages and Limitations, Applications. Radiographic test, Eddy current test, Ultrasonic test, Acoustic emission- Principles, Techniques, Methods, Advantages and Limitations, Applications.

3.1. OVERVIEW OF NDT

- ❖ Non-destructive testing (NDT) is a testing and analysis technique used by industry to evaluate the properties of a material, component, structure or system for characteristic differences or defects and discontinuities without causing damage to the original part.
- ❖ NDT also known as non-destructive examination (NDE), non-destructive inspection (NDI) and non-destructive evaluation (NDE).

1. IMPORTANCE OF NDT

- ❖ To Accident prevention and to reduce cost.
- ❖ For routine or periodic determination of quality of the plants and structures during service.
- ❖ To determine acceptance to a given requirement.
- ❖ To give information on repair criteria.
- ❖ To ensure product reliability.
- ❖ To ensure the safety of operation.
- ❖ To ensure customer satisfaction and to maintain the manufacturer's reputation.
- ❖ To control manufacturing processes and lower manufacturing costs.

- To maintain uniform quality level.

2. ADVANTAGES

(i) Reusable

- There are a number of distinct advantages, the most obvious of which is that the pieces being tested are left undamaged by the process, allowing for an item to be repaired rather than replaced should any problems be found.

(ii) Safe

- It is also a very safe testing method for operators, with most techniques being harmless to humans, although some types of test - such as radiographic testing - still need to be conducted under strict conditions.
- This testing technique can also help prevent injury or fatalities by ensuring structures, components and machinery is safe.
- This testing technique also offers operators peace of mind, knowing that equipment is functioning as it should, preventing future accidents and determining any measures that can be taken for life extension.

(iii) Accurate

- Non-destructive testing is also a very accurate way of inspection since the tests are repeatable and a number of tests can be used together to correlate results.

(iv) Cost effective

- These testing methods are also economical. Unlike destructive testing, NDT is cost effective as it can prevent the need to replace an item before malfunction occurs without destroying the piece itself.

(v) Quality control

- It is also useful for testing of welds and verification of welding procedures to ensure that a welding process has been completed to the correct specification within the bounds of quality control, for example to make sure that the base metal has reached the correct temperature, cooled at the specific rate and that compatible materials have been used to prevent welding defects.

4. STAGES OF WORKING IN NDT

1. Testing

- The first step testing involves in preparation of test material. With help of primary source (dye, ac source, loading), probe, receiver etc. the material is surveyed.

2. Recording & Reporting

- The most of output is displayed in computer.

3. Interpretation &Evaluation

- Based the output report, remedial action is take place and service life is also determined.

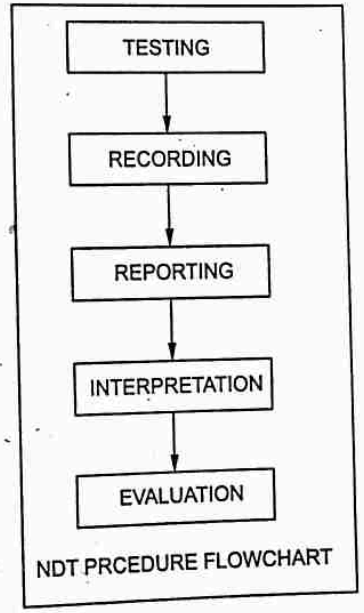


Fig. 3.1. NDT work flow chart

3.2. NON-DESTRUCTIVE TESTING METHODS

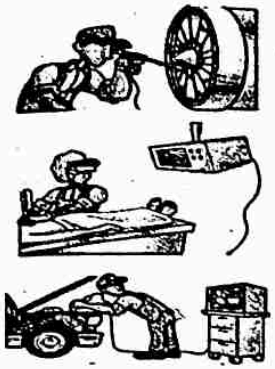
- Acoustic Emission Testing (AE)
- Electromagnetic Testing (ET)
- Ground Penetrating Radar (GPR)

- Laser Testing Methods (LM)
- Leak Testing (LT)
- Liquid Penetrant Testing (PT)
- Magnetic Flux Leakage (MFL)
- Magnetic Particle Testing (MT)
- Radiographic Testing (RT)
- Thermal/Infrared Testing (IRT)
- Ultrasonic Testing (UT)
- Visual Testing (VT)

Table 3.1. Comparison of NDT Capabilities

Technique	Capabilities	Limitations
Visual Inspection	Macroscopic surface flaws	Small flaws are difficult to detect, no subsurface flaws.
Radiography	Subsurface flaws	Smallest defect detectable is 2% of the thickness; Need radiation protection.
Dye penetrate	Surface flaws	No subsurface flaws; Not used for porous materials
Ultrasonic	Subsurface flaws	Material must be good conductor of sound.
Magnetic Particle	Surface / near surface and layer flaws	Limited subsurface capability, only for Ferromagnetic materials.
Eddy Current	Surface and near surface flaws	Difficult to interpret in some applications; only for metals.
Acoustic emission	Can analyze entire structure	Difficult to interpret, expensive equipment.

3.2.1. VISUAL TESTING (VT)



- Visual testing also known as visual inspection or Optical testing is one of the most common techniques which involve the operator looking at the test piece.
- This can be aided by the use of optical instruments such as magnifying glasses or computer-assisted systems (known as 'Remote Viewing').
- This method allows for the detection of corrosion, misalignment, damage, cracks and more.
- Visual testing is inherent in most other types of NDT as they will generally require an operator to look for defects.

1. PRINCIPLE

- The basic procedure used in visual NDT involves illumination of test specimen with light, usually in the visible region.
- The specimen is then examined with eye or by light sensitive device such as photo cells. The surface of the specimen should be adequately cleaned before being inspected.

2. ENVIRONMENT FOR VISUAL INSPECTION

- Inspection must take place in a clean, comfortable environment with adequate lighting. Lighting is very important and can greatly affect the results. It includes Natural daylight, Bright sunlight and Artificial light.

3. TYPES OF VISUAL INSPECTION

- Unaided Visual Inspection
- Aided Visual Inspection

(I) Unaided visual inspection

- It is also known as Direct Visual Inspection. It can be accomplished with the help of naked eye
- It can do without the help of optical aids.

(a) Defects can be detected are

- Absence of cracks
- Corrosion layer
- Surface porosity
- Misalignment of mated parts

(b) Aids of Unaided Visual Inspection

The eyes

- Human eye is the most valuable NDT Tool. Sensitivity of the human eye varies according to the light source.
- Human eye has an excellent visual perception. Yellow green light of wavelength 5560°A is the most suitable light for human eye at normal condition

(ii) Unaided Visual Inspection

- It is also known as indirect Visual Inspection.
- It can be accomplished with the help of some external equipment accomplished with direct visual. It can do with the help of optical aids.

Fig. 3.2. Aids of visual testing

Types of unaided viewing is,

- Direct viewing - Viewing of an object in the operator's immediate presence. This can be unaided or by using equipment
- Remote viewing - Viewing of an object not in the operator's immediate presence. This can only be done using special equipment

The commonly using visual aids are,

- **Magnifying glasses** - It consists of lens with magnification power which can used inspecting area of not accessible. Some types of magnifier incorporate a small battery-powered bulb to provide illumination of the test-surface.
- **Fillet weld gauge** - It usually uses a leaf-type fillet weld gauge to measure the size of fillet welds for standard size.
- **Microscopes** - Microscopes come in a wide variety of magnification ranges, microscope is a multiple element magnifier for proving high magnified image of small defect
- **Computer equipment (remote viewing)** - A modern videoscopes, due to their small size and flexibility, can provide access to internal areas inaccessible to Borescope.
- **Illuminated magnifier** - Inspection Magnifier is highly useful for inspection of small parts and also for online visual quality evaluation.
- **Holography** - Holography is name given to the method of obtaining an accurate 3-D image of a given object. It is used for the NDT of surfaces of highly complicated and precision components without the dis-advantages of having to use a high power microscope. It can provide a record of the image of an entire surface which can be readily compared with that of a standard defect free surface.
- **Borescope** - It is optical instrument for remote viewing of objects. Borescope can have various angles of view: 0° direct, 45° fore-oblique, 90° lateral and 110° retro. Borescope consist of precision illumination system. The size of the visual field usually varies with the diameter, for a given magnification system. The size of the visual field usually varies with the diameter, for a given magnification system.

- **Magnifying Mirrors** - When inspection is not easily accessible, a magnifying mirror can be used.
- **Periscope** - It is an instrument used for remote observation of inaccessible areas. In simple periscope, two right angle reflecting prisms are utilized in combination with a series of lenses.
- **Endoscope** - It is bit superior than Borescope. Magnification factor of 10X is obtained. Available up to smaller dia of 1.7 mm and length upto 100-150mm

4. MATERIAL FACTORS THAT AFFECT VISUAL TESTING

- **Surface Condition**
  - Cleanliness
  - Colour
  - Texture
- **Physical Conditions**
  - Specimen Condition
  - Shape and Size
  - Temperature
- **Environmental Factors**
  - Atmosphere
  - Humidity and Temperature
  - Safety
- **Physiological Factors**
  - Physical Comfort
  - Health , mental attitude, fatigue and test item position

5. ADVANTAGES

- Simple method to perform
- Examination can be performed quickly
- Low-cost method
- Minimal training

- Minimal equipment
- Virtually any component can be examined anywhere on the surface.
- Speed
- Applicability to irregular shapes
- Field mobility

6. DISADVANTAGES

- Inspector training necessary.
- Good eyesight required or eyesight corrected to 20/40.
- Can miss internal defects.
- Report must be recorded by inspector.
- Open to human error.
- Providing adequate viewing angles, sensitivity, resolution, and illumination may be costly.
- Visual testing requires a line of sight to the test surface and lighting adequate to detect and interpret anomalies of interest.
- Visual testing is sometimes limited to component geometry: size, contour, surface roughness, complexity, and discontinuity orientation.

7. APPLICATIONS

- Examining the surface condition of a component
- Examining alignment of mating surfaces
- Checking presence of leaks

8. EXAMPLE OF SOME APPLICATION VISUAL TESTING

Visual test of welds in connections

- In majority of industries for testing of welds, fillet weld gauge is used. The safety limit is checked in limits by using codes
- Surface is cleaned well to ensure free from rust, dirt etc.
- By inspecting irregularities, depth of penetration of weld and discontinuity the detected.

3.2.2. LIQUID PENETRANT TEST

- It also known as liquid penetrant inspection (LPI) or dye penetrant testing is based on the properties of surface wetting and capillary action, which causes a liquid to rise when confined to a small opening. After applying the penetrant and wiping away the excess, the penetrant that rises to the surface can indicate surface-breaking.
- Dye Penetrant Inspection (DPI), also called Liquid Penetrant Inspection (LPI) or Penetrant Testing (PT).
- It is used to detect any surface-connected discontinuities such as cracks from fatigue, quenching, and grinding, as well as fractures, porosity, incomplete fusion, and flaws in joints.

1. PRINCIPLE

- Liquid penetrant testing involves the application of a fluid with low viscosity on the material to be tested.
- This fluid seeps into any defects such as cracks or porosity before a developer is applied which allows the penetrant liquid to seep upwards and create a visible indication of the flaw.
- Liquid penetrant tests can be conducted using solvent removable penetrants, water washable penetrants or post-emulsifiable penetrants.

2. Basic Processing Steps of a Liquid Penetrant Inspection

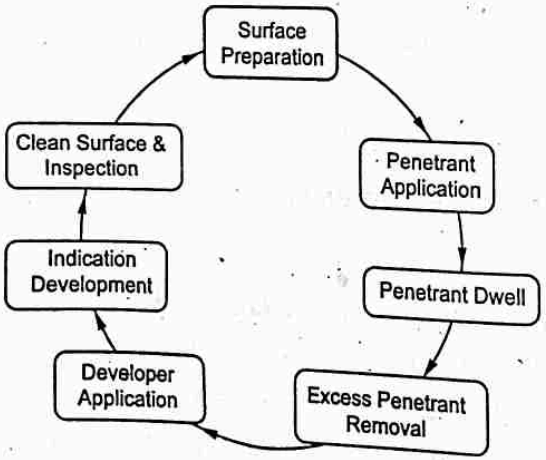


Fig. 3.3. Flow chart in basic process of Liquid Penetrant Inspection

(a) Surface Preparation

- One of the most critical steps of a liquid penetrant inspection is the surface preparation.
- The surface must be free of oil, grease, water, or other contaminants that may prevent penetrant from entering flaws.
- The sample may also require etching if mechanical operations such as machining, sanding, or grit blasting have been performed. These and other mechanical operations can smear metal over the flaw opening and prevent the penetrant from entering.

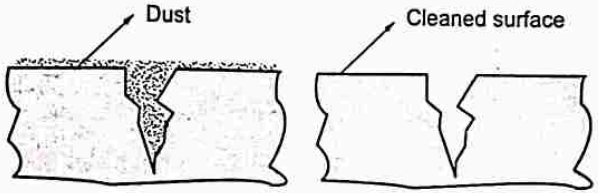


Fig. 3.4. Surface Preparation

(b) Penetrant Application

- Once the surface has been thoroughly cleaned and dried, the penetrant material is applied by spraying, brushing, or immersing the part in a penetrant bath.

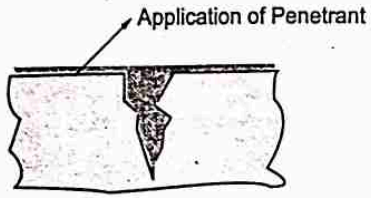


Fig. 3.5. Penetrant Application

(c) Penetrant Dwell

- The penetrant is left on the surface for a sufficient time to allow as much penetrant as possible to be drawn from or to seep into a defect.
- The times vary depending on the application, penetrant materials used, the material, the form of the material being inspected, and the type of defect being inspected for.

- Minimum dwell times typically range from five to 60 minutes. Generally, there is no harm in using a longer penetrant dwell time as long as the penetrant is not allowed to dry.

(d) Excess Penetrant Removal

- This is the most delicate part of the inspection procedure because the excess penetrant must be removed from the surface of the sample while removing as little penetrant as possible from defects.
- Depending on the penetrant system used, this step may involve cleaning with a solvent, direct rinsing with water, or first treating the part with an emulsifier and then rinsing with water.

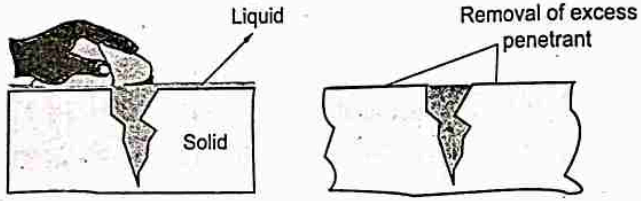


Fig. 3.6. Excess Penetrant Removal

(e) Developer Application

- A thin layer of developer is then applied to the sample to draw penetrant trapped in flaws back to the surface where it will be visible.
- Developers come in a variety of forms that may be applied by dusting (dry powdered), dipping or spraying (wet developers).

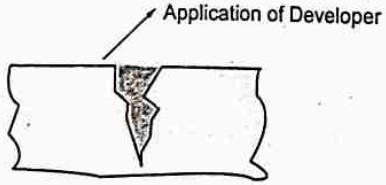


Fig. 3.7. Application of developer

(f) Indication Development

- The developer is allowed to stand on the part surface for a period of time sufficient to permit the extraction of the trapped penetrant out of any surface flaws.
- This development time is usually a minimum of 10 minutes. Significantly longer times may be necessary for tight cracks.

(g) Inspection

- Inspection is then performed under appropriate lighting to detect indications from any flaws which may be present.

(h) Clean Surface

- The final step in the process is to thoroughly clean the part surface to remove the developer from the parts that were found to be acceptable.

3. ADVANTAGES

- High sensitivity to small surface discontinuities.
- Easy inspection of parts with complex shapes.
- Quick and inexpensive inspection of large areas and large volumes of parts/materials.
- Few material limitations (metallic and nonmetallic, magnetic and nonmagnetic, and conductive and nonconductive can all be inspected).
- A visual representation of the flaw are indicated directly on the part surface.
- It is easy and requires minimal amount of training.
- Suitable for parts with complex shapes.
- Portable (materials are available in aerosol spray cans).
- Low cost (materials and associated equipment are relatively inexpensive).

4. DISADVANTAGES

- Only surface breaking defects can be detected.
- Only materials with a relatively nonporous surface can be inspected.
- Pre-cleaning is critical since contaminants can mask defects.
- The surface finish of the specimen after the test is difficult.
- The compatibility of the materials with the specimen.
- The sensitivity required.
- The size, shape and accessibility of the area to be inspected.
- The inspector must have direct access to the surface being inspected.
- Surface finish and roughness can affect inspection sensitivity.
- Multiple process operations must be performed and controlled.

- Post cleaning of acceptable parts or materials is required.
- Chemical handling and proper disposal is required.

5. APPLICATION

- **Aerospace:** Typical Components that are checked by this method include Turbine, rotor disc, blades, aircraft wheels, Casting, forged parts and welded assemblies
- **Automobiles:** Many automotive parts particularly aluminum castings and forging including pistons and cylinder heads are subjected to this form of quality checks before assembly
- **Railways:** LPI to detect fatigue cracking is also used for the regular in service examination of the bogie frames of railway locomotive and the rolling stock
- **Tool and dies:** Field drilling rays, drill pipes, castings and drilling equipment's inspected by this methods.
- **Inspection on reactors and tank:** Tanks, vessels, reactors, piping, dyers in the chemical, petro-chemical industries.

3.2.3. PENETRANTS

- It is used for detection of surface imperfections in non-porous materials and basically consists of applying a flow of liquid to the surface of the material to be tested.
- The liquid, by capillary action, will penetrate the discontinuities and the excess remaining on the surface will be removed by a suitable cleaning system. It will be highly visible or fluoresce brightly to produce easy to see indications.

1. Types of Penetrants

- **Fluorescent Penetrants:** They contain a dye or several dyes that fluoresce when exposed to ultraviolet radiation.
- **Visible Penetrants:** They contain a red dye that provides high contrast against the white developer background.

2. Methods used for excess removal of Penetrants

- Water washable

- Solvent removable
- Post-Emulsifiable
  - Lipophilic
  - Hydrophilic

3.2.4. DEVELOPERS

- The role of the developer is to pull the trapped penetrant material out of defects and spread it out on the surface of the part so it can be seen by an inspector.

1. The six standard forms of developers are

- Dry Powder
- Water Soluble
- Water Suspendable
- Non aqueous
  - Type 1: Fluorescent (Solvent Based)
  - Type 2: Visible Dye (Solvent Based)

3.2.5. MAGNETIC PARTICLE TESTING (MT)

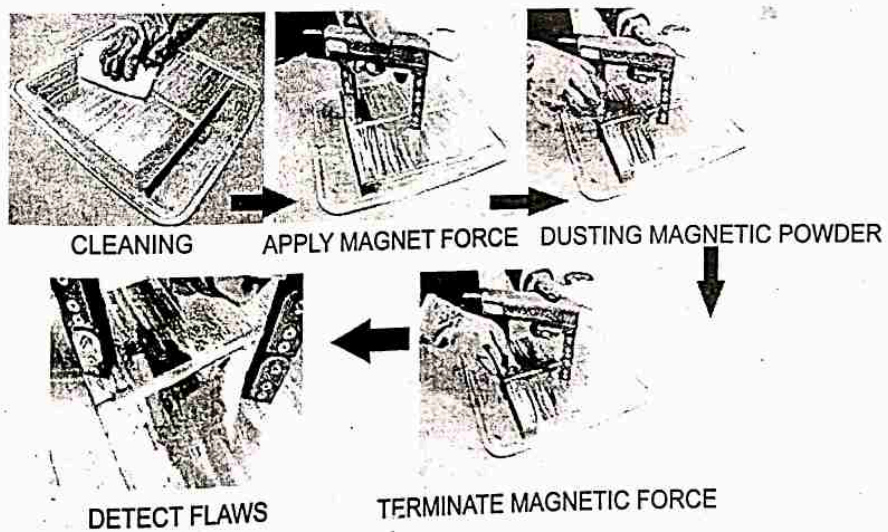


Fig. 3.8. Demo of magnetic particle testing

- A magnetic field is established in a component made from ferromagnetic material. The magnetic lines of force travel through the material and exit and reenter the material at the poles.
- Defects such as crack or voids cannot support as much flux, and force some of the flux outside of the part.
- Magnetic particles distributed over the component will be attracted to areas of flux leakage and produce a visible indication.

1. PRINCIPLE

- This NDT process uses magnetic fields to find discontinuities at or near the surface of ferromagnetic materials. The magnetic field can be created with a permanent magnet or an electromagnet, which requires a current to be applied.
- The magnetic field will highlight any discontinuities as the magnetic flux lines produce leakage, which can be seen by using magnetic particles that are drawn into the discontinuity.

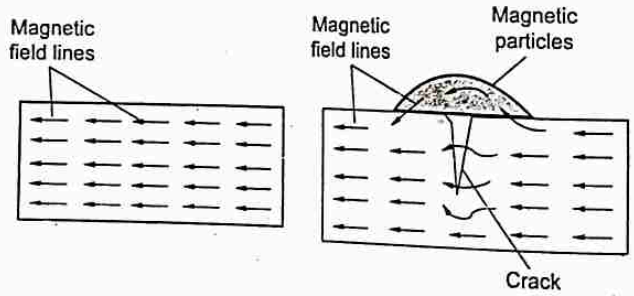


Fig. 3.9. Principle of working

2. MAGNETIC PROPERTIES OF MATERIALS

(a) HYSTERESIS LOOP

- A hysteresis loop shows the relationship between the induced magnetic flux density B and the magnetizing force H. It is often referred to as the B-H loop
- From the hysteresis loop, a number of primary magnetic properties of a material can be determined.
- Retentivity, Residual Magnetism or Residual Flux, Coercive Force, Permeability and Reluctance.

(b) PERMEABILITY

- Permeability describes how easily a material can be magnetized; a material with a high permeability is easier to magnetise than a material with a low permeability
- A material's permeability is determined by dividing the magnetising force applied to a material into the magnetic flux density achieved in the material – permeability has no units.
- There are three material categories that are related to permeability: diamagnetic ,paramagnetic and ferromagnetic

(a) Diamagnetic materials: It have a permeability value slightly less. It will slightly repel a magnetic field.

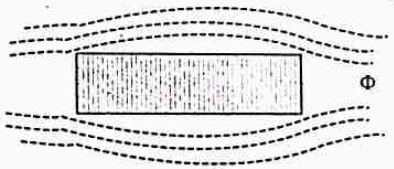


Fig. 3.10. Repel of diamagnetic

(b) Paramagnetic materials: It have a permeability value slightly greater .It is slightly easier for the magnetic flux to pass through the paramagnetic material than to travel through the vacuum

Fig. 3.11. Repel of paramagnetic

(c) Ferromagnetic materials: It have a permeability value much higher, that it is much easier for the magnetic flux to pass through the ferromagnetic material than to pass through the vacuum. Ferromagnetic materials are very strongly attracted by a magnetic field.

Fig. 3.12. Ferromagnetic material

3. TYPES OF MAGNETISATION

- **Circular magnetization** - Circular magnetic field will be produced around the component at right angles to the direction of the electric current which produced it.

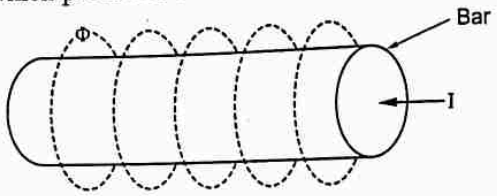


Fig. 3.13. Circular magnetism

- **Longitudinal magnetization** - The magnetic flux flows from pole to pole, we call this longitudinal magnetisation. Discontinuities will be detectable once more at 90° (±45°) to the flux direction.

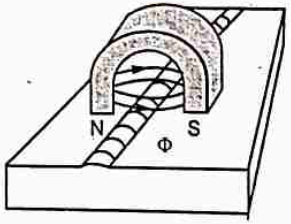


Fig. 3.14. Longitudinal magnetization

4. COMPONENTS IN MAGNETIC PARTICLE INSPECTION (MPI)

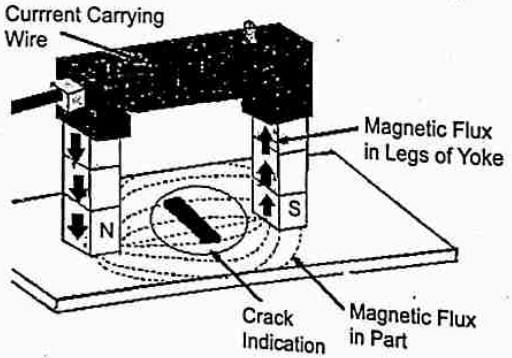


Fig. 3.15. Components of magnetic particle testing

(*a*) Permanent magnet
(*b*) Electromagnetic Yoke

(*c*) Current flow probes
(*d*) Flexible coil
(*e*) Adjacent cable

(*a*) Permanent magnets

- Permanent magnets are sometimes used for magnetic particle inspection as the source of magnetism. The two primary types of permanent magnets are bar magnets and horseshoe (yoke) magnets.

(*b*) Electromagnetic Yoke

- An electromagnetic yoke is a very common piece of equipment that is used to establish a magnetic field. It is basically made by wrapping an electrical coil around a piece of soft ferromagnetic steel.

(*c*) Current flow probes

- Probes are handheld electrodes that are pressed against the surface of the component being inspected to make contact for passing electrical current through the metal.

(*d*) Adjacent cable

- Coils and conductive cables are used to establish a longitudinal magnetic field within a component. When a preformed coil is used, the component is placed against the inside surface on the coil. Coils typically have three or five turns of a copper cable within the molded frame.

(*e*) Portable Power Supplies

- Portable power supplies are used to provide the necessary electricity to the prods, coils or cables. Power supplies are commercially available in a variety of sizes.

5. WORKING OF MAGNETIC PARTICLE TESTING

(*a*) Pretreatment

- The surface must be free of grease, oil or other moisture that could keep particles from moving freely.
- A thin layer of paint, rust or scale will reduce test sensitivity but can sometimes be left in place with adequate results.

(b) Apply the magnetizing force (magnetic particle)

- Use permanent magnets, an electromagnetic yoke, prods, a coil or other means to establish the necessary magnetic flux.

(c) Dust on the dry magnetic particles

- Dust on a light layer of magnetic particles.

(d) Gently blow off the excess powder

- With the magnetizing force still applied, remove the excess powder from the surface with a few gentle puffs of dry air.
- The force of the air needs to be strong enough to remove the excess particles but not strong enough to dislodge particles held by a magnetic flux leakage field.

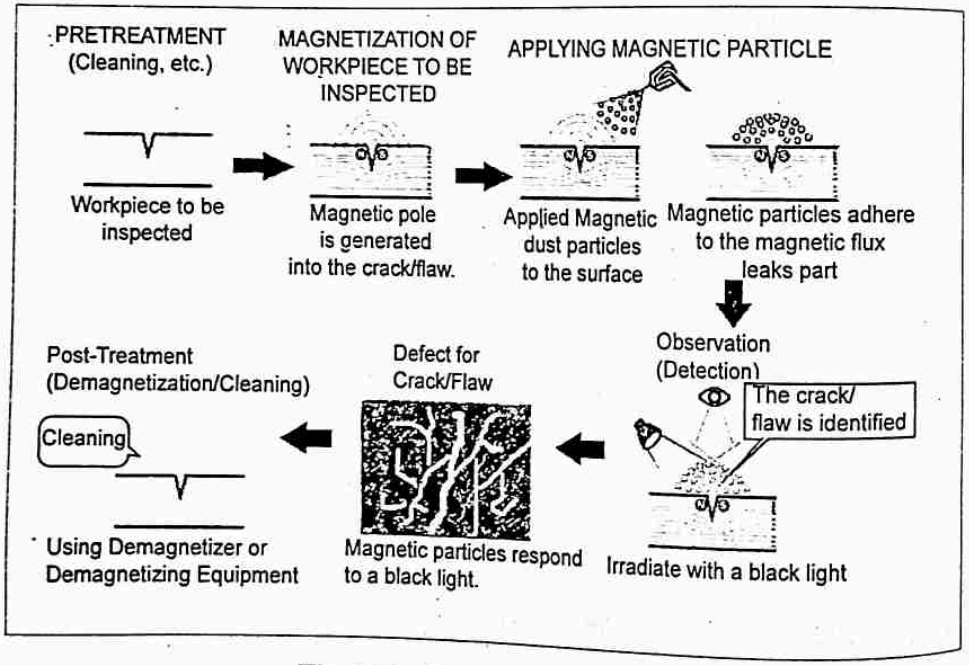


Fig. 3.16. Nature of finding flaws

(e) Terminate the magnetizing force

- If the magnetic flux is being generated with an electromagnet or an electromagnetic field, the magnetizing force should be terminated. If permanent magnets are being used, they can be left in place.

(f) Detect the defects

- Look for areas where the magnetic particles are clustered with the help of visual aids like illuminating light, microscope, naked eyes and magnifying glass etc.,

(g) Post Treatment

- The surface of material should be demagnetized (or) cleaned using demagnetizer (or) demagnetizing equipment.

6. ADVANTAGES

- Can find both surface and near sub-surface defects.
- This inspection formats are extremely portable and low cost.
- Rapid inspection with immediate results.
- Indications are visible to the inspector directly on the specimen surface.
- Can detect defects that have been smeared over.
- Can inspect parts with irregular shapes (external splines, crankshafts, connecting rods, etc.).
- The method can be adapted for site or workshop use.
- It is inexpensive compared to radiography.
- Large or small objects can be examined.

7. LIMITATIONS

- The specimen must be ferromagnetic (e.g. steel, cast iron)
- Paint thicker than about 0.005" must be removed before inspection
- Post cleaning and post demagnetization is often necessary
- Maximum depth sensitivity is typically adopted as 0.100" (deeper under perfect conditions)
- Alignment between magnetic flux and defect is important
- Insensitive to internal defects
- Require magnetization and demagnetization of materials to be inspected
- Require power supply for magnetization
- Coating may mask indication
- Material may be burned during magnetization

8. APPLICATIONS

- Magnetic particle testing or inspection (MT or MPI testing) is used for quality control and materials testing in all major industries. This includes castings, forgings, plates, extruded components, weld joints, electrical and electronic component manufacturing, production of steel, pressure vessels, ships, bridges, motor vehicles, machinery and jet engines.
- The flaws to be detected include cracks, inclusions, pipe, laminations, bursts and flakes.
- Testing effective in detecting fatigue cracks during in-service maintenance inspection of power plants, cement plants, sugar plants, petroleum refinery machinery components and structures

Mainly used to find,

- Fatigue Cracks
- Grinding Cracks
- Inclusions in aerospace blooms, billets, and bars
- Quenching Cracks
- Shrink Cracks
- Stress Corrosion Cracking
- Welding Defects

3.2.6. THERMOGRAPHY TEST

- Infrared testing or thermography uses sensors to determine the wavelength of infrared light emitted from the surface of an object, which can be used to assess its condition.
- A thermographer views an object with a thermal imager to measure the infrared emitted from the surface. However, to confuse matters, heat sources behind the imager can reflect from the surface making the object appear hotter than it really is. Even the heat from the body of the thermographer can cause this effect on objects at ambient temperature

1. PRINCIPLE

- Thermography is a technique of obtaining an image of the heat distribution over the surface of an object. The usual method is to use a special

television camera with an infrared sensitive detector and a lens which transmits infrared radiation. Such cameras can operate at normal video rates.

2. TYPES OF THERMOGRAPHY

- **Passive thermography** uses sensors to measure the wavelength of the emitted radiation and if the emissivity is known or can be estimated, the temperature can be calculated and displayed as a digital reading or as a false Colour image.
- **Active thermography** induces a temperature gradient through a structure. Features within it that affect the heat flow result in surface temperature variations that can be analyzed to determine the condition of a component. Often used to detect near surface delamination or bonding defects in composites.
- **The external excitations/optical excitations** or may be the external energy says so generally this energy is delivered to the surface and then propagated through the material until it encounters flaw examples photographic classes for heat pulsed simulations, halogen lamps for periodic heating.
- **Internal excitations/mechanical excitations** so generally the energy is injected into the specimen in order to stimulate exclusively the defects

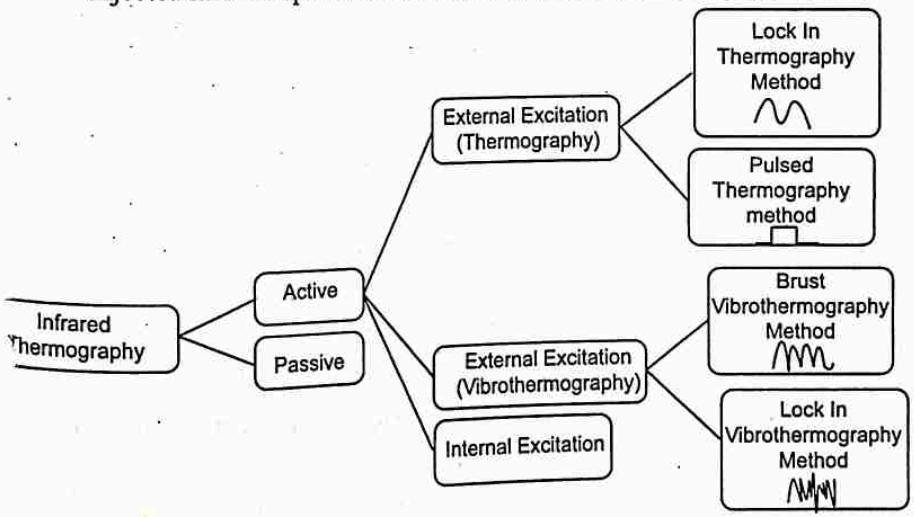


Fig. 3.17. Types of Thermography

3. BASIC AIDS OR COMPONENTS IN THERMOGRAPHY

- **Thermographic camera** so it is also known as infrared camera or maybe the thermal imaging camera. It is a device that forms a heat zone image using infrared radiation it operates in wavelengths as long as 14,000 nanometer.
- There are two basic types of thermographic camera. One is called the cooled infrared detectors another one is called the uncooled infrared detectors.
- **Control unit** is which sets the level of adjustment for halogen lamp and heater.
- **Pc/image processing unit** which displays the defect unit after deep proceess of unit.

4. WORKING OF VARIOUS THERMOGRAPHY TESTING METHODS

(a) Burst Vibrothermography Method

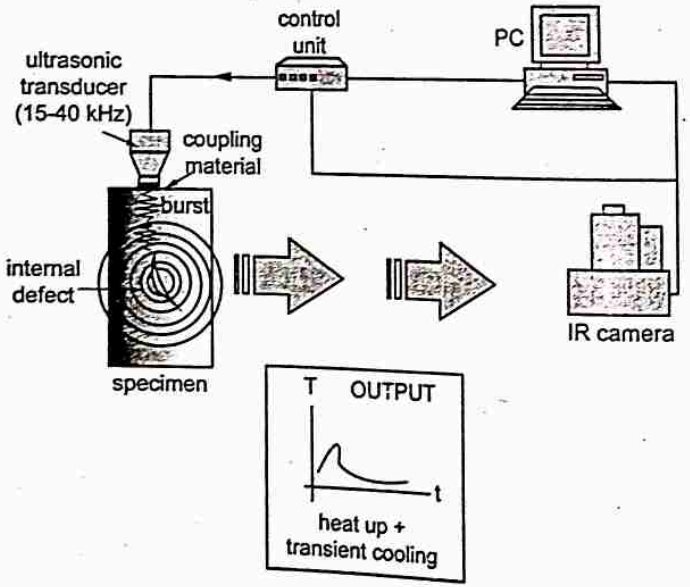


Fig. 3.18. Burst Vibrothermography Method

- Ultra-sonic burst phase thermography is also employed when short ultrasonic bursts are used.
- In this method an ultrasonic transducer with a fixed resonance frequency typically at 20 or 40 kHz has been used to excite high amplitudes of

vibration which rise surface temperature around defect, is large enough to be detected by an infrared camera.

(b) Lock In Vibrothermography Method

- The Lock-In method is suitable for testing components with a low thermal diffusivity Pulse thermography (pulse method).

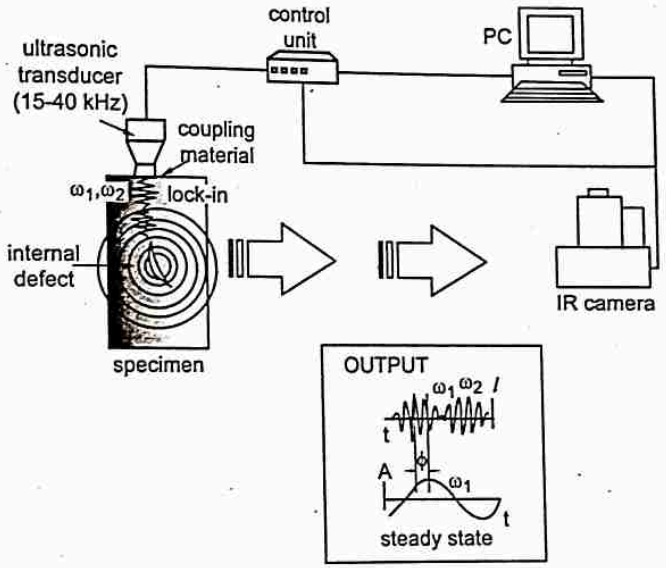


Fig. 3.19. Lock In Vibrothermography Method

- A very short pulse – usually in the units of milliseconds – is used to excite the object.
- A ultrasonic transducer is typically used as an excitation source.
- The advantage of this method is the speed of the analysis and a possibility to estimate the defects depth.
- The disadvantage is a limited to of detection capabilities based on geometrical orientation of defects.

Lock In Thermography Method

- Lock-In thermography is a periodic excitation method). When the input energy (Halogen lamps) wave penetrates the object's surface, it absorbed.
- The reflected portion of the wave causing an interference pattern in the local surface temperature.

- When the input wave reaches areas within the object when thermo physical properties are not same (due to defect) compared to surrounding the input wave is partially reflected.
- It has the advantage that it can be used on large surfaces and it puts a low thermal energy on the part being inspected.
- The disadvantage is a longer measurement time and dependence of detection capabilities on a geometrical orientation of defects

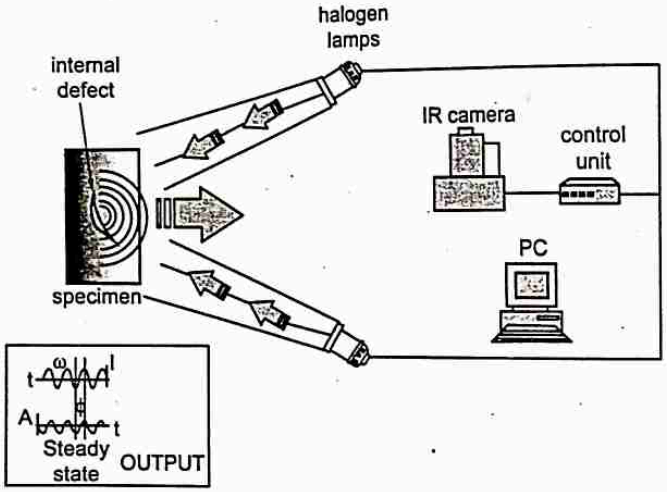


Fig. 3.20. Lock In Thermography Method

(d) Pulsed Thermography method

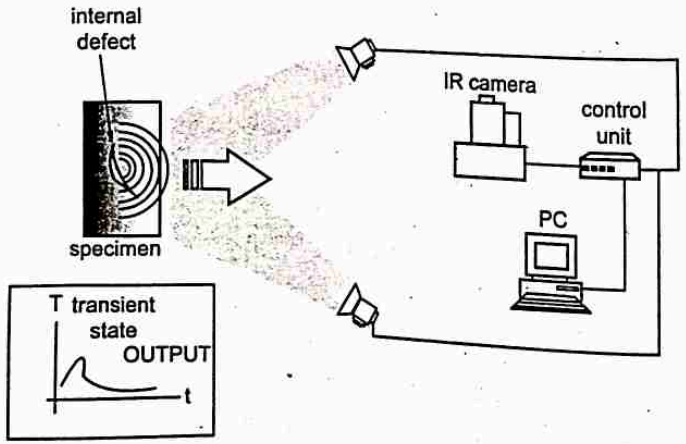


Fig. 3.21. Pulsed Thermography method

- Pulsed thermography is a classical optical excitation thermography technique. In pulsed thermography, high-energy lamps are often used to produce a uniform heating source on the specimen surface.
- The heat transmits through the inspected specimen to the subsurface defects or damages, and then returns to the specimen surface.
- A uniform temperature rise will be recorded if there are no defects in the specimen. If there are defects such as voids or delamination, a localized high-temperature zone will be observed above the defect due to the insulation effect.

5. ADVANTAGES

- Data collection system can record temperature changes with time
- High-speed, portable, and non-contact
- Ability to inspect large areas
- Effective prevention of test scrap
- Contactless testing with low thermal stress
- Simple analysis of large, uneven surfaces
- Categorization of different types of defects

6. DISADVANTAGES

- Risk of damage the sample(e.g., overheating)
- Limitations of inspected thickness
- Variable emissivity of materials
- Dependence from thermal contrast
- Expensive instrumentation require qualified personnel accuracy

7. APPLICATIONS

- Used largely in Aerospace industry ,Automotive industry and Power industry.
- Quality assurance for bonded, welded, soldered and other joints by means of cavity detections (e.g. on vehicle interior parts).
- Localization of defects in joints such as cavities, defective welding seams/points.
- Testing of metallic and non-metallic materials/material compounds.
- Tests of internal structures, such as fractures or impacts in honeycomb lightweight constructions.

3.2.7. RADIOGRAPHIC TESTING

- Radiographic Testing (RT) is a non-destructive testing (NDT) method which uses either x-rays or gamma rays to examine the internal structure of manufactured components identifying any flaws or defects.

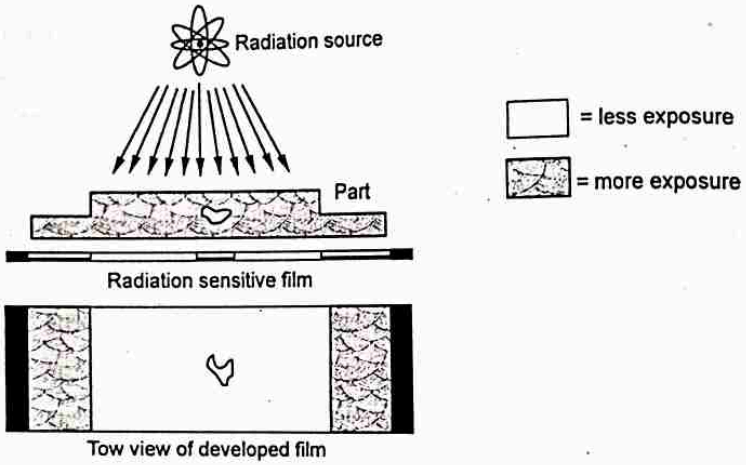


Fig. 3.22. Radiographic Testing

- X-rays are commonly used for thin or less dense materials while gamma rays are used for thicker or denser items.
- The term radiography usually implies a radiographic process that produces a permanent image on film (conventional radiography) or paper (paper radiography or xeroradiography), although, in a broad sense, it refers to all forms of radiographic inspection.
- When inspection involves viewing of a real-time image on a fluorescent screen or image intensifier, the radiographic process is termed real-time inspection. When electronic, non-imaging instruments are used to measure the intensity of radiation, the process is termed radiation gaging.
- Neutron radiography refers to radiographic inspection using neutrons rather than electromagnetic radiation.

1. PRINCIPLE

- In Radiography Testing the test-part is placed between the radiation source and film (or detector). The radiation passed through a test piece to detect defects.

- The results can be processed using film radiography, computed radiography, computed tomography or digital radiography. The method is used, the radiation will show discontinuities in the material due to the strength of the radiation.

2. TYPES OF RADIOGRAPHIC TESTING

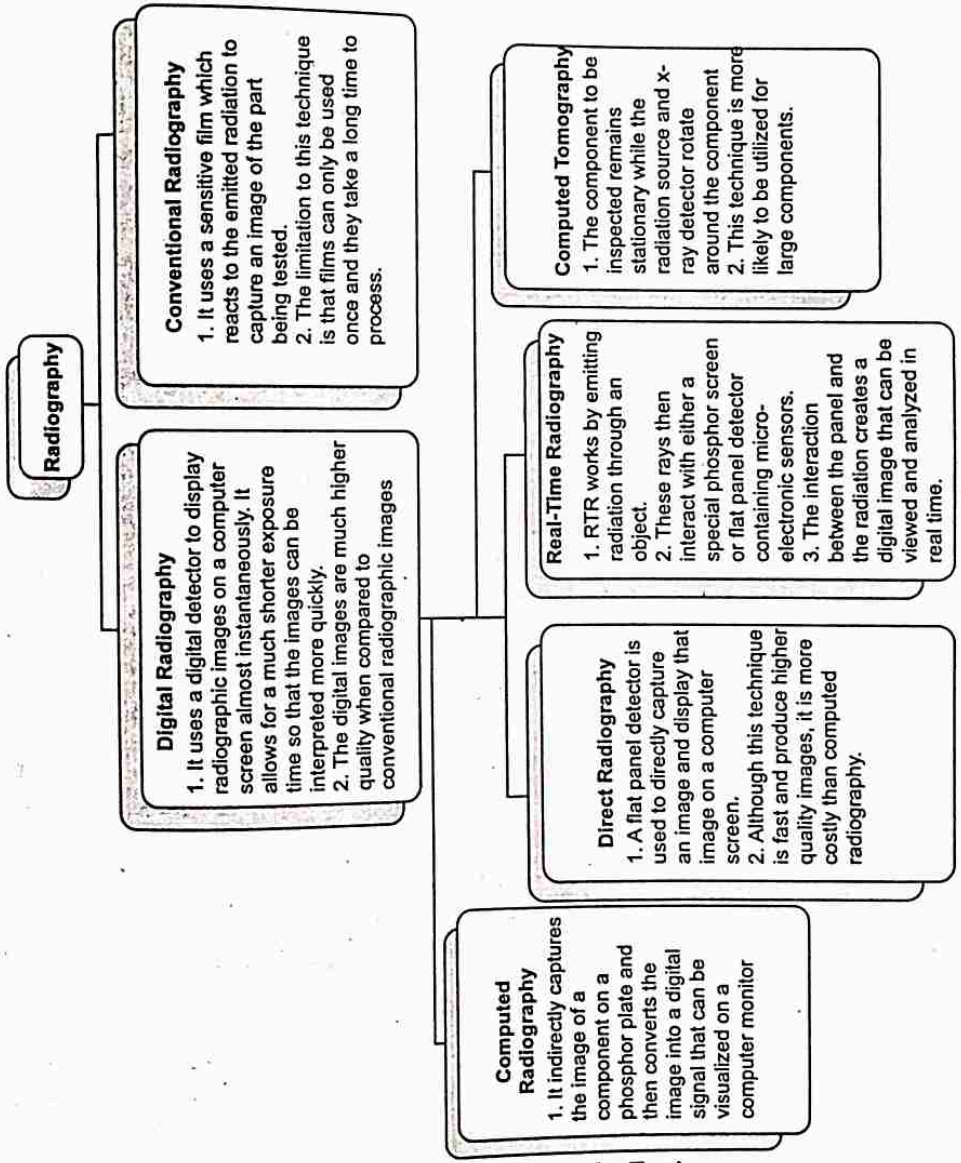


Fig. 3.23. Types of Radiography Testing

3. BASIC COMPONENTS OF RADIOGRAPHIC TESTING

(a) Source

- X-rays are generated by directing a stream of high speed electrons at a target material such as tungsten, which has a high atomic number. When the electrons are slowed or stopped the interaction with the atomic particles of the target, X-radiation is produced.
- The neutron, energy is released in the form of gamma rays. Two of the most common industrial gamma-ray sources for industrial radiography are Iridium-192 and Cobalt-60.

(b) Radiographic Film

- When X-rays or gamma-rays or light strike the film, some of the halogen atoms are liberated from the silver halide crystal and thus leaving the silver atoms alone.
- This change is detected by a method is called a "latent (hidden) image". When the film is exposed to a chemical solution (developer) the reaction results in the formation of black.

4. WORKING PRINCIPLE OF RADIOGRAPHIC TESTING

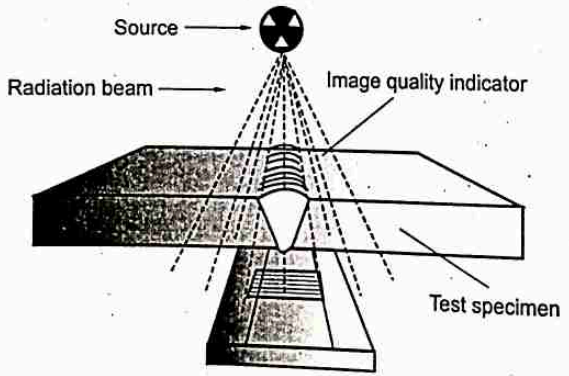


Fig. 3.24. Working principle of radiographic testing

- The testing specimen part to be placed between the radiation source (x-ray or gamma source) and a piece of film which records the defect data.
- The undefeated part will stop some of the radiation. Thicker and denser area will allow less radiation to pass through. The discontinuity allows rays to pass.

- The property of the film will vary with the amount of radiation reaching the film through the test object. These differences in "absorption" can be recorded on film, or electronically. The energy of the radiation affects its penetrating power.
- Higher energy radiation can penetrate thicker and denser materials.
- The radiation energy and exposure time must be controlled to properly image the region of interest.

5. RADIOGRAPH FILM ANALYSIS

- If an object has a high density, ie a thicker object, it absorbs more radiation causing less radiation to hit the film, which produces a lighter image.
- If an object has a low density, ie when the through section is reduced or there is a lower-density material such as slag (compared to the surrounding material), it will absorb less radiation causing more radiation to hit the film, producing a darker image.
- The image on the film cannot initially be seen; this is called the latent image and can only be seen when the film is developed. The quality of this image mainly depends upon two properties;
- Density- This is the degree of blackness on the radiograph. There will be minimum and maximum amounts of density to make the radiograph readable and give the required sensitivity.
- Contrast- Radiographic contrast is the degree of difference between density fields on a radiograph. If there are only blacks and whites on a radiograph, this would be high contrast. If only tones of a similar density are on the graph, this would be low contrast.

6. SAFETY ASPECTS OF RADIATION TEST

- **Film badges/TLDs** (thermoluminescent dosemeters)

  The detectors worn by all industrial radiographers that measure the dose a radiographer receives over a period of time, usually one month.

- **Survey meters** (dose rate meters)

  The instruments that can measure dose rates per unit time.

- **Audible alarms**

  These are alarm/warning devices that should always be worn by radiographers working with gamma radiation.

- **Pocket dosemeters (exposure meters)**

  These meters record an accumulative amount of radiation and can be used for measuring the dose received, for instance over one day, instead of waiting for the monthly badge results.

- **Audio/visual alarms**

  These are alarms, such as the 'Gamma Alert', that are placed inside the radiation area and normally have an amber flashing light

7. ADVANTAGES

- Both surface and internal discontinuities can be detected.
- Significant variations in composition can be detected.
- Can be used for inspecting hidden areas (direct access to surface is not required).
- Permanent test record is obtained.
- Good portability especially for gamma-ray sources.
- Minimum surface preparation required.
- Verify internal flaws on complex structures.
- Isolate and inspect internal components.
- Automatically detect and measure internal flaws.
- Measure dimensions and angles within the sample without sectioning.
- Sensitive to changes in thickness, corrosion, flaws and material density changes.

8. DISADVANTAGES

- Hazardous to operators and other nearby perscnnel.
- High degree of skill and experience is required for exposure and interpretation.
- The equipment is relatively expensive (especially for x-ray sources).
- The process is generally slow

- Highly directional (sensitive to flaw orientation).
- Depth of discontinuity is not indicated.
- It requires a two-sided access to the component.
- Many safety precautions for the use of high intensity radiation.
- Many hours of technician training prior to use.
- Access to both sides of sample required.
- Orientation of equipment and flaw can be critical.
- Determining flaw depth is impossible without additional angled exposures.
- Expensive initial equipment cost.

9. APPLICATIONS

- Industrial Radiographic testing is used extensively on castings and weldments.
- Radiography is also well suited for testing of semiconductor devices for cracks, broken wires, unsoldered connections, foreign material and misplaced components.
- Sensitivity of radiography to various types of flaws depends on many factors, including type of material, type of flaw and product form.
- Both ferrous alloys can be radiographed, as well as non-metallic materials and composites.

Used in fields of,

- Aerospace industries
- Military defense
- Offshore industries
- Marine industries
- Power-gen industries
- Petrochem industries
- Waste Management
- Automotive industries
- Manufacturing industries
- Transport industries

3.2.8. ELECTROMAGNETIC TESTING (ET) OR EDDY CURRENT TESTING

- This testing method uses an electric current or magnetic field which is passed through a conductive part.
- Eddy current testing uses an alternating current coil to induce an electromagnetic field into the test piece, alternating current field measurement and remote field testing both uses a probe to introduce a magnetic field, with RFT generally used to test pipes.

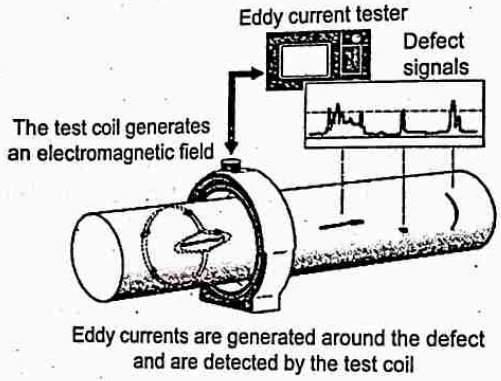


Fig. 3.25. Eddy current tester

1. PRINCIPLE

- An electromagnetic inductor is used to generate a magnetic field. When this field is introduced in the surface of the test piece, it generates so called "eddy currents" in the material.

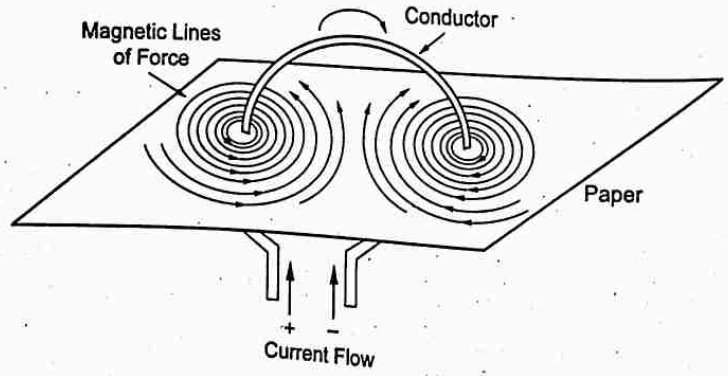


Fig. 3.26. Principle of eddy current flowing

- These currents generate their own magnetic field which resists the initial field created by the inductor. When a discontinuity disturbs the eddy currents, this can be registered by measuring the resulting change in impedance of the coil.

2. METHODS OF EDDY CURRENT TESTING

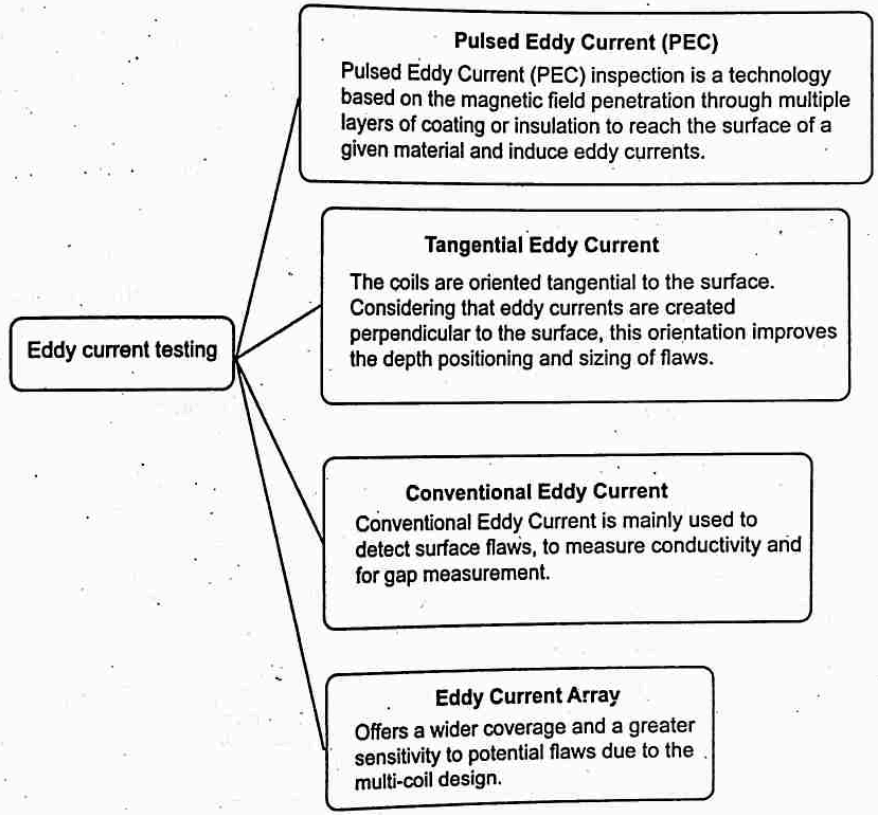


Fig. 3.27. Types of eddy current testing

3. COMPONENTS OF EDDY CURRENT TESTING

- **Eddy probe (AC source, Electromagnetic coil, Display unit, Receiver coil, Exciting coil, Display unit)** - AC voltage source for the purpose of the test can generate a primary electromagnetic alternating field. The exciter coil and the receiver coil normally have coil axes parallel to each other, so that the primary alternating magnetic field of the exciter coil induces an AC voltage in the receiver coil.

4. WORKING OF EDDY CURRENT TESTING

- When eddy current probe brought close to the testing material, an alternating current flows through a wire coil and generates an oscillating magnetic field.
- The electrical currents are called eddy currents because the flow in circles at and just below the surface of the material. The Eddy Current generates a new superposed magnetic field. This field is detected by a receiver coil.
- Interruptions in the flow of eddy currents, caused by imperfections, dimensional changes, or changes in the materials conductive and permeability properties, can be detected with the proper equipment like probs.
- Eddy current testing can be used on all electrically conducting materials with a reasonably smooth surface. The figure shows the difference in flawless and flaw surface with impedance graph

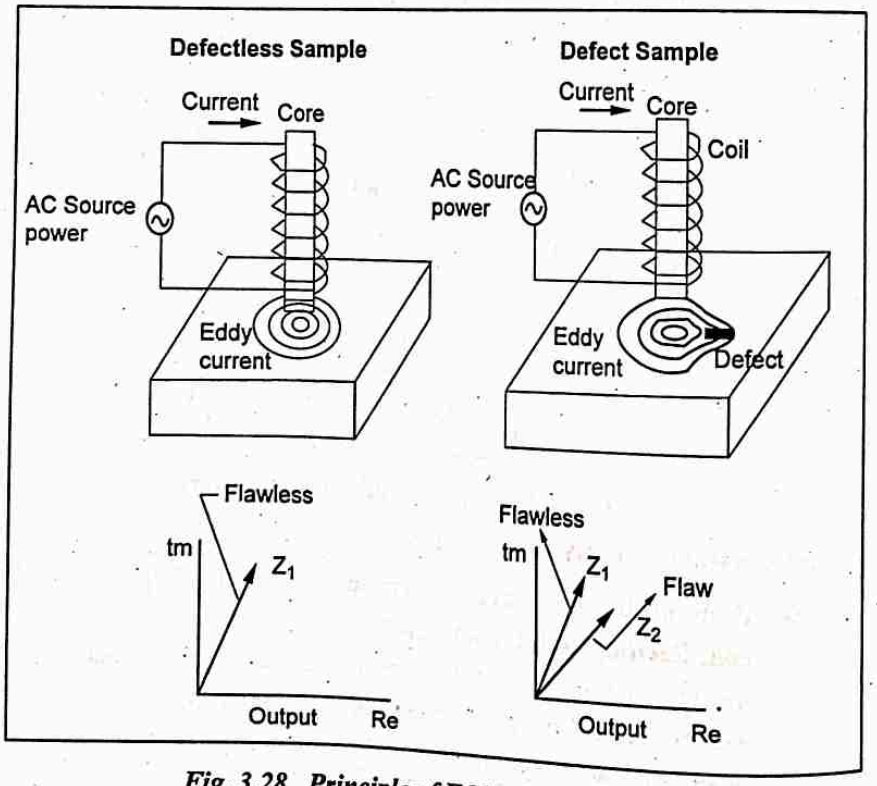


Fig. 3.28. Principle of Eddy current testing

- The test equipment consists of a generator (AC power supply), a test coil and recording equipment, e.g. a galvanometer or an oscilloscope
- Used for crack detection, material thickness measurement (corrosion detection), sorting materials, coating thickness measurement, metal detection, etc.

4. THE STRENGTH OF THE EDDY CURRENTS PRODUCED DEPENDS ON

- Electrical conductivity of the specimen
- Magnetic permeability (for a ferromagnetic specimen)
- Stand-off distance between the specimen and coil.
- AC frequency used in the exciting coil dimensions of the coil and specimen.

5. FACTORS THAT AFFECT EDDY CURRENT INSPECTION

- Material conductivity
- Permeability
- Frequency
- Geometry
- Proximity/Lift-Off
- Depth of Penetration
- Eddy Current Testing and Industry

6. ADVANTAGES

- Sensitive to small cracks and other defects
- Detects surface and near surface defects
- Inspection gives immediate results
- Equipment is very portable
- Method can be used for much more than flaw detection
- Minimum part preparation is required
- Test probe does not need to contact the part
- Inspects complex shapes and sizes of conductive materials
- Able to detect surface and near-surface cracks as small as 0.5mm

- Able to detect defects through several layers, including non-conductive surface coatings, without interference from planar defects
- Effective on test objects with physically complex geometries
- Provides immediate feedback

7. LIMITATIONS

- Can only be used on conductive materials
- The depth of penetration is variable
- Very susceptible to magnetic permeability changes .
- Unable to detect defects that are parallel to the test object's surface
- Only conductive materials can be inspected
- Surface must be accessible to the probe
- Skill and training required
- Surface finish and roughness may interfere
- Reference standards needed for setup
- Depth of penetration is limited
- Flaws such as delamination that lie parallel to the probe coil winding and probe scan direction are undetectable.

8. APPLICATIONS

- It is often applied for surface crack detection and material sorting. Material sorting is used to ensure that the proper materials are in use and to verify component materials or assembly features (such as the orientation or position of a subcomponent in an assembly).
- **Weld Inspection** - To scan the surface for open surface cracks on weld caps and in heat affected zones.
- **Conductivity Testing** - Eddy current testing's ability to measure conductivity can be used to identify and sort ferrous & nonferrous alloys, and to verify heat treatment.
- **Surface Inspection** - Surface cracks in machined parts and metal stock can be readily identified with eddy current.

- **Corrosion Detection** - Low frequency probes can be used to locate corrosion on second and third layers of metal that cannot be inspected ultrasonically.
- **Bolt Hole Inspection** - Cracking inside bolt holes can be detected using bolt hole probes, often with automated rotary scanners.
- **Tubing inspection** - Both in-line inspection of tubing at the manufacturing stage and field inspection of tubing like heat exchangers are common eddy current applications. Both cracking and thickness variations can be detected.

3.2.9. EDDY PROBE

- A coil of conductive wire is excited with an alternating electrical current. This wire coil produces an alternating magnetic field around itself.
- This wire coil produces an alternating magnetic field around itself. The magnetic field oscillates at the same frequency as the current running through the coil. When the coil approaches a conductive material, currents opposite to the ones in the coil are induced in the material eddy currents.
- An eddy current probe is arranged at a small distance (test distance) to a surface of a test specimen to be tested, which consists at least in the region of the surface of an electrically conductive material.

1. TYPES OF EDDY CURRENT PROBE

- Surface probes - Used for identifying flaws on and below metal surfaces, usually large diameter to accommodate lower frequencies for deeper penetration, or for scanning larger areas.
- Pencil probes - Smaller diameter probes housing coils built for high frequencies for high resolution of near surface flaws.
- Bolt hole probes - Designed to inspect the inside of a bolt hole. These probes can be rotated by hand or automatically using a rotary scanner.
- Donut probes - Designed to inspect aircraft fastener holes with fasteners in place.
- Sliding probes - Also used in testing aircraft fastener holes, offering higher scan rates than donut probes.

- ID probes - Used for inspection of heat exchangers and similar metal tubing from the inside, available in a variety of sizes.
- OD probes - Used for inspection of metal tubing and bars from the outside, with the test piece passing through the coil

2. DESIGN OF PROBES

The probe material must be chemically compatible with the component. In brief, probe design is usually done considering the following,

- Geometry of the component e.g. rod, tube, plate etc.
- Type of discontinuity expected e.g. fatigue cracks, conductivity variation etc.
- Likely location of defect e.g. surface, sub-surface
- Coil impedance and its matching with the bridge circuit of the EC instrument
- Frequency range of the probe i.e. for simultaneous multi-frequency excitation
- Inspection requirement e.g. detection, evaluation of length, depth etc.
- Material characteristics e.g. ferromagnetic or non-ferromagnetic
- Coil response to a notch, drilled hole or other reference discontinuity
- Field distribution in space and eddy current flow distribution in the material
- Shape and dimensions of core, coil /coils and lift-off characteristics
- Environmental characteristics such as wear, temperature and chemical attack

3.2.10. ULTRASONIC TESTING (UT)

- **Ultrasonic testing (UT)** is a non-destructive testing techniques based on the propagation of ultrasonic waves (high frequency sound waves) are transmitted into materials to detect internal flaws or to characterize materials.
- The sound frequencies used to perform ultrasonic testing with range of 0.1 to 20 MHz and the wavelength in the range 1 to 10 mm. The velocity depends on the material and is in the range 1000-6000 m/s.

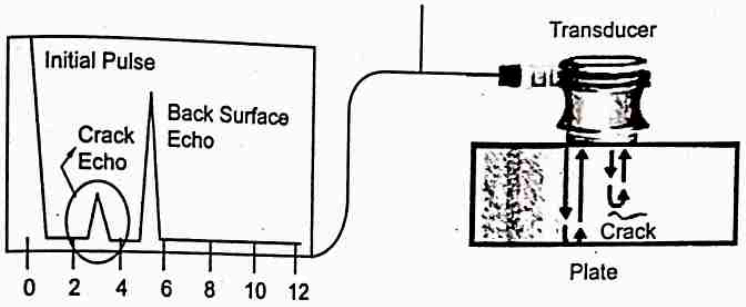


Fig. 3.29. Ultrasonic testing set up

1. PRINCIPLE

- Ultrasonic methods of NDT use beams of mechanical waves (vibrations) of short wavelength and high-frequency, transmitted from a small probe and detected by the same or other probes. Such mechanical waves can travel large distances in fine-grain metal, in the form of a divergent wave with progressive attenuation.

2. BASIC COMPONENTS IN ULTRASONIC TESTING

(a) Ultrasonic waves

- Ultrasonic waves are sound wave is very similar to light waves in that they can be reflected, refracted, and focused.
- Reflection and refraction occurs when sound waves interact with interfaces of differing acoustic properties.

(b) Probe

- Sound beam is emitted through a probe. The probe is made up of a piezoelectric material. Piezoelectric material has the ability of converting mechanical energy into electrical energy. It is reversible; hence an electrical energy can be converted into mechanical energy or sound energy. Sometimes the probe will act as transducer and receiver.
- Three types of probes are generally used in industries;
  - Normal Probe
  - TR probe
  - Angle Probe

(c) Transducer

- Transducer converts it to sound beam, these sound beam travels into the test object.
- A couplant is a material (usually liquid) that facilitates the transmission of ultrasonic energy from the transducer into the test specimen. Couplant is generally necessary because the acoustic impedance mismatch between air and solids (i.e. such as the test specimen) is large.

(d) Receiver

- Receiver receives 'crack echo'/'back surface echo' from the material.

(e) Display unit

- The received signals can be displayed as visual signals on cathode ray tube (CRT) or liquid crystal display (LCD) screen of the machine.
- The reflected signal strength is displayed versus the time from signal generation to when an echo was received.

3. DATA INTERPERTATION

There are three main types of display for flaw detectors: the A-scan, the B-scan and the C-scan presentation.

- An **A-scan presentation** is the most common display used in ultrasonic testing. It shows returning signal amplitudes vertically and the elapsed time or distance horizontally (depth).

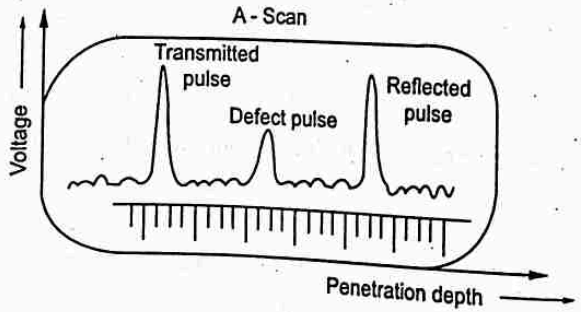


Fig. 3.30. A-scan presentation

- With **B-scan displays**, a cross-sectional view of the component under test is seen. The display shows the depth of reflectors and is used to determine

the cross-sectional size, location (both position and depth) and, with large discontinuities, can show the shape and orientation to a certain degree.

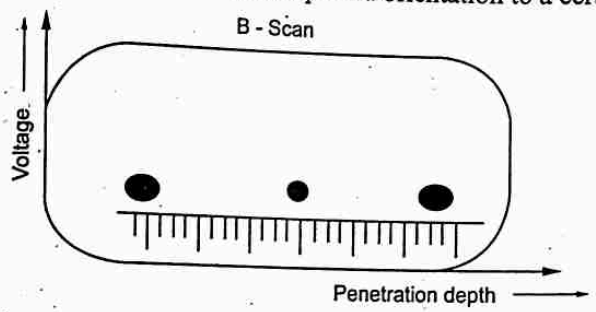


Fig. 3.31. B-scan

- A **C-scan display** is built up using raster scanning (X versus Y) over the component surface. It is mainly used with automated immersion equipment and is well suited for use with through-transmission systems.

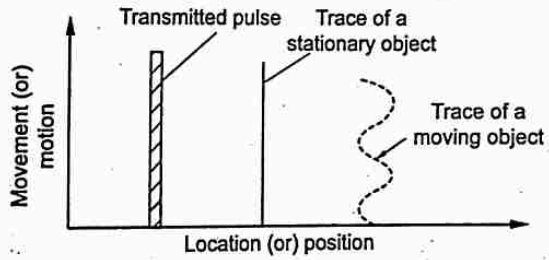


Fig. 3.32. C-scan display

4. METHODS IN ULTRASONIC TESTING

- Ultrasonic testing is a very versatile inspection method, and inspections can be accomplished in a number of different ways.
- Ultrasonic inspection techniques are commonly divided into three primary classifications.
  - Pulse-echo and Through Transmission
  - Contact and Immersion
  - Normal Beam and Angle Beam

(a) Pulse-echo method

- This is the method most commonly utilized in the ultrasonic testing of materials.

- The transmitter and receiver probes are on the same side of the specimen and the presence of a defect is indicated by the reception of an echo before that of the back wall echo.

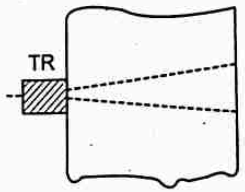


Fig. 3.33. Pulse-echo method probe

(b) Through Transmission method

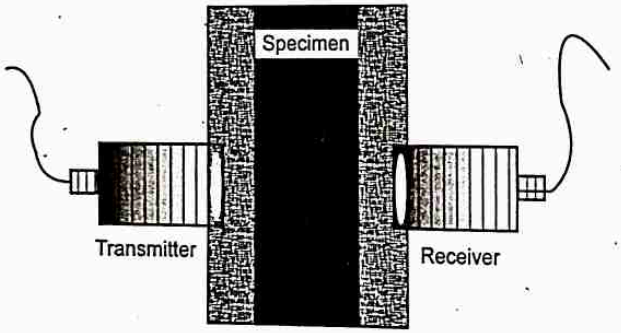


Fig. 3.34. Through Transmission method

- Transmitted and receiving probes are on the opposite side of the specimen. Presence of defect is indicated by variation in transmission signal. Method not shows exact location of damage.

(c) Contact type

- In the contact type, the probe is placed in direct contact with the test specimen with a thin liquid film used as a couplant for better transmission of ultrasonic waves in to the test specimen. Contact type subdivided into normal and angle beam technique
- In the **normal beam technique** the ultrasonic beam is projected perpendicularly in to the test specimen. This technique may use single, double or SE normal beam probes. With the single probe, the transducer of the probe acts as both transmitter and receiver.

- **The angle beam technique** is used to transmit ultrasonic waves in to a test specimen at a predetermined angle to the test surface.

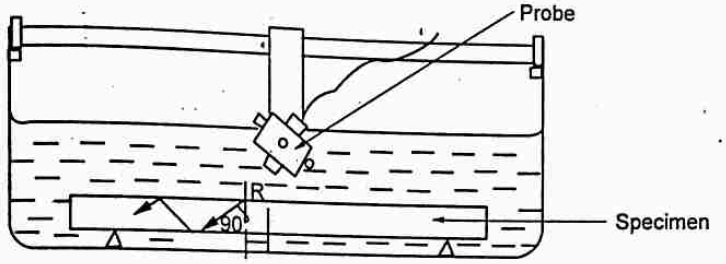


Fig. 3.35. The angle beam technique

D. IMMERSION TYPE

- In the immersion type, a waterproof probe is used at some distance from the test specimen and the ultrasonic beam is transmitted in to the material through a water path or water column.

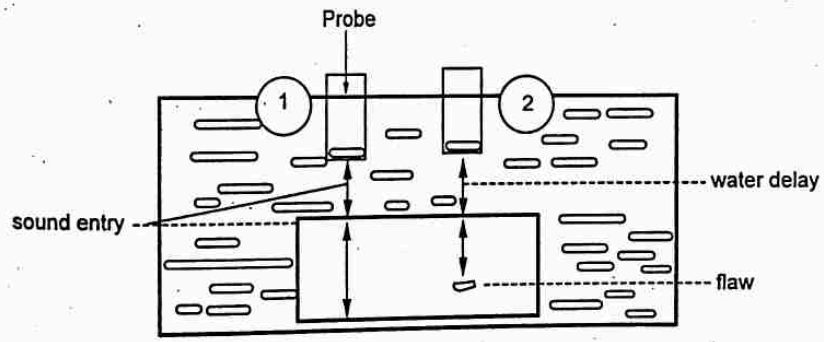


Fig. 3.36. immersion type

5. WORKING OF ULTRASONIC TESTING

- The technique detects internal, hidden discontinuities that may be deep below the surface.
- The sound energy is introduced and propagates through the materials in the form of waves.
- When there is a discontinuity (such as a crack) in the wave path, part of the energy will be reflected back from the flaw surface.
- The reflected wave signal is transformed into an electrical signal by the transducer and is displayed on a screen.

- Knowing the velocity of the waves, travel time can be directly related to the distance that the signal traveled.
- From the signal, information about the reflector location, size, orientation and other features can sometimes be gained.

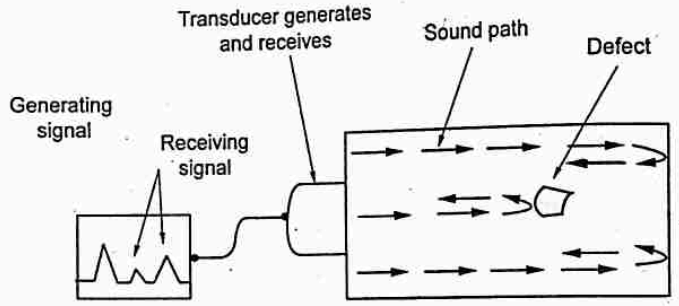


Fig. 3.37. Working of Ultrasonic testing

6. ADVANTAGES

- Capable of portable or highly automated operation.
- Can be performed on all types of materials.
- High accuracy and reproducibility in flaws detection.
- Generally only one surface needs to be accessible.
- Detection of surface and subsurface defects.
- Superior depth of penetration compared to other test methods.
- Minimal specimen preparation is required.
- Instantaneous results produced by using electronic equipment. Detailed images can be produced with automated systems.
- Non-hazardous to operations or nearby personnel.

7. DISADVANTAGES

- Surface must be accessible to probe and couplant.
- Skill and training required is more extensive than other technique.
- Surface finish and roughness can interfere with inspection.
- Thin parts may be difficult to inspect.
- Linear defects oriented parallel to the sound beam can go undetected.

- Cast iron and other coarse grained materials are difficult to inspect due to low sound transmission and high signal noise
- Linear flaws oriented parallel to the direction of the sound beam may go undetected
- Reference standards are required for equipment calibration and for the characterization of flaws.

8. APPLICATIONS

- Checking the quality of welds in pipes for the offshore oil industry
- Flaw detection and evaluation of materials
- Used in industries, Military defence, Offshore and marine industries
- Flaw detection (cracks, inclusions, porosity, delamination etc.)
- Corrosion/erosion wall thickness gauging
- Bond integrity assessment
- Estimation of grain size in metals
- Estimation of void content in composites and plastics

3.2.11. ACOUSTIC EMISSION

- This is a passive NDT technique, which relies on detecting the short bursts of ultrasound emitted by active cracks under a load. Sensors dispersed over the surface the structure detect the AE.
- It is even possible to detect from plasticization in highly stressed areas before a crack forms.

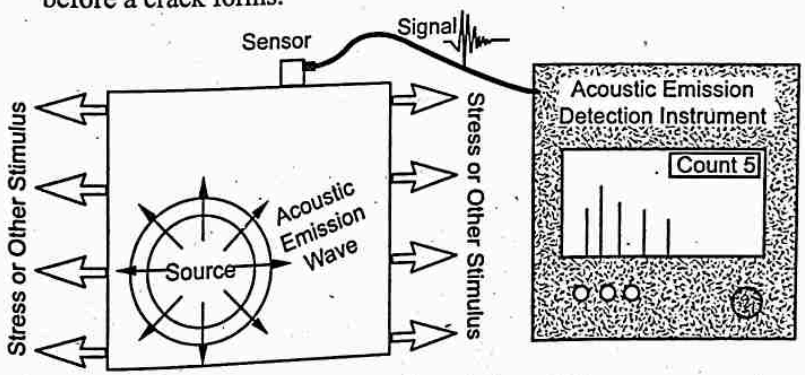


Fig. 3.38. Acoustic emission setup

- Frequently a method for use during proof tests of a pressure vessel; AE testing is also a continuous Structural Health Monitoring (SHM) method, for example on bridges. Leaks and active corrosion are detectable AE sources too.

1. PRINCIPLE

- Acoustic Emission (AE) refers to the generation of transient elastic waves produced by a sudden redistribution of stress in a material. When a structure is subjected to an external stimulus (change in pressure, load, or temperature), localized sources trigger the release of energy, in the form of stress waves, which propagate to the surface and are recorded by sensors.

2. BASIC COMPONENTS IN ACOUSTIC EMISSION TESTING

(a) Source

- The transient elastic waves that result from a sudden strain energy release within a material due to the occurrence of microstructural changes.
- Preamplifier is amplifies the initial signal.

(b) Sensor

- It is sensitivity in low ultrasonic frequency.
- The sensor can hear breaking of a single grain in metal, single fiber in fiber reinforced concrete and burst of tiny glass bubble in glass. It is made up of ceramic.

(c) Cable

- It transmit data Acoustic Emission device. Typically coaxial is used.

(d) Data acquisition device

- It performs filtration of signals parameter evaluation, data analysis and charting.

3. WORKING OF ACOUSTIC EMISSION

- Acoustic emission testing works by mounting small sensors onto a component under test.
- The component is stressed; the built-up state of stress spontaneously discharges at a leak, thereby generating sound impulses. As the damage grows in the component, there is a greater release of energy.

- The energy thus discharged is received by sensors applied on the surface of the tested object. The rates in which the acoustic emission is detected, the activity, and the intensity of the acoustic emission, the loudness, are monitored and used for assessing structural integrity and for health monitoring of components.
- The signals are received at different times by different sensors. By measuring these differences in time, the sound source can be located.

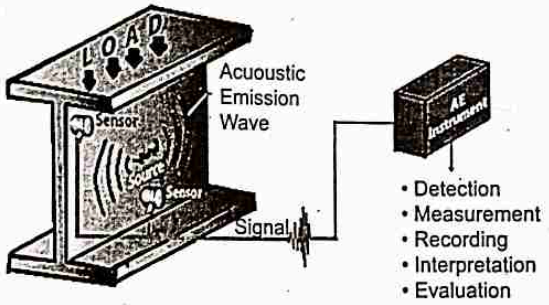


Fig. 3.39. Working of Acoustic Emission

3. ADVANTAGES

- High sensitivity.
- Early and rapid detection of defects, flaws, cracks etc.
- Real time monitoring.
- Cost Reduction test.
- Defective area location: only critical defects provide sustainable Acoustic Emission sources.
- Minimization of plant downtime for inspection, no need for scanning the whole structural surface. Minor disturbance of insulation.
- Ability to detect a range of damage mechanisms including, but not limited to, fiber breakages, friction, impacts, cracking, delamination and corrosion in their early stages, before they become significant issues.
- Can be conducted during operation, during qualification (proof) testing or development testing.
- Operational in hazardous environments, including high temperatures, high pressures and corrosive and nuclear environments.

- Can detect damages in defects that are difficult to access with conventional non-destructive testing techniques.

4. DISADVANTAGE

- Limited to assessing structural integrity or machine health by locating issues, further inspection is usually required to fully diagnose issues.
- Cannot detect defects that may be present, but that do not move or grow.
- Can be slower than other non-destructive testing techniques.

5. APPLICATION

- Continuous structural health monitoring (SHM) - bridges, metallic structures, mines, etc.
- Periodic testing - pressure vessels, pipelines, bridges, cables, storage tanks
- Transformer reliability monitoring
- Tube leak monitoring
- Structural integrity evaluation
- Tank bottom testing
- Corrosion detection
- Tube trailers & high pressure gas cylinders
- Reactor & high energy piping testing
- Aging aircraft evaluation

3.2.12. SELECTION OF NDT PROCESS

When planning an NDT inspection, following factors should made in account

- Cost.
- Economic criteria.
- Feasibility of NDT methods available.
- Quality assurance level achieved.
- The minimum detectable flaw size, shape, and orientation of the defect.
- The sensitivities and limitations of the NDT method.
- The type of damage (flaw, defect, discontinuity) mechanism to be inspected.

- Type of material to be tested.
- Where the defect is located (surface or internal).
- Working conditions and location.

1. RELIABILITY OF NONDESTRUCTIVE TESTING METHODS

- A simplified breakdown of the complexity and relative requirements of the five most frequently used NDT techniques is shown in table 3.2.

Table 3.2. Relative uses and merits of various nondestructive testing methods

Test method	Ultrasonics	X-ray	Eddy current	Magnetic particle	Liquid penetrant
Capital cost	Medium to high	High	Low to medium	Medium	Low
Consumable cost	Very low	High	Low	Medium	Medium
Time of results	Immediate	Delayed	Immediate	Short delay	Short delay
Effect of geometry	Important	Important	Important	Not too important	Not too important
Access problems	Important	Important	Important	Important	Important
Type of defect	Internal	Most	External	External	Surface breaking
Relative sensitivity	High	Medium	High	Low	Low
Formal record	Expensive	Standard	Expensive	Unusual	Unusual
Operator skill	High	High	Medium	Low	Low
Operator training	Important	Important	Important	Important	Less important

Test method	Ultrasonics	X-ray	Eddy current	Magnetic particle	Liquid penetrant
Training needs	High	High	Medium	Low	Low
Portability of equipment	High	Low	High to medium	High to medium	High
Dependent on material composition	Very	Quite	Very	Magnetic only	Little
Ability to automate	Good	Fair	Good	Fair	Fair
Capabilities	Thickness gaging; Some composition testing	Thickness gaging	Thickness gaging; Grade sorting	Defects only	Defects only

2. COMPARISON OF MAJOR NDT TEST

Table 3.3. Comparison of NDT test

Application	Characteristics detected	Advantages	Limitations	Example of use
Ultrasonic	Changes in acoustic impedance caused by cracks, non-bonds, inclusions, or interfaces	Can penetrate thick materials; excellent for crack detection; can be automated	Normally requires coupling to material either by contact to surface or immersion in a fluid such as water. Surface needs to be smooth.	Adhesive assemblies for bond integrity; laminations; hydrogen cracking

Application	Characteristics detected	Advantages	Limitations	Example of use
Radiography	Changes in density from voids, inclusions, material variations; Placement of internal parts is detected	Can be used to inspect wide range of materials and thicknesses; Versatile; Film provides record of inspection	Radiation safety requires precautions; Expensive; Detection of cracks can be difficult unless perpendicular to x-ray film.	Pipeline welds for penetration, inclusions, and voids; Internal defects in castings
Visual optical	Surface characteristics such as finish, scratches, cracks, or color; strain in transparent materials; Corrosion	Often convenient; Can be automated	Can be applied only to surfaces, through surface openings or to transparent material	Paper, wood, or metal for surface finish and uniformity
Eddy current	Changes in electrical conductivity caused by material variations, cracks, voids, or inclusions	Readily automated; Moderate cost	Limited to electrically conducting materials; limited penetration depth	Heat exchanger tubes for wall thinning and cracks
Liquid penetrant	Surface openings due to cracks, porosity, seams, or folds	Inexpensive, Easy to use, readily portable,	Flaw must be open to surface. Not useful on porous	Turbine blades for surface cracks or porosity;

Application	Characteristics detected	Advantages	Limitations	Example of use
		Sensitive to small surface flaws	materials or rough surfaces	Grinding cracks
Magnetic particle	Leakage magnetic flux caused by surface or near-surface cracks, voids, inclusions, or material or geometry changes	Inexpensive or moderate cost, both to surface and near- surface flaws sensitive	Limited to ferromagnetic material; Surface preparation and post-inspection demagnetization may be required	Railroad wheels for cracks; large castings

3.2.13. DISCONTINUITIES

- **Discontinuities:** Any imperfection or interruption in the normal physical structure or configuration of a product (cracks, laps, inclusion, etc). Discontinuity may or may not affect the usefulness of the product
- **Defect:** A discontinuity whose size, shape, orientation, location or properties makes it detrimental to the useful service of the product in which it occurs or exceeds the accept/reject criteria for the given design. Defect is a type of discontinuity.
- **Flaw:** It is defined as "an imperfection or discontinuity that may be detectable by nondestructive testing and is not necessarily rejectable." A flaw is also something that can occur in various sizes, shapes, orientations, locations, and can even only be isolated to a tiny portion of the material properties within a material volume.

Types of Discontinuities

- Inherent discontinuities - The discontinuities that originate during the initial casting process (when the metal is casted into ingots for further processing) and also it includes the discontinuities that are produced when metal is casted as parts of any given shape.
- Primary processing discontinuities - The discontinuities that originate during hot or cold forming processes (extrusion, forging, rolling, drawing, welding, etc.).
- Secondary procession discontinuities- The discontinuities that originate during grinding, machining, heat treating, plating and related finishing operations.
- Service discontinuities - The discontinuities that originate or develop while the component is in service. The service conditions (loading, mechanical and chemical environment, maintenance) of a component affect its expected life.

3.2.14. FACTORS INFLUENCING SENSITIVITY OF INSPECTION

The things can influence the sensitivity of inspections, but some of the main items to consider are: geometrical complexity, material density, surface roughness, and accessibility.

- **Geometrical complexity** - The simple rod, bar, or panel, something that can access from just about any direction, then flaw detection method will not be driven by geometrical complexity. The irregular shapes is largely affected during testing.
- **Material density** - Density and thickness of material can be critical to flaw sensitivity. Distinguishing a very small flaw can be nearly impossible in this situation.
- **Surface roughness** - The surface is where many of the inspections need to contact, so a rough surface makes for a difficult inspection, or no inspection at all.
- **Accessibility** - As with surface roughness, accessibility can quickly rule out UT and ET because you have to be able to have adequate probe contact. PT and MT are usually pretty good at limited areas of accessibility.

TWO MARK QUESTION WITH ANSWER

1. ***Define NDT.***
   - ❖ Non-destructive testing (NDT) is a testing and analysis technique used by industry to evaluate the properties of a material, component, structure or system for characteristic differences or defects and discontinuities without causing damage to the original part.
2. ***Write importance of NDT.***
   - ❖ To ensure product reliability
   - ❖ To ensure the safety of operation
   - ❖ To ensure customer satisfaction and to maintain the manufacturer's reputation
   - ❖ To control manufacturing processes and lower manufacturing costs
   - ❖ To maintain uniform quality level
3. ***What are the advantages of using NDT?***
   1. Reusable
   2. Safe
   3. Accurate
   4. Cost effective
   5. Quality control
4. ***What are the major 5 NDT methods?***

   The major 5 NDT Methods are:
   - ❖ Ultrasonic Testing
   - ❖ Radiography Testing
   - ❖ Magnetic Particle Testing
   - ❖ Dye Penetrant Testing
   - ❖ Eddy Current Testing
5. ***What are stages in NDT testing?***
   1. Testing
   2. Recording & Reporting
   3. Interpretation & Evaluation

6. ***Define Borescope.***
   - ❖ It is optical instrument for remove viewing of objects. Borescope can have various angles of view: 0° direct, 45° fore-oblique, 90° lateral and 110° retro.
   - ❖ Borescope consist of precision illumination system.
   - ❖ The size of the visual field usually varies with the diameter, for a given magnification system. The size of the visual field usually varies with the diameter, for a given magnification system.
7. ***What are the factors affecting the choice of NDT method***
   - ❖ Cost
   - ❖ Economic criteria.
   - ❖ Feasibility of NDT methods available.
   - ❖ Quality assurance level achieved.
   - ❖ The minimum detectable flaw size, shape, and orientation of the defect
   - ❖ The sensitivities and limitations of the NDT method
   - ❖ The type of damage (flaw, defect, discontinuity) mechanism to be inspected.
   - ❖ Type of material to be tested
8. ***What aids used for visual testing?***
   - ❖ Magnifying glasses
   - ❖ Fillet weld gauge-
   - ❖ Microscopes
   - ❖ Computer equipment (remote viewing)
   - ❖ Illuminated magnifier
   - ❖ Holography
9. ***Define liquid penetrant inspection***
   - ❖ It is based on the properties of surface wetting and capillary action, which causes a liquid to rise when confined to a small opening. After applying the penetrant and wiping away the excess, the penetrant that rises to the surface can indicate surface-breaking.

10. ***What is purpose penetrant in liquid penetrant inspection?***
    - ❖ The liquid, by capillary action, will penetrate the discontinuities and the excess remaining on the surface will be removed by a suitable cleaning system. It will be highly visible or fluoresce brightly to produce easy to see indications.
11. ***What is the role of developer in liquid penetrant inspection?***
    - ❖ The role of the developer is to pull the trapped penetrant material out of defects and spread it out on the surface of the part so it can be seen by an inspector.
12. ***Write the principle of working in magnetic particle testing?***
    - ❖ This NDT process uses magnetic fields to find discontinuities at or near the surface of ferromagnetic materials. The magnetic field can be created with a permanent magnet or an electromagnet, which requires a current to be applied.
    - ❖ The magnetic field will highlight any discontinuities as the magnetic flux lines produce leakage, which can be seen by using magnetic particles that are drawn into the discontinuity.
13. ***What are type of magnetization in magnetic particle testing?***

    Longitudinal magnetization- the magnetic flux flows from pole to pole, we call this longitudinal magnetisation. Discontinuities will be detectable once more at 90° (± 45°) to the flux direction.

    Circular magnetization - Circular magnetic field will be produced around the component at right angles to the direction of the electric current which produced it.
14. ***What are limitations of using magnetic particle testing?***
    - ❖ The specimen must be ferromagnetic (e.g. steel, cast iron)
    - ❖ Paint thicker than about 0.005" must be removed before inspection
    - ❖ Post cleaning and post demagnetization is often necessary
    - ❖ Insensitive to internal defects
    - ❖ Require magnetization and demagnetization of materials to be inspected
    - ❖ Require power supply for magnetization

- ❖ Coating may mask indication
- ❖ Material may be burned during magnetization

15. ***What are the major components used in thermography Method?***
    - ❖ Thermographic camera
    - ❖ Control unit
    - ❖ Pc/image processing unit
16. ***Define Pulsed thermography***

    It is a classical optical excitation thermography technique. In pulsed thermography, high-energy lamps are often used to produce a uniform heating source on the specimen surface.
17. ***What are the advantages of thermography Method compared to other NDT?***
    - ❖ Data collection system can record temperature changes with time
    - ❖ High-speed, portable, and non-contact
    - ❖ Ability to inspect large areas
    - ❖ Effective prevention of test scrap
    - ❖ Contactless testing with low thermal stress
18. ***Define Radiographic Testing.***
    - ❖ Radiographic Testing (RT) is a non-destructive testing (NDT) method which uses either x-rays or gamma rays to examine the internal structure of manufactured components identifying any flaws or defects.
19. ***How densities of material influence the radiographic testing?***
    1. If an object has a high density, ie a thicker object, it absorbs more radiation causing less radiation to hit the film, which produces a lighter image.
    2. If an object has a low density, ie when the through section is reduced or there is a lower-density material such as slag (compared to the surrounding material), it will absorb less radiation causing more radiation to hit the film, producing a darker image.
20. ***Define eddy current.***
    - ❖ An electromagnetic inductor is used to generate a magnetic field. When this field is introduced in the surface of the test piece, it generates so called "eddy currents" in the material.

21. *Difference between Digital Radiography and Conventional Radiography.*

Digital Radiography	Conventional Radiography
It uses a digital detector to display radiographic images on a computer screen almost instantaneously.	It uses a sensitive film which reacts to the emitted radiation to capture an image of the part being tested.
It allows for a much shorter exposure time so that the images can be interpreted more quickly.	The limitation to this technique is that films can only be used once and they take a long time to process.

22. *What are the types of Eddy current testing?*

- ❖ Pulsed Eddy Current (PEC)
- ❖ Tangential Eddy Current
- ❖ Conventional Eddy Current
- ❖ Eddy Current Array

23. *How the eddy current used for finding defects in the material?*

- ❖ The electrical currents are called eddy currents because the flow in circles at and just below the surface of the material.
- ❖ Interruptions in the flow of eddy currents, caused by imperfections, dimensional changes, or changes in the materials conductive and permeability properties, can be detected with the proper equipment like probs.

24. *What are the factors affecting eddy current inspection?*

- ❖ Material conductivity
- ❖ Permeability
- ❖ Frequency
- ❖ Geometry
- ❖ Proximity/Lift-Off
- ❖ Depth of Penetration
- ❖ Eddy Current Testing and Industry

25. *What are various application of eddy current testing?*

- ❖ Weld Inspection

- ❖ Conductivity Testing
- ❖ Surface Inspection
- ❖ Corrosion Detection

26. *Define ultrasonic testing.*

- ❖ Ultrasonic testing (UT) is a non-destructive testing techniques based on the propagation of ultrasonic waves (high frequency sound waves) are transmitted into materials to detect internal flaws or to characterize materials

27. *What is the principle of working in acoustic emission test?*

- ❖ Acoustic Emission (AE) refers to the generation of transient elastic waves produced by a sudden redistribution of stress in a material.
- ❖ When a structure is subjected to an external stimulus (change in pressure, load, or temperature), localized sources trigger the release of energy, in the form of stress waves, which propagate to the surface and are recorded by sensors.

28. *Differentiate between Radiography, Eddy current and Ultrasonic.*

Parameter	Radiography	Eddy current	Ultrasonic
Source	X ray , δ ray	Magnetic field	Ultrasonic wave made by piezoelectric or laser
Material	All types of engineering materials that do not absorb the whole wavelength of the ray	Only conductive materials, not cellular materials	All type of engineering materials (metals or plastics)
Geometry	Suitable for complex weld geometry	Need for special probes for different geometries	Need for special probes for different geometries

Parameter	Radiography	Eddy current	Ultrasonic
Type of defects and position	Surface and subsurface defects, all types of flaws. Not suitable for very fine defects	Surface and subsurface defects. Not suitable for deep flaws. All types of flaws	Surface and subsurface defects. Suitable for deep flaws. All types of flaws
Advantages	Determine the position and type of defects, ability for automation	Portable. Suitable for poor access areas. No need for paint or coat removing. No consumable Materials	Determine the length, location and type of defects. Portable.
Limitations	Poor resolution. Access to both sides of the part is required. The size of defect is not accurate	Defect direction, conductive materials, clean and smooth enough surface required	Defect direction Sometimes access to both sides or ends of the detail is required
Applications	Crack detection in weld pipeline	Crack detection of coated weld pipeline, inspecting of fatigue crack	Spot welding control, inspection of SAW in pipeline
Weld process (Example)	Electric resistance welding Laser beam welding Electron beam welding Gas metal arc welding	Laser beam welding Gas metal arc welding Gas tungsten arc welding	Resistance spot welding Gas metal arc welding Friction stir welding Laser beam welding Electron beam welding

29. *Define Inherent discontinuities.*

- The discontinuities that originate during the initial casting process (when the metal is casted into ingots for further processing) and also it includes the discontinuities that are produced when metal is casted as parts of any given shape.

30. *What are the advantages of using acoustic emission test?*

- High sensitivity.
- Early and rapid detection of defects, flaws, cracks etc.
- Real time monitoring
- Cost Reduction
- Defective area location: only critical defects provide sustainable Acoustic Emission sources.

REVIEW QUESTIONS

1. What are the varies advantages of using NDT test?
   Ans: Section No. 3.1 **Page No:** 3.2
2. Compare and contrast the major NDT test with various parameters.
   Ans: Section No. 3.2.12 **Page No:** 3.52
3. Explain the visual test with aids used, advantages and disadvantages.
   Ans: Section No. 3.2.1 **Page No:** 3.5
4. Explain the penetration test with step process and its application.
   Ans: Section No. 3.2.2 **Page No:** 3.10
5. What do you understand by NDT test? And explain the role of Nondestructive testing in manufacture process.
   Ans: Section No. 3.1 **Page No:** 3.1
6. What do you understand by magnetic hysteresis? Explain different magnetization technique using magnetic particle testing with their advantages and disadvantages.
   Ans: Section No. 3.2.5 **Page No:** 3.16
7. Classify the NDT methods. Justify any three methods.
   Ans: Section No. 3.2 **Page No:** 3.3

8. What is ultrasonic testing? Explain types of transducer.

   Ans: Section No. 3.2.10 **Page No:** 3.43

9. Write principle of radiography testing. Explain the working in detail.

   Ans: Section No. 3.2.7 **Page No:** 3.27

10. With output line diagram explain the ultrasonic flaw detector in detail.

    Ans: Section No. 3.2.10 **Page No:** 3.45

11. What is meant by thermography? Explain in detail.

    Ans: Section No. 3.2.6 **Page No:** 3.22

12. Why couplant used in Ultrasonic testing? Explain test with advantages and disadvantages.

    Ans: Section No. 3.2.10 **Page No:** 3.42, 3.46

13. Define eddy probe. Describe types and design of probes.

    Ans: Section No. 3.2.9 **Page No:** 3.39

14. Explain the radiography with safety measures.

    Ans: Section No. 3.2.7 **Page No:** 3.31

15. Write short note on

    - Eddy current testing
    - Acoustic emission testing

    Ans: Section No. 3.2.8, 3.2.11 **Page No:** 3.34, 3.47

16. Write a case study on NDT test used in welding industry.

17. The steel deck truss bridge in Trichy was 90-year-old, constructed across Kollidam river that connects Srirangam with mainland Tiruchirapalli. Use proper NDT test for monitoring bridge condition and explain step procedure of using NDT in bridge for structural health monitoring.

UNIT IV

MATERIAL CHARACTERIZATION TESTING

SYLLABUS

Macroscopic and Microscopic observations, Optical and Electron microscopy (SEM and TEM) - Principles, Types, Advantages and Limitations, Applications. Diffraction techniques, Spectroscopic Techniques, Electrical and Magnetic Techniques - Principles, Types, Advantages and Limitations, Applications.

4.1. OVERVIEW

- Characterization, when used in materials science, refers to the broad and general process by which a material's structure and properties are probed and measured. It is a fundamental process in the field of materials science, without this no scientific understanding of engineering materials could be determined.
- The Materials Characterization has a wide variety of characterization techniques in the areas of Microscopy, Spectroscopy, and Macroscopic techniques which help to increase the different degrees of understanding why different materials show different properties and behaviour.
- Materials characterizing are aimed at the features of materials quantitatively; this is often closely related to the analysis, modelling and simulation, and the qualitative characterization of materials through testing.
- **"Characterization describes those features of composition and structure (including defects) of a material that are significant for a particular preparation, study of properties, or use, and suffice for reproduction of the material."**

Material Characterization used for identification of,

- Contaminants

- Purity
- Active ingredients
- Polymer additives
- Fillers
- Solvents
- Failure Analysis
- Identification of Unknown Substances and Contaminants
- De-formulation and Reverse Engineering
- Material Comparisons
- Specialized Method Development

METHODS OF MATERIALS CHARACTERIZATION

- Chemical Characterization
- Toxicological Characterization
- Physical Characterization
- Electrical Characterization
- Morphological Characterization
- Mechanical Characterization

4.1.1. OBJECTIVES OF MATERIALS CHARACTERIZATION

- To measure accurately the physical properties of materials
- To measure accurately the chemical properties of materials
- To determine accurately the structure of a material at atomic and microscopic level structures

4.1.2. COMMON APPLICATIONS OF MATERIALS CHARACTERIZATION

- Surface Chemical Analysis
- Near Surface Chemical Analysis
- Atomic & NanoScale Chemical Analysis
- Surface Imaging

- Defect Analysis
- Analytical Imaging
- Non-Destructive Internal Imaging

4.1.3. CLASSIFICATION BASED ON APPLICATION

- From a realization of application, a classification of materials characterization methods can be outlined in a simplified manner.

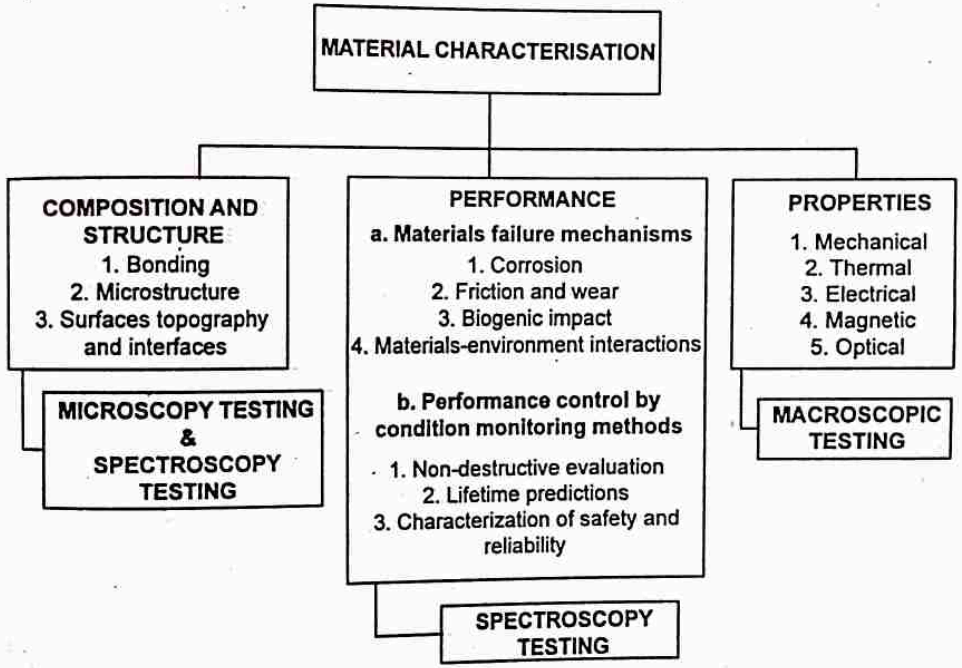


Fig. 4.1. Materials characterization chart

4.2. SCALE

- The scale of the structures observed in materials characterization ranges from angstroms, such as in the imaging of individual atoms and chemical bonds, up to centimeters, such as in the imaging of coarse grain structures in metals.
- The geometric length scale of materials has more than twelve orders of magnitude.

Table 4.1. Ranges of scale

Scale	Range
Nano scale	1 to 100 nanometers
Micro scale (Micro-devices and micro systems)	1 to 1000 micro- meters
Macro scale	Millimeter to the kilometer scale

4.3. MATERIAL CHARACTERIZATION TESTING

- Material characterization is the process of measuring and determining physical, chemical, mechanical and microstructural properties of materials.
- Based on scale of testing, there are two types,
  - Microscopy testing
  - Macroscopic testing
- Based on testing of composition, there are two types,
  - Spectroscopy & Nuclear spectroscopy

4.3.1. MICROSCOPY

- Microscopy is a technique that allows the determination of both the composition and the structure of a material.
- It is essentially the process of viewing the structure on a much finer scale not possible with the naked eye
- The properties of materials with extremely fine features and defects those are only possible to observe using microscopy techniques.

MICROSCOPIC PROPERTIES OF MATERIALS

- Contaminants & Purity
- Ingredients
- Chemical bonding
- Molecular pattern
- Crystal structure & lattice bonding
- Nano Size
- Ions etc.,

COMMON MICROSCOPY INSTRUMENTS INCLUDE

- **Optical Microscope**
- **Scanning Electron Microscope (SEM)**
- **Transmission Electron Microscope (TEM)**
- Field Ion Microscope
- Scanning Tunneling Microscope
- Scanning probe microscopy
- Atomic Force Microscope
- X-ray diffraction topography

4.3.2. SPECTROSCOPY & NUCLEAR SPECTROSCOPY

- This group of techniques uses a range of principles to reveal the chemical composition, composition variation, crystal structure and photoelectric properties of materials. Some common instruments include:
  - Ultraviolet-visible spectroscopy
  - Fourier transform infrared spectroscopy
  - Thermoluminescence
  - Photoluminescence
  - Energy-dispersive X-ray spectroscopy
  - Wavelength dispersive X-ray spectroscopy
  - Electron energy loss spectroscopy
  - X-ray photoelectron spectroscopy
  - Auger electron spectroscopy
  - X-ray Photon Correlation Spectroscopy

4.3.3. MACROSCOPIC

- In which some physical and chemical changes are observed. In this, changes can be observed by the naked eye.
- This simple process can yield a large amount of information about the material such as the colour of the material, its luster (does it display a metallic luster), its shape (whether it displays a regular, crystalline form), its composition (is it made up of different phases), its structural features (does it contain porosity) etc.

MACROSCOPIC PROPERTIES OF MATERIALS

- Density
- Volume
- Strength
- Hardness
- Roughness etc.,

COMMON MACROSCOPIC INSTRUMENTS INCLUDE

- Mechanical testing, including tensile, compressive, torsional, creep, fatigue, toughness and hardness testing
- Differential thermal analysis
- Dielectric thermal analysis
- Thermo gravimetric analysis
- Differential scanning calorimetry
- Impulse excitation technique
- Ultrasound techniques, including resonant ultrasound spectroscopy and time domain ultrasonic testing methods

4.4. BASIC TERMINOLOGY

4.4.1. MAGNIFICATION

- Magnification on a microscope refers to the amount or degree of visual enlargement of an observed object or enlargement of image.

Table 4.2. Methods of magnification

Method	Purpose	Examples
Relative Size Magnification	Increasing the actual size of the object being viewed	Larger print material
Relative Distance Magnification	Reducing the distance between the object and the eye	Move object closer to the eye
Angular Magnification	Increasing angular subtense of the image being viewed	Telescope, magnifier

- Magnification is measured by multiples, such as 2x, 4x and 10x, indicating that the object is enlarged to twice as big, four times as big or 10 times as big, respectively.
  - Magnification = Image ÷ Object

Table 4.3. Magnification vs instrument

Magnification	Instrument
1x	Naked eye
2x to 5x	Magnifying glass
10x to 20x	Stereoscopic microscope
50x to 1500 x	Upright/inverted microscope
2,000x to 1,000,000x	Electron microscope

4.4.2. RESOLUTION

- Resolution is defined as the ability to distinguish two very small and closely-spaced objects as separate entities.
- Resolution is determined by certain physical parameters that include the wavelength of light, and the light-gathering power of the objective and condenser lenses.

4.4.3. LENS

- The observation magnification is the product of the magnifications of each of the lenses. This generally ranges from 10x to 1,000x with some models even reaching up to 2000x magnification. Common types of lens include,

1. OBJECTIVE LENS

- The objective lens consists of several lenses to magnify an object and project a larger image. According to the difference of the focal distance, lenses of different magnifications are available, such as 4x, 10x, 40x, and 50x.
  - Achromatic lens
  - Semi-apochromatic lens (fluorite lens)
  - Apochromatic lens

- Plan lens
- Immersion lens

2. OCULAR LENS (EYEPIECE)

- A lens to be mounted on the observer side. The image magnified by the objective lens is further magnified by the ocular lens for observation. An ocular lens consists of one to three lenses and is also provided with a mechanism, called a field stop, which removes unnecessary reflected light and aberration.
- Different types are available according to the magnification they provide, such as 7x and 15x.
  - Huygens lens
  - Ramsden lens
  - Periplan lens
  - Compensation lens
  - Wide-field lens
  - Super-field lens

3. CONDENSER LENS

- A lens to be mounted under the stage. This lens can adjust the amount of light to uniformly illuminate objects. It is useful for observation at high magnification.
- There are various types of condenser lenses, ranging from general "abbe condensers" to "achromatic condensers" that correct color aberration.
  - Abbe condenser
  - Achromatic condenser
  - Universal condenser

4.4.4. ABERRATION

- Aberration is a property of optical systems such as lenses that causes light to be spread out over some region of space rather than focused to a point.
- Aberrations cause the image formed by a lens to be blurred or distorted, with the nature of the distortion depending on the type of aberration

- Chromatic aberration
- Spherical aberration.

4.4.5. NUMERICAL APERTURE

- The numerical aperture of a microscope objective is a measure of its ability to resolve fine specimen detail. The value for the numerical aperture is given by,
  - Numerical Aperture (NA) = $n \sin \alpha$

4.4.6. DEPTH OF FIELD

- Depth of field is the axial depth of the space on both sides of the object plane within which the object can be moved without detectable loss of sharpness in the image, and within which features of the object appear acceptably sharp in the image while the position of the image plane is maintained.

4.4.7. DEPTH OF FOCUS

- Depth of focus is the axial depth of the space on both sides of the image plane within which the image appears acceptably sharp while the positions of the object plane and of the objective are maintained.

4.5. OPTICAL MICROSCOPE

- The optical microscope, also referred to as a light microscope, is a type of microscope that commonly uses visible light and a system of lenses to generate magnified images of small objects.

1. PRINCIPLE

- The functioning of the light microscope is based on its ability to focus a beam of light through a specimen, which is very small and transparent, to produce an image. The image is then passed through one or two lenses for magnification for viewing. The transparency of the specimen allows easy and quick penetration of light.

2. CONSTRUCTION

- The object is placed on a stage and may be directly viewed through one or two eyepieces on the microscope.

3. TYPES OF MICROSCOPE

1. **Bright field microscope:** Transparent objects can be illuminated from below but the solid objects can be illuminated with light coming through and to produce a quality image. It is also known as a compound light microscope. Common types are,
   (*a*) **Simple microscope:** A simple microscope is a microscope that uses only one lens for magnification, and is the original light microscope. It is used to obtain small magnifications. A single biconvex lens magnifies the size of the object to get an enlarged virtual image
   (*b*) **Compound microscope:** The compound microscope uses a set of many lenses in order to maximize magnification. It magnifies the size of the object by a complex system of lens arrangement. It has a series of two lenses; the objective lens and the ocular lens, to magnify the size of the object.
2. **Dark field microscope:** The object is illuminated against a dark background.
3. **Polarized light microscope:** Polarized light may be used to determine crystal orientation of metallic objects.
4. **Phase-contrast microscope:** Phase-contrast imaging can be used to increase image contrast by highlighting small details of differing refractive index.
5. **Fluorescence Microscope:** It is used to view material stained with fluorescent dyes for specific purposes.
6. **Digital microscope:** A digital microscope is a microscope equipped with a digital camera allowing observation of a sample via a computer.

OTHER MICROSCOPE TYPES

There are many variants of the compound optical microscope design for specialized purposes. Some of these are physical design differences allowing specialization for certain purposes:

- **Stereo microscope,** a low-powered microscope which provides a stereoscopic view of the sample, commonly used for dissection.
- **Comparison microscope,** which has two separate light paths allowing direct comparison of two samples via one image in each eye.

- **Inverted microscope,** for studying samples from below; useful for cell cultures in liquid, or for metallography.
- **Epifluorescence microscope,** designed for analysis of samples which include fluoro phores.
- **Confocal microscope,** a widely used variant of epifluorescent illumination which uses a scanning laser to illuminate a sample for fluorescence.

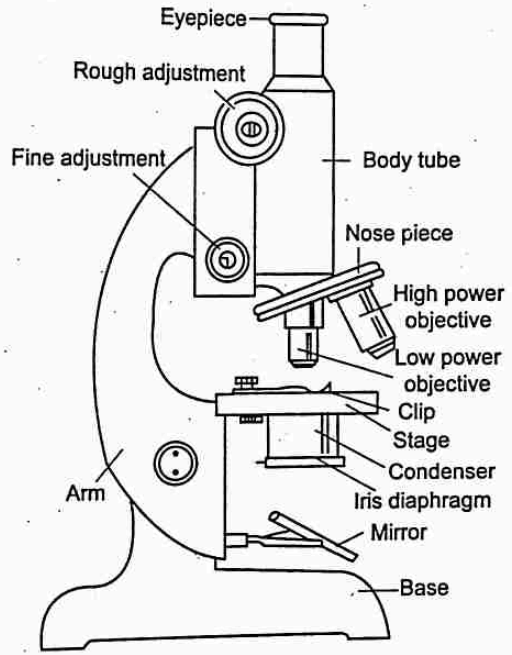


Fig. 4.2. Typical cross section of optical microscope

4. PREPARATION OF SPECIMEN

Basically preparation of specimen for optical microscopy is done by respective processes.

- Cut required part of specimen.
- Mount the specimen in mounting press.
- Grind the specimen as per to requirements.
- Polish the specimen.
- Etching.

5. COMPONENTS

- **Eyepiece (ocular lens):** It is a cylinder containing two or more lenses; its function is to bring the image into focus for the eye.
- **Objective turret** (or) revolver or revolving nose piece (to hold multiple objective lenses)
- **Objective lenses:** There will be around three objective lenses screwed. These arrangements are designed to be parfocal, which means that when one changes from one lens to another on a microscope, the sample stays in focus.
- **Diaphragm and condenser:** The condenser is a lens designed to focus light from the illumination source onto the sample.
- **Focus knobs** (to move the stage)
  - Coarse adjustment knob
  - Fine adjustment knob
- **Stage** (to hold the specimen)
- **Light source** (a light or a mirror)

6. WORKING

- The stage moves up and down when you turn a thumb wheel on the side of the microscope. By raising and lowering the stage, you move the lenses closer to or further away from the object you're examining, adjusting the focus of the image to see.
- The slide is held in place by two metal clips, one on either side.
- Light traveling up from the mirror passes through the glass slide, specimen, and cover slip to the objective lens (the one closest to the object). This makes the first magnification; it works by spreading out light rays from the specimen so they appear to come from a bigger object. The objective "lens" usually consists of more than one lens.
- A selection of other objective lenses can be used to magnify the specimen by more or less.
- The eyepiece lens (the one closest to your eye) magnifies the image from the objective lens, rather like a magnifying glass.

- On some microscopes, you can move the eyepiece up and down by turning a wheel. This gives you fine control or "fine tuning" of the focus.

7. MAGNIFICATION

- The maximum magnification power of optical microscopes is typically limited to around 1000x because of the limited resolving power of visible light.
- The magnification of a compound optical microscope is the product of the magnification of the eyepiece (say 10X) and the objective lens (say 100x), to give a total magnification of 1,000X.

8. ADVANTAGES

- Measuring microscopes are used for precision measurement.
- It is relatively easy to use.
- It is small and lightweight.
- It offers high levels of observational quality.
- It is unaffected by electromagnetic fields.
- It does not require radiation to operate.
- It requires very little training.
- It allow you to observe living organisms.
- It have a minor maintenance cost compared to other models.
- It can use fluorescent lights to display a sample visually.
- It is fully adjustable to the comfort level of the user.

9. DISADVANTAGES

- Resolution limit of optical microscopes .Due to diffraction, even the best classic optical microscope is limited to a resolution of 0.2 micro meters.
- Low magnification
- Separate sample Preparation
- Poor surface view
- Light microscopes cannot operate in darkness.
- Light microscopes cannot provide three-dimensional renderings.

10. APPLICATION

- Optical microscopy is used extensively in microelectronics, nanophysics, biotechnology, pharmaceutical research, mineralogy and microbiology.
- Optical microscopy is used for medical diagnosis.
- In industrial use, binocular microscopes are common.
- In certain applications, long-working-distance or long-focus microscopes are beneficial.
- An item may need to be examined behind a window, or industrial subjects may be a hazard to the objective.

4.6. ELECTRON MICROSCOPY

- An **electron microscope** is a microscope that uses a beam of accelerated electrons as a source of illumination.
- As the wavelength of an electron can be up to 100,000 times shorter than that of visible light photons, electron microscopes have a higher resolving power than light microscopes and can reveal the structure of smaller objects.

TYPES OF ELECTRON MICROSCOPE

- Transmission Electron Microscope
- Scanning Electron Microscope

4.6.1. SCANNING ELECTRON MICROSCOPE (SEM)

- A scanning electron microscope (SEM) uses a focused electro probe to extract structural and chemical information point by point on the specimen. It use wide range of scale from nanometer to micrometer.

1. PRINCIPLE

- A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons.
- The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample.

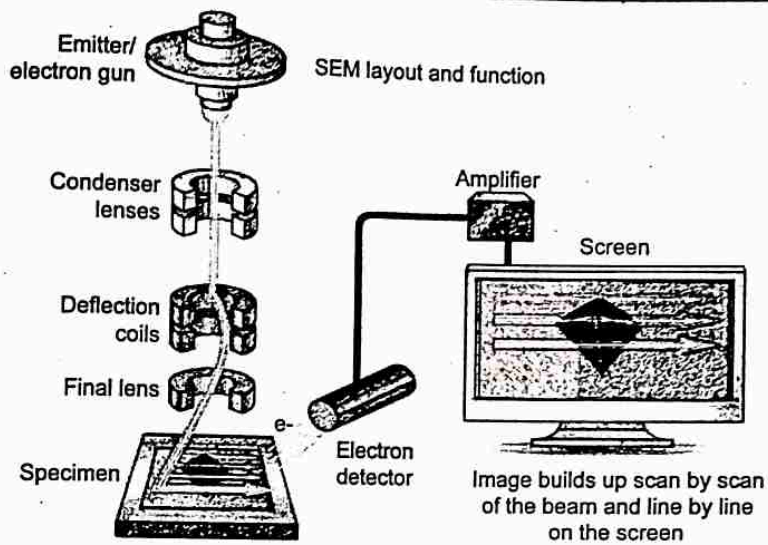


Fig. 4.3. Working nature of SEM

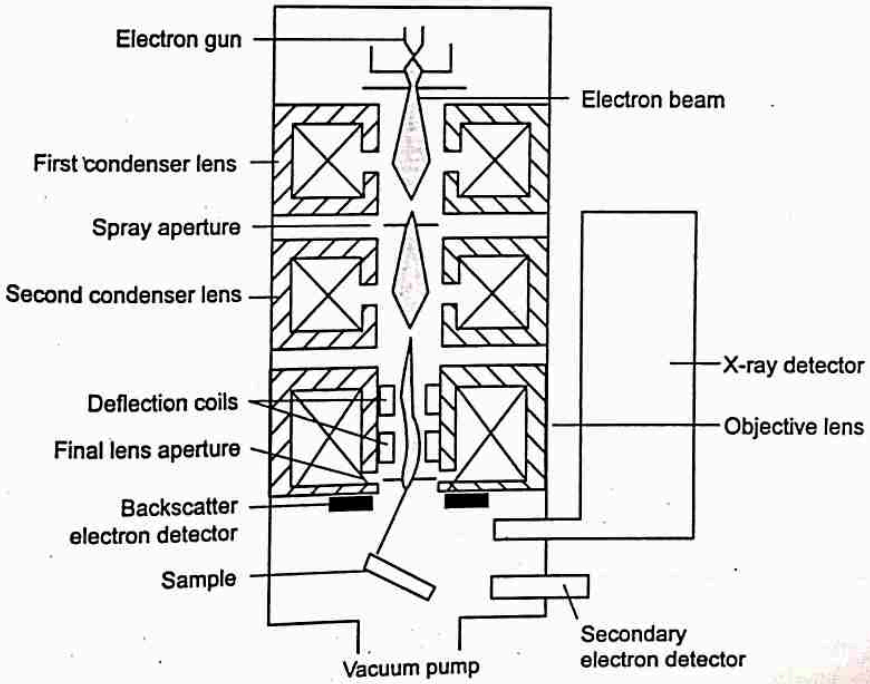


Fig. 4.4. Sectional view of SEM

2. COMPONENTS OF SEM

- **Electron gun:** It produces the high energy electron. Tungsten is normally used in guns because it has the highest melting point thereby allowing it to be electrically heated for electron emission, and its low cost.
- **Condenser lens:** It placed below electron gun. It is used to adjust the width (intensity) of the electron beam as per requirement. The main purpose is focusing the electron beam.
- **Vacuum chamber:** SEMs require a vacuum to operate. Without a vacuum, the electron beam generated by the electron gun would meet at constant interference from air particles in the atmosphere. The specimen chamber must be kept at a high vacuum of 10^{-3} to 10^{-4} Pa.
- **Deflector coils:** The scanning coils deflect the electron beam horizontally and vertically over the specimen surface. This is also called rastering.
- **Secondary electron detector:** A fluorescent substance (scintillator) is coated on the tip of the detector and a high voltage of about 10 kV is applied to it. The secondary electrons from the specimen are attracted to this high voltage and then generate light when they hit the scintillator. Then, the light is converted to electrons, and these electrons are amplified as an electric signal.
- **Image Display and Recording:** The output signals from the secondary electron detector are amplified and then transferred to the display unit.
- **Specimen stage** - The platform on which a specimen sits while being imaged.

3. CONSTRUCTION

- It consists of an electron gun. A magnetic condensing lens is used to condense the electron beam. The scanning coil is arranged in-between magnetic condensing lens and the sample.

4. SPECIMEN LOADING STAGES

- The specimen must meet the following requirements before it is loaded to the SEM stage:
  - Surface preparation
  - Mounting specimen
  - Specimen coating

(a) SURFACE PREPARATION

- **Fracturing-** When a specimen is a structural object, such as semiconductor device fracturing the specimen in this specific direction enables you to obtain a flat cross section.
- **Cutting-** If a specimen is soft like a polymer, it can be cut.
- **Mechanical polishing-** For many metal or mineral specimens, mechanical polishing is applied.
- **Milling by the ion beam-** A focused ion beam (FIB) system enables you to obtain a cross section with a high positional accuracy of a few hundreds of nanometers
- **Contrast enhancement-** Surfaces of cross sections are chemically or physically etched to form irregularity on the surface and internal structures are observed using secondary electron images.

(b) MOUNTING SPECIMEN

- **Bulk specimens-** Bulk specimens are fixed to the specimen mount by conductive paste or conductive double-sided adhesive tape. If a bulk specimen has a relatively uniform shape, it is clamped with an exclusive specimen holder.
- **Powders and particles-** These specimens are dusted on conductive paste or double-sided adhesive tape

(c) SPECIMEN COATING

- If a specimen is nonconductive, its surface needs to be coated with a thin metal film so that the surface has conductivity.

5. WORKING OF SEM

- In SEM an electron beam is emitted from an electron gun fitted with a tungsten filament cathode.
- The electron beam, which typically has an energy ranging from 0.2 keV to 40 keV, is focused by one or two condenser lenses to a spot about 0.4 nm to 5 nm in diameter.

- The beam passes through pairs of scanning coils or pairs of deflector plates in the electron column, which deflect the beam in the x and y axes so that it scans in a raster fashion (rectangular area) of the sample surface.
- When the primary electron beam interacts with the sample, the electrons lose energy by repeated random scattering and absorption.
- The energy exchange between the electron beam and the sample results in the reflection of high-energy electrons by elastic scattering, emission of secondary electrons, backscattered electrons and characteristic X-rays by inelastic scattering and the emission of electromagnetic radiation, each of which can be detected by specialized detectors.
- The beam current absorbed by the specimen can also be detected and used to create images of the distribution of specimen current.
- Electronic amplifiers of various types are used to amplify the signals, which are displayed as variations in brightness on a computer monitor.

6. OUTPUT-TYPES SCATTERED ELECTRONS

(a) X-RAYS

- X-rays, emitted from beneath the sample surface, can provide element and mineral information.

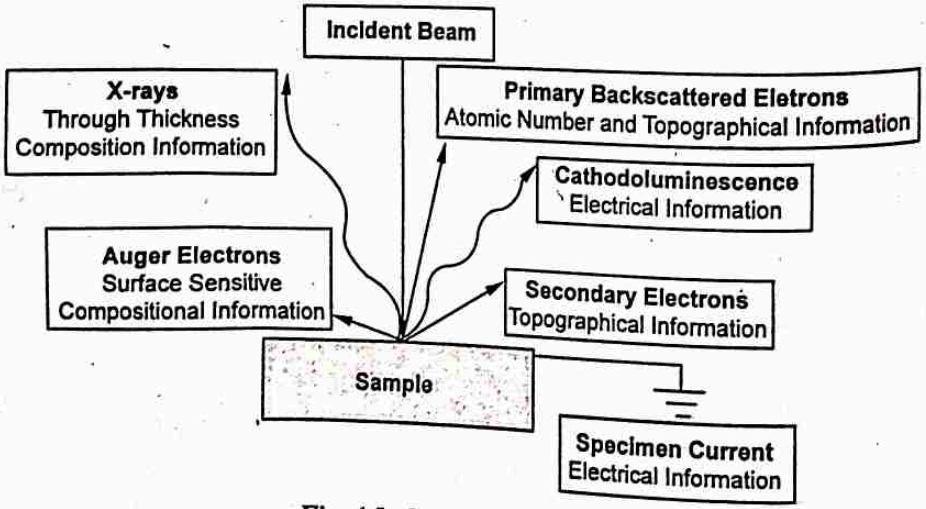


Fig. 4.5. Scattering Electrons

(b) SECONDARY ELECTRONS

- When the incident electron beam enters the specimen, secondary electrons are produced from the emission of the valence electrons of the constituent atoms in the specimen.
- Secondary electron image information used for surface morphology

(c) BACKSCATTERED ELECTRONS

- Backscattered electrons are those scattered backward and emitted out of the specimen, when the incident electrons are scattered in the specimen.
- This feature can be used to observe the topography of the surface.

7. MAGNIFICATION

- Magnification in an SEM can be controlled over a range of about 6X orders of magnitude from about 10 to 3,000,000 times.
- Magnification is therefore controlled by the current supplied to the scanning coils, or the voltage supplied to the deflector plates, and not by objective lens power.

8. APPLICATIONS

1. THE SEM ALSO EXCELS IN PRODUCING

- Detailed surface topography images
- Failure analysis
- Dimensional analysis
- Process characterization
- Reverse engineering
- Particle identification
- Surface 3D
- Elemental analysis

2. IDEAL USES

- High resolution surface topography images.
- Elemental microanalysis and particle characterization

9. ADVANTAGES

- Rapid, high-resolution imaging.

- Quick identification of elements present
- Excellent depth of field (~100X that of optical microscopy)
- Versatile platform that supports many other analysis techniques
- Low vacuum mode enables imaging of insulating and hydrated samples

10. LIMITATIONS

- Size restrictions may require cutting the sample.
- The size is not portable.
- SEMs are expensive and large.
- Maintenance involves keeping a steady voltage, currents to electromagnetic coils and circulation of cool water.
- SEMs are limited to solid, inorganic samples small enough to fit inside the vacuum chamber that can handle moderate vacuum pressure.
- SEMs carry a small risk of radiation exposure
- Training is required to operate.

4.6.2. TRANSMISSION ELECTRON MICROSCOPY (TEM)

- A **Transmission Electron Microscope** (TEM) utilizes energetic electrons to provide morphologic, compositional and crystallographic information on samples. The transmitted electrons that have passed through the thin sample are detected to form images, which is the reason to call it "transmission" electron microscopy.
- At a maximum potential magnification of 1 nanometer, TEMs are the most powerful microscopes.

1. PRINCIPLE

- An image is formed from the interaction of the electrons with the sample as the beam is transmitted through the specimen. The image is then magnified and focused onto an imaging device, such as a fluorescent screen, a layer of photographic film, or a sensor such as a scintillator attached to a charge-coupled device.

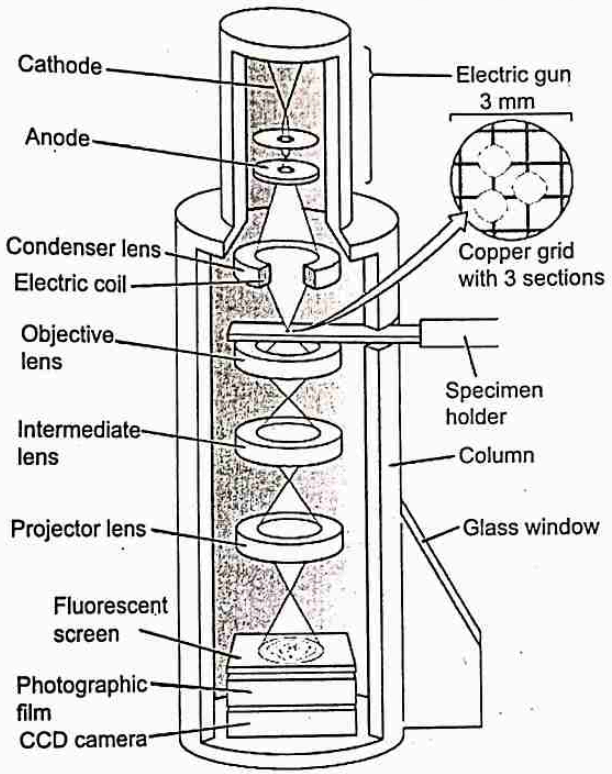


Fig. 4.6. Working of TEM

2. METHOD OF SPECIMEN PREPARATION

- **Ultra microtome:** Specimens must be very thin so that electrons are able to pass. This may be done by cutting very thin slices of a specimen's using an ultra-microtome.
- **Ultrasonic disk cutting:** For most electronic materials, it is a common sequence of preparation technique.
- **Dimpling**: Dimpling is a preparation technique that produces a specimen with a thinned central area and an outer rim of sufficient thickness to permit ease of handling.
- **Ion milling:** Ion milling is traditionally the final form of specimen preparation. In this process, charged argon ions are accelerated to the specimen surface by the application of high voltage. The ion impingement upon the specimen surface removes material as a result of momentum transfer.

- **Mechanical milling:** Mechanical polishing is also used to prepare samples for imaging on the TEM. A diamond, or cubic boron nitride polishing compound Polishing needs to be done to a high quality, to ensure constant sample thickness and to remove any scratches across the region of interest.
- **Chemical etching:** Certain samples may be prepared by chemical etching, particularly metallic specimens. These samples are thinned using a chemical etchant, such as an acid, to prepare the sample for TEM observation.
- **Ion etching:** Ion etching is a sputtering process that can remove very fine quantities of material. Ion etching uses an inert gas passed through an electric field to generate a plasma stream that is directed to the sample surface.
- **Replication:** It common use is for examining the fresh fracture surface of metal alloys.

3. CONSTRUCTION

- It consists of an electron gun. The specimen is placed in between the condensing lens and the objective lens. The magnetic projector lens is placed above the fluorescent screen.

4. COMPONENTS OF TEM

- **Electron Source** - The emission source or cathode, which may be a tungsten filament or needle. The gun is connected to a high voltage source (typically ~100 – 300 kV) and it emit electrons either by thermionic or field electron emission into the vacuum.
- **Electromagnetic lenses** - Electron lenses are designed to act similar like optical lens, by focusing parallel electrons at some constant focal distance. These focuses selected magnetic properties, such as magnetic saturation, hysteresis and permeability.
- **Vacuum chamber** - To increase the mean free path of the electron gas interaction, it is evacuated to low pressures, typically on the order of 10^{-4} Pa

- **Condensers** – Condensers consists of condenser lenses, the objective lenses, and the projector lenses. The condenser lenses are responsible for primary beam formation, while the objective lenses focus the beam that comes through the sample itself. The projector lenses are used to expand the beam onto the phosphor screen or other imaging device, such as film.
- **Sample stage** - Stage designs include specimen holder into the vacuum with minimal loss of vacuum in other areas of the microscope.

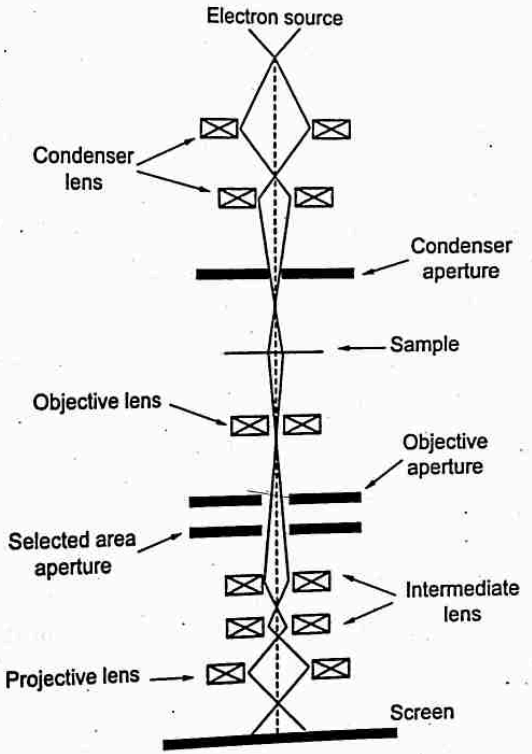


Fig. 4.7. Cross sectional view of TEM

- **Phosphor or fluorescent screen** - Imaging systems in a TEM consist of a phosphor screen, which may be made of fine (10 – 100 μm) particulate zinc sulfide, for direct observation by the operator and optionally an image recording system such as photographic film
- **Condenser lens** - The first electromagnetic lens that the electron beam encounters. Focuses the electrons onto the specimen.

- **Objective aperture** - A small laser-bored hole in a flat strip of molybdenum placed near the objective lens. Adjustment of this aperture strip can aid in adjustment of contrast of the image.

5. WORKING

- TEMs employ a high voltage electron beam in order to create an image.
- An electron gun at the top of a TEM emits electrons that travel through the microscope's vacuum tube.
- Rather than having a glass lens focusing the light, it employs an electromagnetic lens which focuses the electrons into a very fine beam.
- This beam then passes through the specimen, which is very thin (typically, sample thickness is less than 200 nm, depending on the composition of sample and the expected information from TEM characterization) and the electrons either scatter or hit a fluorescent screen at the bottom of the microscope.
- During transmission, the speed of electrons directly correlates to electron wavelength; the faster electrons move, the shorter wavelength and the greater the quality and detail of the image.
- An image of the specimen with its assorted parts shown in different shades according to its density appears on the screen. The image becomes visible when the electron beam hits a fluorescent screen at the base of the machine. This is analogous to the phosphor screen at the front of an old-fashioned TV.

6. OPERATION MODES OF TEM

- After interaction with the sample, on the exit surface of the specimen two types of electrons exist – unscattered (which will correspond to the bright central beam on the diffraction pattern) and scattered electrons (which change their trajectories due to interaction with the material).
- The two basic operation modes of TEM
  - Imaging mode
  - Diffraction mode

7. RESOLUTION

- TEMs can produce images with resolution down to 0.2nm. This resolution is smaller than the size of most atoms and therefore shows the true structural arrangement of atoms in the sample material.

8. LIMITATIONS

- Significant sample preparation time (1-4hrs)
- Small sampling volumes and samples are typically ~100nm thick.
- Some materials are not stable in the high energy electron beam
- TEMs are large and very expensive
- Laborious sample preparation
- Operation and analysis requires special training
- Samples are limited to those that are electron transparent, able to tolerate the vacuum chamber and small enough to fit in the chamber
- Images are black and white
- Electron microscopes are sensitive to vibration and electromagnetic fields and must be housed in an area that isolates them from possible exposure.
- A Transmission Electron Microscope requires constant upkeep including maintaining voltage, currents to the electromagnetic coils and cooling water.

9. ADVANTAGES

- The highest spatial resolution elemental mapping of any analytical technique (0.2nm (2Å) image resolution)
- Small area crystallographic information
- Strong contrast between crystalline vs amorphous materials without chemical staining.
- TEMs offer the most powerful magnification, potentially over one million times or more
- TEMs have a wide-range of applications and can be utilized in a variety of different scientific, educational and industrial fields
- TEMs provide information on element and compound structure

- Images are high-quality and detailed
- TEMs are able to yield information of surface features, shape, size and structure

10. APPLICATIONS

- Metrology at 0.2nm resolution
- Identification of nm-sized defects on integrated circuits, including embedded particles and via residues
- Determination of crystallographic phases at the nanometer scale
- Catalyst studies
- Nanometer scale elemental maps
- Super lattice characterization
- Crystal defect characterization (dislocations, grain boundaries, voids, stacking faults)
- Microstructure and nanostructure: size and morphology
- Crystal structure determination through electron diffraction
- Chemical information – composition and bonding (EDS, EELS) from single points, line scans or maps
- Energy filtered imaging (EFTEM)
- TEMs can be used in semiconductor analysis and production and the manufacturing of computer and silicon chips.
- Colleges and universities can utilize TEMs for research and studies.

4.6.3. COMPARISON BETWEEN SEM AND TEM

Table 4.4. Contrast Nature of SEM and TEM

Category	SEM	TEM
Source electrons	Scattered electrons	Transmitted electrons
Process of working	Scattering absorption	Diffraction
Energy	1-30kV	60-300kV

Category	SEM	TEM
Environment	Air/vacuum	Vacuum
Specimen thickness	Any thickness	Typically less than 150nm
Output	3D image formation	2D projection image of inner structure
Property identification	Roughness or contamination detection	Structural defects or impurities
Magnification	2 million level magnification	50 million level magnification.
Field of view	Large	Limited
Optimum resolution	0.4 nanometer resolution	0.5 angstroms resolution
Image formation	Electron are captured and countered by detector image on PC	Direct image on fluorescent screen or PC screen with LCD
Operation	Little sample preparation.	Laboratory sample preparation.
Amount of sample	Huge amount of sample	Minimum sample amount
Cost	Cost is low	Cost is 2 to 3 times higher than SEM
Sample usage	Invasive	Non-invasive, sample can used again

4.7. DIFFRACTION TECHNIQUES

4.7.1. DIFFRACTION

- Diffraction refers to various phenomena that occur when a wave encounters an obstacle or a slit. It is defined as the bending of waves around the corners of an obstacle or through an aperture into the region of geometrical shadow of the obstacle/aperture.

1. FUNDAMENTALS OF DIFFRACTION

- **Refraction** the change in direction of a wave passing from one medium to another caused by its change in speed.
- **Interference** the net effect of the combination of two or more wave trains moving on intersecting or coincident paths. The effect is that of the addition of the amplitudes of the individual waves at each point affected by more than one wave.
- **Reflection**, abrupt change in the direction of propagation of a wave that strikes the boundary between different mediums.
- **A diffraction grating** is an arrangement equivalent to a large number of parallel slits of equal widths and separated from one another by equal opaque spaces. They are two types reflection and transmission gratings.

2. DIFFRACTION PRINCIPLE

- **Bragg's law** is which determines the angles of coherent and incoherent scattering from a crystal lattice. When X-rays are incident on a particular atom, they make an electronic cloud move just like an electromagnetic wave.

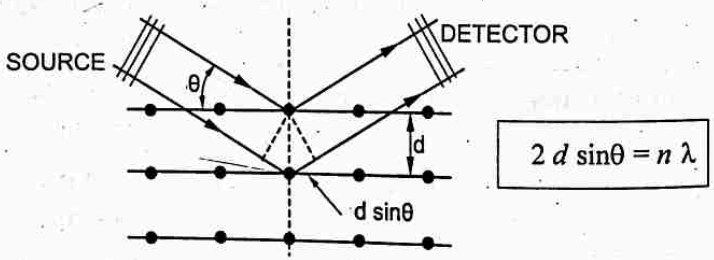


Fig. 4.8. Bragg's law

There are two conditions for constructive interference of waves:

1. The angle of incidence must equal the angle of reflection
2. The difference in path length must be an integral number of wavelengths

3. PATTERN OF DIFFRACTION

- **Fresnel's Diffraction:** Forms cylindrical wave front with source of screen at finite distance.

- **Fraunhofer diffraction:** Forms plane wave fronts with observation distance at infinite distance.

4. COMMON METHODS OF DIFFRACTION

1. Electron diffraction
2. Neutron diffraction
3. X-ray diffraction

5. FACTORS AFFECTING INTENSITY OF DIFFRACTION

1. Structure factor
2. Polarization factor
3. Lorentz factor
4. Multiplicity factor
5. Temperature factor
6. Absorption factor

6. ADVANTAGES, LIMITATION AND APPLICATION FOR COMMON METHODS OF DIFFRACTION

(a) ADVANTAGES

- Data generation is quick.
- Testing is cheap but equipment installation is costlier

(b) LIMITATION

- Sample preparation is complex.
- May have chance of absorption of radiation.
- Source is costlier.
- Most of diffraction method need vacuum.

(c) APPLICATION

- Diffraction methods offer a unique way to measure micro stresses in crystalline materials, because each phase will have its own diffraction pattern giving information on the stresses in that phase .
- It is also the measurement of changes in crystal plane spacing in different directions with respect to the specimen surface.

TERMS	X-RAY DIFFRACTION	NEUTRON DIFFRACTION	ELECTRON DIFFRACTION
ENERGY	❖ X-ray have energy, E=104 e V	❖ Neutrons have the energy, E =0.08 e V	❖ Electrons have the energy , E = 40 e V
ECONOMY	❖ X-ray is the cheapest the most convenient and widely used method.	❖ Neutron sources in the world are limited so neutron diffraction is a very special tool and very expensive.	❖ Electron beam can easily produce by cathode tube, and easily available.
INTERACTION	❖ X-rays interact with the spatial distribution of the valence electrons.	❖ Neutrons are scattered by the atomic nuclei through the strong nuclear forces.	❖ Electrons are charged particles and interact with matter through the Coulomb forces (positively charged atomic nuclei).
SCATTERING	❖ Atomic scattering power decreases as scattering angle increase.	❖ Atomic scattering power decreases as increases angle.	❖ Atomic scattering power is change erratically with angle.
METHODS	❖ 1. Powder Diffraction ❖ 2. Single-Crystal Diffraction	❖ 1.Nuclear Scattering ❖ 2.Magnetic Scattering	❖ 1. Diffraction/ Elastic Scattering ❖ 2. Inelastic Scattering

COMMON METHODS OF DIFFRACTION TECHNIQUES

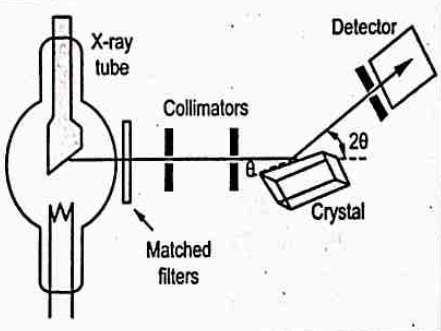
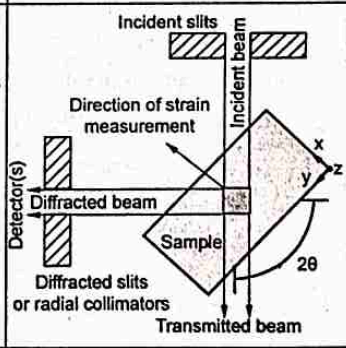
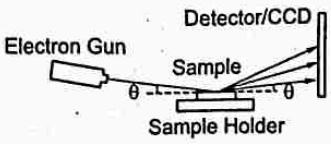
Table 4.5. methods of diffraction techniques

TERMS	X-RAY DIFFRACTION	NEUTRON DIFFRACTION	ELECTRON DIFFRACTION
DEFINITION	❖ X-ray diffraction (XRD) OR X-ray crystallography (XRC) is the determination of the atomic and molecular structure of a crystal, in which the crystalline structure causes a beam of incident X-rays to diffract into many specific directions. ❖ It produce a three-dimensional picture of the density of electrons within the crystal.	❖ Neutron diffraction or elastic neutron scattering is the application of neutron scattering to the determination of the atomic and/or magnetic structure of a material. ❖ A sample to be examined is placed in a beam of thermal or cold neutrons to obtain a diffraction pattern that provides information of the structure of the material.	❖ Electron diffraction is similar to X-ray diffraction (XRD) is the phenomenon resulting from the interaction between electrons and crystalline materials, producing a pattern of rings or spots that characterize the sample
WAVELENGTH	❖ Wavelength needed for crystal diffraction of the order of λ=1Å which is same size as an atom	❖ Wavelength needed for crystal diffraction of the order of λ = 2Å	❖ Wavelength needed for crystal diffraction of the order of λ = 1Å

TERMS	X-RAY DIFFRACTION	NEUTRON DIFFRACTION	ELECTRON DIFFRACTION
STRENGTHS	❖ Powerful and rapid (< 20 min) technique for identification of an unknown mineral ❖ Useful for unambiguous mineral determination ❖ Minimal sample preparation is required ❖ XRD units are widely available ❖ Data interpretation is relatively straight forward ❖ Measurement under atmosphere pressure. ❖ X-ray are not observed very much by air, so the specimen need not be in evacuated chamber	❖ Momentum transfer around interatomic distance ❖ Highly penetrating: measure bulk properties, can benefit from large samples, extreme sample environment (high/low temperature, magnetic field, pressure etc.,) ❖ Polarization is possible ❖ Neutrons interact with unpaired electrons. ❖ Magnetic structure and spin excitations can be studied	❖ Crystal cell symmetry ,cell parameters can be easily extracted from electron diffraction patterns ❖ Diffracted beam have high intensity. ❖ Can handle nano -size crystals. ❖ Small amount of material needed.
LIMITATIONS	❖ Gives better result only for homogeneous and single phase material	❖ Low intensity or resolution, large samples, and statistical noise.	❖ Sample size and preparation is tedious. ❖ The material must be less

TERMS	X-RAY DIFFRACTION	NEUTRON DIFFRACTION	ELECTRON DIFFRACTION
WORKING	❖ X-rays are generated in a cathode ray tube by heating a filament to produce electrons ❖ When electrons have sufficient energy to dislodge inner shell electrons of the target material, characteristic X-ray spectra are produced. ❖ As the sample and detector are rotated, the intensity of the reflected X-rays is recorded.	❖ The sample is placed within a neutron beam and the angles at which the neutrons are deflected or scattered by the material are recorded to generate a "diffraction pattern" from which structural information can be extracted.	❖ This experiment involves directing a beam of electrons through a carbon target, scattering the electrons, and analyzing the pattern produced on a luminescent screen.
ELEMENTS	❖ X-Ray Tube (Source) ❖ Sample Holder ❖ X-Ray Detector	❖ Neutron Generator ❖ Vacuum Pump. ❖ Sample Holder Quartz ❖ Detector	❖ The Electron Gun ❖ Carbon Target ❖ Luminescent Screen
			❖ 3. Small Angle Scattering ❖ 4.Reflectometry Diffraction

TERMS	X-RAY DIFFRACTION	NEUTRON DIFFRACTION	ELECTRON DIFFRACTION
	widely used for the identification of unknown crystalline materials (e.g. minerals, inorganic compounds). ❖ Determining lattice mismatch between film and substrate and to inferring stress and strain ❖ Determining dislocation density and quality of the film by rocking curve measurements ❖ Measuring super lattices in multilayered epitaxial structures ❖ Determining the thickness, roughness and density of the film using glancing incidence x-ray reflectivity measurements ❖ Make textural measurements, such as the orientation of grains, in a polycrystalline sample	structure ❖ Locating Light atoms ❖ Heavy atoms that absorb x-ray strongly ❖ Similar atomic no / Isotopes are studied ❖ Magnetic properties and single crystal study analysis	density

TERMS	X-RAY DIFFRACTION	NEUTRON DIFFRACTION	ELECTRON DIFFRACTION
	❖ Requires standard reference for inorganic compounds ❖ Requires tenths of a gram of material which must be ground into a powder. ❖ For mixed materials, detection limit is ~ 2% of sample ❖ Non-isometric crystal is complicated ❖ Peak overlay may occur	❖ Penetrating background hard to control and need large samples ❖ Some elements strongly absorb ❖ Hard to manipulate, accelerate, detect, etc.,	than 200 nm thick in order to pass the electron beam through. ❖ Observation of very small portion of the material.
			
APPLICATION	❖ X-ray powder diffraction is most	❖ Used for determination of	❖ It is used to assess the defect

4.8. SPECTROSCOPY TECHNIQUES

1. SPECTRUM

- Spectrum is a plot of the response as a function of wavelength or more commonly frequency.

2. SPECTROSCOPY

- Spectroscopy deals with the production, measurement, and interpretation of spectra arising from the interaction of electromagnetic radiation with matter.
- Spectroscopic methods are very informative and widely used for both quantitative and qualitative analysis.

3. SPECTROMETRY

- It is the measurement of these **Spectrum** responses and an instrument which performs such measurements is a spectrometer or spectrograph.

4. SPECTROPHOTOMETRY

- Spectrophotometry is a quantitative approach of measuring the relative energy i.e. emitted, transmitted or reflected in the visible or UV regions as a function of wave length or wave number.

5. ELECTROMAGNETIC SPECTRUM

- Electromagnetic radiation is a form of energy that is transmitted through space at enormous velocities. Electromagnetic radiation, or light, is described by the properties of both waves and particles nature.
- In dealing with phenomena such as reflection, refraction, interference, and diffraction, but electromagnetic radiation is conveniently modeled as waves.
- An electromagnetic wave is characterized by several fundamental properties, including its frequency, velocity, amplitude, phase angle, polarization, and direction of propagation.
- The entire electromagnetic spectrum, from the lowest to the highest frequency (longest to shortest wavelength), includes all **radio waves (e.g., commercial radio and television, microwaves, radar), infrared radiation, visible light, ultraviolet radiation, X-rays, and gamma rays.**

Nearly all frequencies and wavelengths of electromagnetic radiation can be used for spectroscopy.

Table 4.6. Distinct feature of electromagnetic radiation

Name of Spectroscopy	Type of Radiation used	Wavelength	Relative Energy	What it does to the molecule/atom	What it tells us about the atom/molecule
Photoelectron Spectroscopy	X-rays	0.01 to 10 nm	Very high	Removes core electrons	In Atomic structure, it gives information about how tightly the electrons are held by the nucleus
UV-visible Spectroscopy	Ultraviolet	50-400 nm	High	Excites valence electrons	Identify of a molecular element
UV-Visible Spectroscopy	Visible light	400 - 800 nm	Medium	Excites valence electrons	Concentration of a molecule
IR (vibrational) Spectroscopy	Infrared	2.5-50 μm	Low	Changes the vibrations in covalent bonds	Types of bonds/atoms/ functional groups within a molecule
Microwave (rotational) Spectroscopy	Microwave	0.3 mm - 0.5 m	Very low	Changes the rotations of the atoms in covalent bonds	Location of hydrogen atoms within a molecule

6. PRINCIPLE OF SPECTROSCOPY

- The beam of electromagnetic radiation onto a sample, and observe how it responds to such a stimulus. The response is usually recorded as a function of radiation wavelength. A plot of the response as a function of wavelength is referred to as a spectrum.

- The Beer-Lambert law states that the quantity of light absorbed by a substance dissolved in a fully transmitting solvent is directly proportional to the concentration of the substance and the path length of the light through the solution.

7. COMMON METHODS OF SPECTROSCOPY

- The method of Spectroscopy differ with respect to the species to be analyzed (such as molecular or atomic spectroscopy), the type of radiation–matter interaction to be monitored (such as absorption, emission, or diffraction), and the region of the electromagnetic spectrum used in the analysis.
- Spectroscopic methods based on the absorption or emission of radiation in the ultraviolet (UV), visible (VIS), infrared (IR), and radio (nuclear magnetic resonance, NMR)
- Each of these methods is distinct in that it monitors and different types of molecular or atomic transitions.

8. COMMON TYPE OF SPECTROSCOPY

- Ultraviolet-visible spectroscopy (UV-vis)
- Electron Spin Resonance spectroscopy
- Atomic spectroscopy
- Infrared spectroscopy and Raman spectroscopy
- Mass spectrometry
- Nuclear spectroscopy(nuclear magnetic resonance)

9. APPLICATION OF SPECTROSCOPIC ANALYSIS

- Understanding constitution of matter from atoms to complex molecules
- Studies on diverse materials existing in nature from deep sea studies to space missions
- Investigations of crime samples
- Analysis and development of whole range of man-made materials of human consumption
- Studies on environmental samples
- Mineralogy

10. ADVANTAGES OF SPECTROSCOPIC ANALYSIS

- Cure monitoring of composites using optical fibers.
- Estimate weathered wood exposure times using near infrared spectroscopy.
- Measurement of different compounds in food samples by absorption spectroscopy both in visible and infrared spectrum.
- Measurement of toxic compounds in blood samples
- Non-destructive elemental analysis
- Electronic structure research with various spectroscopes.
- Quantitative and qualitative analysis

11. DISADVANTAGES OF SPECTROSCOPIC ANALYSIS

- The radiation may be easily contaminated
- Cost of spectroscopy equipment is high.
- Not suitable for all kind of material.
- Need low working temperature at certain condition.

4.8.1. TYPES OF SPECTROSCOPY

4.8.1.1. ATOMIC SPECTROSCOPY OR FLAME SPECTROSCOPY

- Atomic spectroscopy is based upon the absorption or emission of electromagnetic radiation by atomic particles. Spectroscopic determination of atomic species can only be performed on a gaseous medium in which the individual atoms or elemental ions. This method is widely applied to a wide range of metals and nonmetals.
- Energy transitions of outer electrons of atoms after volatilization in a flame
- Liquid solution saimples are aspirated into a burner or nebulizer/burner combination, desolvated, atomized, and sometimes excited to a higher energy electronic state.
- Atomic spectroscopy is the study of the electromagnetic radiation absorbed and emitted by atoms.

1. PRINCIPLE

- The electrons of the atoms in the atomizer can be promoted to higher orbitals for a short amount of time by absorbing a set quantity of energy (i.e. Light of a given wavelength).

- This amount of energy is specific to a particular electron transition in a particular element, and in general, each wavelength corresponds to only one Element. This gives the technique its elemental selectivity.

2. TYPES

- **Absorption** - Light of a wavelength characteristic of the element of interest radiates through the atom vapor. The atoms absorb some of the light. The amount absorbed is measured.
- **Emission** - Sample is heated to excitation/ionization of the sample atoms. Excited and ionized atoms decay to a lower energy state through emission. Intensity of the light emitted is measured.
- **Fluorescence** – A short wavelength is absorbed by the sample atoms, a longer wavelength (lower energy) radiation characteristic of the element is emitted and measured.

3. COMPONENTS

- Lamp source - Hollow cathode lamp or diode laser source
- Nebulizer - The nebulizer forms a mist or aerosol of the sample; this is done by forcing the sample at high velocities through a narrow tube
- Atomizer- Atomize sample to atomic state
- Monochromator - Responsible for production of narrow band of radiation
- Detector - Photo sensitive element

Table 4.7. Comparative of AAS and AES

Activities	Atomic absorption spectroscopy	Atomic emission spectroscopy
Process measured	Absorption (light absorbed by unexcited atoms)	Emission (light emitted by exited atoms)
Use of flame	Atomization	Atomization and excitation
Instrumentation	Uses light source	Do not use light source (independent of light source)

4. CONSTRUCTION & WORKING

- The first step in all atomic spectroscopic procedures is atomization, a process in which a sample is volatilized and decomposed to produce gas-phase atoms and ions.
- Atomization is a critical step in all atomic spectroscopy. Several methods are used to atomize samples for atomic spectroscopic studies. E.g. inductively coupled plasmas, flames, and electro thermal atomizers; Flames and electro thermal atomizers are widely used in atomic absorption spectrometry, while the inductively coupled plasma is employed in optical emission and in atomic mass spectrometry.

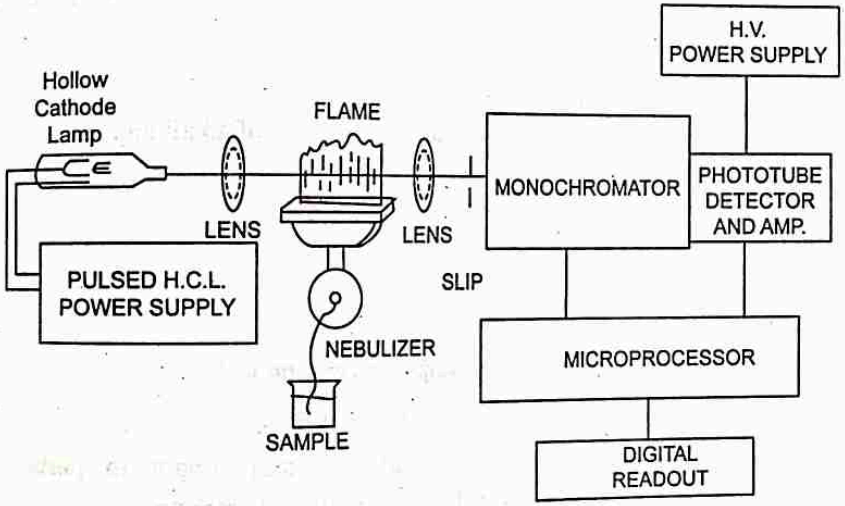


Fig. 4.9. Working of flame spectroscopy

- In the components of an atomic absorption or flame absorption apparatus, the flame can be considered to be a dilute gaseous solution of the atomized sample held in place by the aspirator-burner. Radiation from a suitable source is passed through the atomized sample and into the slit of a photometer or spectrophotometer.
- Radiation of specific wavelength is emitted by the hollow cathode lamp onto the gaseous atoms in the atomizer
- The monochromator focuses the specific wavelengths onto the detector.
- The detector finds the amount of light absorbed.
- The concentration of atoms in the sample is directly proportional to the absorbance.

5. APPLICATIONS

- Level of metals could be detected in tissue samples like Aluminum in blood and Copper in brain tissues
- Presence of metals as an impurity or in alloys could be found easily
- Determination of elements in the agricultural and food products
- Determination of lead in petrol
- Determination of calcium and magnesium in cement.
- AAS is an analytical technique used for the qualitative and quantitative
- Determination of the elements present in different samples like food, water and wastewater sample, nanomaterial, biomaterials, forensics (blood sample), and industrial wastes.
- Qualitative and quantitative analysis of metals.
- Emission techniques are for routine determination of alkali metals.

6. ADVANTAGES

- High sensitivity
- Easy to use
- Inexpensive
- The method of analysis is very simple and economical.
- It is quick, convenient, selective and sensitive analysis.
- Even very low concentrations (parts per million/ppm to parts per billion/ppb range) of metals in the sample can be determined.
- This method compensates for any unexpected interfering material present in the sample solution.
- This method can be used to estimate elements which are rarely analysed.

7. DISADVANTAGES

- Different cathode lamp for different elements
- Can detect only metals and some non-metals
- Only one element detected
- The elements such as carbon, hydrogen and halides cannot be detected due to their non-radiating nature.
- The accurate concentration of the metal ion in the solution cannot be measured.

- It cannot directly detect and determine the presence of inert gases.
- It does not provide the information about the molecular structure of the metal present in the sample.
- Only liquid samples may be used.

4.8.1.2. UV/VISIBLE SPECTROSCOPY

- UV-V is Spectrometry is based upon absorption of electromagnetic radiation in the visible and ultraviolet regions of the spectrum resulting in changes in the electronic structure of ions and molecules. The wavelength of UV and visible light are substantially shorter than the wavelength of infrared radiation. The UV–V is spectrum ranges from 200 to 700 nm. When a molecule or ion absorbs ultraviolet or visible radiation it undergoes a change in its valence electron transition.

1. PRINCIPLE

- Diminution of a beam of light after it passes through a sample or after reflection from a sample surface. Absorption measurements can be at a single wavelength or over an extended spectral range.
- Energy transitions of bonding and non-bonding outer electrons and molecules, usually delocalized electrons.

2. COMPONENTS

- Light Source - Tungsten filament lamps (or) Hydrogen-Deuterium lamps
- Monochromator- Monochromators generally is composed of prisms and slits. The radiation emitted from the primary source is dispersed with the help of rotating prisms. The various wavelengths of the light source which are separated by the prism are then selected by the slits such the rotation of the prism results in a series of continuously increasing wavelength to pass through the slits for recording purpose. The beam selected by the slit is monochromatic and further divided into two beams with the help of another prism.
- Detector-One of the photocell receives the beam from sample cell and second detector receives the beam from the reference.
- Recording devices- Computer stores all the data generated and produces the spectrum of the desired compound.

3. WORKING

- Polychromatic light from the source is focused on the entrance slit of a monochromator, which selectively transmits a narrow band of light. This light then passes through the sample area to the detector. The absorbance of a sample is determined by measuring the intensity of light reaching the detector without the sample (the blank) and comparing it with the intensity of light reaching the detector after passing through the sample.

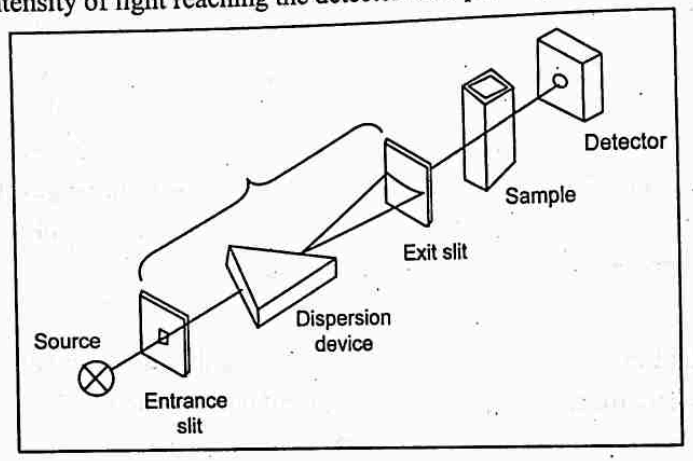


Fig. 4.10. Working of UV spectroscopy

4. APPLICATION

- Routine qualitative and quantitative measurement.
- Used to find relative purity of a solution.
- Widely applicability to both organic and inorganic compounds.

5. ADVANTAGES

- Minimum damage to sample.
- Better result at lower concentration.
- Very rapid calibration.
- High sensitivity
- Good accuracy

6. DISADVANTAGES

- Lack of sensitivity.
- Instrument is expansive.
- Have limited application to identify the functional group or particular molecule as a result of absorption spectra.

Table 4.8. Common methods of spectroscopy

Method	Definition & Principle of Working	Application	Advantages	Disadvantages
Infrared (IR) spectroscopy	❖ Infrared (IR) spectroscopy or vibrational spectroscopy is an analytical technique that takes advantage of the vibrational transitions of a molecule. ❖ When light impinges upon a molecule and interacts with the electron cloud & the bonds of that molecule. The incident photon excites the molecule einto a virtual state.	❖ NanoScale semiconductor analysis ❖ Identification of compounds ❖ Quantitative analysis ❖ Information regarding functional groups of molecules and constitution of molecules can be deduced from IR spectrum ❖ Qualitative analysis and fingerprinting of purified molecules of intermediate size.	❖ The crystalline structures can be obtained for smaller molecules ❖ Used for microscopic analysis ❖ Comparatively cheaper	❖ Molecular structures are too complex to study. ❖ The radiation deforms easily. ❖ The low operation temperature of 15 - 90 K. ❖ Lower ,sensitivity, because scattering effect is weaker ❖ Not suited for aqueous solutions. ❖ Sample preparation necessary

Mass spectroscopy	❖ The sample is converted to rapidly moving positive ions by electron bombardment and charged particles are separated according to their masses. ❖ Determination of the abundance of positively ionized molecules and fragments.	❖ Mainly used in research, but has high potential in metallurgy field. ❖ To find surface analysis of components.	❖ Small sample size. ❖ Fast method. ❖ Nondestructive ion detection. ❖ It does not absorb or emit light. ❖ High sensitivity.	❖ Difficult to non-volatile component. ❖ It does not provide structural information. ❖ Expansive equipment
Nuclear Magnetic Resonance spectroscopy	❖ The alignment (polarization) of the magnetic nuclear spins is applied in an constant magnetic field. ❖ Detection of magnetic moment associated with an odd number	❖ To study molecular physics, crystals and non-crystalline materials. ❖ Good choice for analyzing dangerous samples. ❖ The prediction results are provided to	❖ Easy to find 3d structure. ❖ The motion of domains can be examined. ❖ Used to find dielectric constant, the polarity and other properties.	❖ Not suitable for high molecular weight component. ❖ Very less resolving power. ❖ Cost of investigation increases with more accuracy .

	❖ Atomic vibrations involving a change in dipole moment and a change in polarizability, respectively.	❖ Mainly used in research. ❖ To detect explosives ❖ The technique is used is to study changes in chemical bonding ❖ To find crystallographic orientation of sample		
Electron Spin Resonance spectroscopy	❖ In this method, electron spins that are excited instead of the spins of atomic nuclei ❖ Detection of magnetic moment associated with unpaired electrons.	❖ Detection of changes in the environment of free radicals introduced into biological assemblies, e.g. membranes. ❖ EPR sspectroscopy is particularly useful for studying metal complexes or organic radicals	❖ It is highly specific. ❖ It is very sensitive. ❖ Less period of testing. ❖ Used for low concentration upto 1μM	❖ Not suitable for all materials ❖ For paramagnetic materials need low temperature

	of protons in an atomic nucleus.	control systems via analogue or digital outputs from the spectrometer.		
Spectrofluorimetry	❖ It is a type of electromagnetic spectroscopy which analyzes fluorescence from a sample. ❖ It involves using a beam of light, usually ultraviolet light, that excites the electrons in molecules of certain compounds and causes them to emit light of a lower energy ❖ Absorbed radiation emitted at longer wavelengths.	❖ Fluorescence spectroscopy is used in, among others, biochemical, medical, and chemical research fields for analyzing organic compounds.	❖ More sensitive at even low concentration. ❖ More precision is obtained. ❖ Applied even for non-fluorescent material.	❖ Not useful for all type of component. ❖ Contamination may happen in fluorescence.

4.9. ELECTRICAL AND MAGNETIC TECHNIQUES

4.9.1. ELECTRICAL TECHNIQUES

❖ Electrical properties are a key physical property of conducting materials. It is often necessary to accurately measure the resistivity of materials.

COMMON METHODS

❖ Dielectric strength
❖ Electrochemical Impedance Spectroscopy (EIS)
❖ Arc resistance
❖ EMF shielding test
❖ Two-probe method and four-probe method

4.9.1.1. ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

❖ Electrochemical Impedance Spectroscopy (EIS) is a highly sensitive characterization technique used to establish the electrical response of chemical systems in a nondestructive manner. It is an electrochemical technique to measure the impedance of a system in dependence of the AC potentials frequency.

1. PRINCIPLE

❖ An electrochemical cell is used to house the chemical reaction and is electrically connected to the electrochemical spectrometer to obtain the electrical response of an electrolytic solution. EIS systems are operated using computer programs specifically designed for EIS testing. Therefore, prior to conducting an EIS experiment it is essential that all components of the system be attained.

2. COMPONENTS

❖ Three electrodes (working electrode, counter electrode, reference electrode)
❖ Electrolytic solution
❖ Insulating material
❖ Display unit

3. CONSTRUCTION AND WORKING

- EIS studies utilize a three electrode mode which is comprised of a working electrode (the sample material), a counter electrode (commonly graphite or platinum), and a reference electrode.
- While electrode geometries may vary the general experimental setup remains similar to the procedure outlined below.

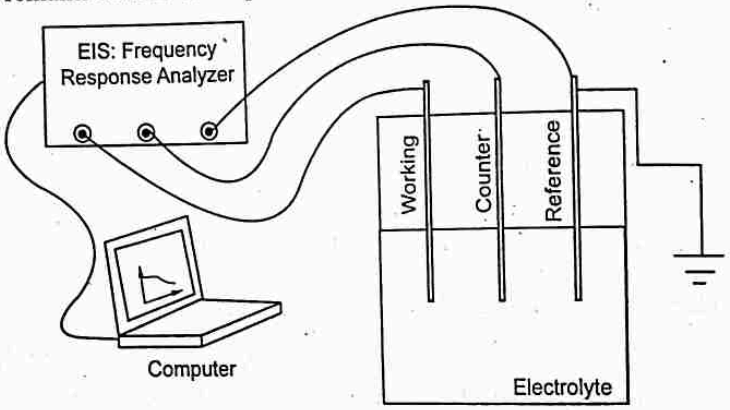


Fig. 4.11. Working of Electrochemical Impedance Spectroscopy (EIS)

- The three electrodes are mounted on an electrode stage and secured. The electrolytic solution is prepared and transferred to the sample container.
- A metallic sample container would provide additional pathways for electrons during experimentation leading to a reduction in the EIS current response as electrons move into the metal rather than the reference electrodes.
- Therefore, the sample container should be composed of an insulating material, such as glass or plastic, which will not interfere with the transfer of electrons during testing. The electrode mount is then placed on the sample container such that a portion of each electrode is submerged in the electrolytic solution.
- Four leads are used to attach the three electrodes to the EIS frequency response analyzer.
- A working lead and a counter lead is used to carry current, whereas the working sense lead and reference leads are used to sense voltage.

- The Electrochemical Impedance Spectroscopy (EIS) working sense lead connects the exposed end of the electrode to the EIS. The reference lead is attached to the reference electrode and the counter lead is connected to the counter electrode.
- The fourth lead is recommended to ground the system during testing. Once all leads are connected and by stimulus the data is collected from computer generated data.
- The impedance produced during electrochemical experimentation can be evaluated through use of one or more equivalent circuits.

4. ADVANTAGES

- Useful on high resistance materials such as paints and coatings.
- Time dependent data is available
- Non- destructive.
- Quantitative data available.
- Use service environments.

5. DISADVANTAGES

- Expensive.
- Complex data analysis for quantification.

6. APPLICATION

- It provides information about the corrosion kinetics and coatings evaluation.
- It is an accurate and reproducible technique suitable for highly resistive environments.
- It provides data about the electrochemical control mechanism, indicating if corrosion occurs by activation, concentration or diffusion.
- It characterizes the state of the rebar and the morphology of the corrosion.
- It allows for monitoring of the evolution of the passive or active state over time.

4.9.2. MAGNETIC TECHNIQUES

- Magnetic methods are potential methods for evaluation of surface manifestations such as microstructural degradation, residual stresses,

surface roughness and defect detection in surface coatings of magnetic substrates.

COMMON METHODS

- **Magnetic Adhesive Force Method:** Magnetic adhesive force method uses the distance dependency of the magnetic attractive force between a ferromagnetic substrate and a permanent magnet touching the surface of coating, which must be made from a non-magnetic material. Used to find holding power of magnet.
- **Magnetically Inductive Method:** Another popular method is the magnetically inductive method which is based on measuring the magnetic flux that passes through a non-ferro magnetic coating into a ferromagnetic substrate.
- **Magnetic Barkhausen Emission Method:** Magnetic flux perturbations and acoustic emissions are generated when an induced magnetic field is swept in a hysteresis loop in ferromagnetic materials. This is referred to as Magnetic Barkhausen Emissions (MBE). Surface characteristics such as hardness, residual stress and fatigue damage have been shown to influence Barkhausen activity and MBE technique is routinely used for evaluation of these characteristics both on production-line and in operating components.

4.9.3. ELECTROMAGNETIC TECHNIQUE

- Electromagnetic methods have very high potential for material characterization and well known nondestructive testing.
- Electromagnetic techniques are able to indicate nondestructively and quickly changes of residual stresses, texture, microstructure states, and mechanical properties, and are, therefore, very useful tools for materials characterization and damage assessment of in-service engineering components.

1. PRINCIPLE

- Magnetic hysteresis occurs when an external magnetic field is applied to a ferromagnetic such as iron and the atomic dipoles align themselves with it.

Even when the field is removed, part of the alignment will be retained: the material has become magnetized.

ELECTROMAGNETIC METHODS

- Magnetic Barkhausen noise
- Incremental permeability
  - Non-resonant Methods (transmission/reflection method)
  - Resonant Method
- Upper harmonics
- Incremental permeability

1. ADVANTAGES

- Nondestructive technique
- Used for Nano material characterization
- Immediate result.
- Accuracy in measurement of dielectric losses

2. DISADVANTAGES

- Limitation of lateral resolution.
- Need very thin samples
- Characterization limited to dielectric permittivity.
- Multiple steps
- Need technical knowledge
- Presence of air gaps may reduce the accuracy.

3. APPLICATIONS

- Used for purpose of finding micro structure, texture, hardness depth, phase content, residual stress, aging and grain size.

TWO MARK QUESTIONS WITH ANSWERS

1. ***Difference between microscopic and macroscopic observation.***

Microscopic Observation	Macroscopic Observation
Microscopic system is the one with objects or phenomena not visible with the naked eye and magnification instrument is necessary	**Macroscopic** system is the one with objects or phenomena visible with the naked eye and sometimes with magnifying instruments.
Scale of 1 to 100 nanometers and 1 to 1000 micro- meters	Scale of Millimeter to the kilometer scale
Needs very high magnification power	Needs very low magnification of 10x
The structural arrangement of atoms and bonds etc are observed	The appearance and physical arrangement is viewed by naked eye
This simple process can yield a large amount of information about the material such as ❖ The colour of the material ❖ Its lustre (does it display a metallic lustre) ❖ Its shape (whether it displays a regular, crystalline form) ❖ Its composition (is it made up of different phases) ❖ Its structural features (does it contain porosity) etc.	It is necessary because many of the properties of materials are dependent on extremely fine features and defects that are only possible to observe using one of the following techniques in this field.

2. ***Define magnification***

❖ Magnification on a microscope refers to the amount or degree of visual enlargement of an observed object or enlargement of image.

❖ Magnification is measured by multiples, such as 2x, 4x and 10x, indicating that the object is enlarged to twice as big, four times as big or 10 times as big, respectively.

Magnification = Image ÷ Object

3. ***Define resolution.***

❖ Resolution is defined as the ability to distinguish two very small and closely-spaced objects as separate entities.

❖ Resolution is determined by certain physical parameters that include the wavelength of light, and the light-gathering power of the objective and condenser lenses.

4. ***What are the types lenses used in microscope?***

❖ Objective lens

❖ Ocular lens (eyepiece)

❖ Condenser lens

5. ***Difference between optical and electron microscope.***

Optical Microscope	Electron Microscope
It uses the source of light.	The light source is replaced by a beam of very fast moving electrons
The minor work in specimen preparation	The specimen usually has to be specially prepared and held inside a vacuum air has been pumped out (because electrons do not travel very far in air).
Lens ,light source and reflective mirror is used	The lenses are replaced by a series of coil- shaped electromagnets through which the electron beam travels.
Low resolution and magnification (500x to 1000x)	High resolution and magnification (10000x app.)
Operation type is mechanical	Operation type is electrical
Relatively easy to carry and inexpensive	Relatively large and expensive

6. ***Give some common method of microscopic observation and macroscopic observation.***

Microscopic observation

- Optical Microscope
- Scanning Electron Microscope (SEM)
- Transmission Electron Microscope (TEM)
- Field Ion Microscope
- Scanning Tunneling Microscope

Macroscopic observation

- Mechanical testing, including tensile, compressive, torsional, creep, fatigue, toughness and hardness testing
- Differential thermal analysis
- Dielectric thermal analysis

7. ***Write major contrast between SEM and TEM***

Category	SEM	TEM
Source electrons	Scattered electrons	Transmitted electrons
Process of working	Scattering absorption	Diffraction
Energy	1-30kV	60-300kV
Environment	Air/vacuum	Vacuum
Specimen thickness	Any thickness	Typically less than 150nm
Output	3D image formation	2D projection image of inner structure
Magnification	2 million level magnification	50 million level magnification.
Image formation	Electron are captured and countered by detector image on PC	Direct image on fluorescent screen or PC screen with LCD
Amount of sample	Huge amount of sample	Minimum sample amount

8. ***Write principle of SEM and TEM***

- A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons.
- In **TEM,** an image is formed from the interaction of the electrons with the sample as the beam is transmitted through the specimen. The image is then magnified and focused onto an imaging device, such as a fluorescent screen, a layer of photographic film, or a sensor such as a scintillator attached to a charge-coupled device.

9. ***Why specimen preparation is important in microscopic technique.***

- Specimen preparation is important in any microscopic technique with proper preparation methods facilitating examination and interpretation of microstructural features.
- Improper preparation methods may obscure features, and even create artifacts that may be misinterpreted.

10. ***State the uses of scattered electrons in TEM .***

- **X-rays**-element and mineral information.
- **Secondary Electrons**-Secondary electron image for surface morphology
- **Backscattered Electrons**-This feature can be used to observe the topography of the surface.

11. ***State Difference between Diffraction and Inference.***

Inference	Diffraction
Interference is due to the super position of two different waves from coherent source	Diffraction is super position of secondary wavelets
Fringes width is constant	Fringes width are vary
Have same intensity	Have varying intensity

12. ***State Diffraction Principle.***

- **Bragg's law** is which determines the angles of coherent and incoherent scattering from a crystal lattice. When X-rays are incident on a particular atom, they make an electronic cloud move just like an electromagnetic wave.

13. ***Write the methods of Diffraction.***

1. Electron diffraction
2. Neutron diffraction
3. X-ray diffraction

14. ***Define spectroscopy.***

- Spectroscopy deals with the production, measurement, and interpretation of spectra arising from the interaction of electromagnetic radiation with matter.
- Spectroscopic methods are very informative and widely used for both quantitative and **qualitative analyses.**

15. ***What are the methods of Spectroscopy?***

- Ultraviolet-visible spectroscopy (UV-vis)
- Electron Spin Resonance spectroscopy
- Atomic spectroscopy
- infrared spectroscopy and Raman spectroscopy
- Mass spectrometry
- Nuclear spectroscopy(nuclear magnetic resonance)

16. ***Difference between Raman and IR spectroscopy.***

Raman spectroscopy	Infrared spectroscopy
It is due to the scattering of light by vibrating molecules	It is the result of absorption of light by vibrating molecules.
The vibration is active if it causes to change in polarizability	The vibration is active if it causes to change in dipole moment
The molecule need not possess a permanent dipole moment	The vibration concerned change in dipole moment due to vibration
Water can be used as solvent	Water cannot be used as intense absorption of IR
Sample preparation is not elaborate. Any state of sample is used	Sample preparation is elaborate. Gaseous sample can be rarely used. It diffused in

Raman spectroscopy	Infrared spectroscopy
Gives an indication of covalent character of molecule	Gives an ionic character in the molecule
Cost of instrument is high	Comparatively inexpensive
Weak in intensity	Strong in intensity
Optical system: Glass, quartz	Optical system: NaCl, KBr
Record by using a beam of monochromatic radiation	Record by using a beam of radiation having a large number of frequencies

17. ***Write about types in IR spectroscopy.***

There are four types of instruments for infrared absorption measurements available:

- Dispersive grating spectrophotometers for qualitative measurements .
- Non dispersive photometers for quantitative determination of organic species in the atmosphere .
- Reflectance photometers for analysis of solids .
- Fourier transform infrared (FT-IR) instruments for both qualitative and quantitative measurements.

18. ***Write about sample preparation in IR spectroscopy.***

- Sampling techniques for IR spectroscopy
- Gas sample - In sample tube of 10 cm length fitted with IR transparent holder
- Liquids - The think film formed between NaCl plates
- Solid - Pellet or as a Nujol mull (Nujol is a viscous mineral oil (hydrocarbon)) in which the solid is finely suspended

19. ***Write objectives of material characterization.***

- To measure accurately the physical properties of materials
- To measure accurately the chemical properties of materials
- To determine accurately the structure of a material at atomic and microscopic level structures

20. *Define scale.*

- The scale of the structures observed in materials characterization ranges from angstroms, such as in the imaging of individual atoms and chemical bonds, up to centimeters, such as in the imaging of coarse grain structures in metals.

REVIEW QUESTIONS

1. Explain various methods of microscopic technique with neat sketch.
   Ans: Section No. 4.3 **Page No:** 4.4
2. Explain SEM with principle of working.
   Ans: Section No. 4.6.1 **Page No:** 4.14
3. Write about advantages and limitation of TEM.
   Ans: Section No. 4.6.2 **Page No:** 4.25
4. What is optical microscope and how it is working?
   Ans: Section No. 4.5 **Page No:** 4.9
5. Write short note on various method of sample preparation in SEM and TEM.
   Ans: Section No. 4.6 **Page No:** 4.17, 4.21
6. Write comparison between X ray diffraction and electron diffraction.
   Ans: Section No. 4.7 **Page No:** 4.30
7. Explain diffraction techniques with principle of working.
   Ans: Section No. 4.7 **Page No:** 4.28
8. How spectroscopy is working with different principles of electromagnetic radiation?
   Ans: Section No. 4.8 **Page No:** 4.39
9. Write short note on
   (*a*) IR spectroscopy
   (*b*) UV spectroscopy
   (*c*) Mass spectroscopy
   Ans: Section No. 4.8 **Page No:** 4.45
10. Explain in detail about electrical and magnetic techniques.
    Ans: Section No. 4.9 **Page No:** 4.49

❑❑

UNIT V

OTHER TESTING

SYLLABUS

Thermal Testing: Differential scanning calorimetry, Differential thermal analysis. Thermo- mechanical and dynamic mechanical analysis: Principles, Advantages, Applications. Chemical Testing: X-Ray Fluorescence, Elemental Analysis by Inductively Coupled Plasma-Optical Emission Spectroscopy and Plasma-Mass Spectrometry.

5.1. OVERVIEW

- **Materials testing**, measurement of the characteristics and behaviour of such substances as metals, ceramics, or plastics etc. under various conditions.
- Investigators may construct mathematical models that utilize known material characteristics and behaviour to predict capabilities of the structure.

Materials testing breaks down into major categories

- Mechanical testing & Non destructive testing
  - Testing for physical & chemical properties
  - Testing for thermal properties
  - Testing for electrical properties
  - Testing for resistance to corrosion, Radiation and Biological deterioration

5.2. THERMAL ANALYSIS

- Thermal analysis is a form of analytical technique most commonly used in the branch of materials science where changes in the properties of materials are examined with respect to temperature.

- It is a group of techniques in which changes of physical or chemical properties of the sample are monitored against time or temperature, while the temperature of the sample is programmed.
- The temperature program may involve heating or cooling at a fixed rate, holding the temperature constant (isothermal), or any sequence of these.
- The sample is subjected to a predefined heating or cooling program.
- The sample is usually in the solid state and the changes that occur on heating include melting, phase transition, sublimation, and decomposition.

5.2.1. THERMAL PROPERTIES

- Thermal properties of material decide how it reacts when it is subjected to heat fluctuation (excessive heat or very low heat, for example). The major thermal properties are described in table 5.1.

Table 5.1. Thermal properties

S. No	Properties	Description
1.	Thermal conductivity	It is determining temperatures as a function of time along the length of a bar or across the surface
2.	Specific heat	It is defined as heat absorbed per unit mass per degree change in temperature
3.	Thermal expansion	Expansion due to heat is usually measured in linear fashion as the change in a unit length of a material caused by a one-degree change in temperature.
4.	Thermal stress	The stress experienced by a body due to either thermal expansion or contraction is called thermal stress.
5.	Thermo-Elastic Effect	When a solid is subjected to a load, work is done on it and it changes in volume. This will appear in the form of rise of temperature of solid when it is in stretched. Similarly when the solid is rapidly relaxed, it will cool. This warming or cooling phenomenon is called thermo elastic effect.

S. No	Properties	Description
6.	Thermal Shock	The ability of material to withstand thermal stresses due to sudden and severe changes in the temperature at the surface of a solid body.
7.	Melting point or heat resistance	Melting point or softening point is a significant temperature level as it represents transition point between solid and liquid phases.
8.	Emissivity of Materials	The emissivity (ε) of the surface of a material is its effectiveness in emitting energy as thermal radiation and varies between 0.0 and 1.0.
9.	Latent Heat of Fusion of Materials	Latent heat is the amount of heat added to or removed from a substance to produce a change in phase.
10.	Latent Heat of Vaporization of Materials	Certain amount of energy is involved in this change of phase, When a material changes phase from solid to liquid or from liquid to gas.

5.2.2. THERMAL TESTING

- Thermal Testing involves testing a product at the extremes of its intended use thermal environment for heating rate, temperature and airflow or gaseous atmosphere or vacuum with measuring case temperatures on individual components to determine the effect on product performance and long-term reliability.
- It measures based on dynamic relationships between temperature, Mass, Volume and Heat of reaction.

Major methods of Thermal testing,

- Differential thermal analysis
- Dilatometer
- Differential scanning calorimetry
- Dynamic mechanical analysis
- Thermogravimetric analysis

- Thermo mechanical analysis
- Thermo optical analysis

Other common methods of thermal methods

- Dielectric thermal analysis
- Evolved gas analysis
- Laser flash analysis
- Derivatography

Table 5.2. Parameters of thermal testing

S.No	Method	Parameter testing
1.	Thermogravimetric Analysis	Mass changes
2.	Differential Thermal Analysis	Temperature Difference
.3.	Differential Scanning Calorimetry	Heat Difference
4.	Evolved Gas Analysis	Gas Decomposition
5.	Thermo Mechanical Analysis	Deformation And Dimension
6.	Dilatometer	Volume
7.	Dielectric thermal analysis	Electrical properties
8.	Thermo optical analysis	Optical properties

5.2.3. THERMOGRAVIMETRIC ANALYSIS (TGA)

- The Thermogravimetric analysis (TGA) is a type of thermo analytical testing performed on materials to determine changes in weight in relation to changes in temperature.
- The TGA relies on a high degree of precision in three measurements: weight, temperature and temperature change.
- The TGA is commonly employed in research and testing to determine characteristics of materials, to determine degradation temperatures, absorbed moisture content of materials, the level of inorganic and organic components in materials, decomposition points of explosives and solvent residues.

5.2.4. DIFFERENTIAL SCANNING CALORIMETRY

- DSC measures the energy absorbed or released from a sample as a function of time or a temperature profile.
- DSC is useful to make the measurements for melting points, heats of reaction, glass transition, and heat capacity

1. PRINCIPLE

- Differential scanning calorimetry (DSC) is based on the principle; sample and reference are maintained at the same temperature, even during a thermal event (in the sample). The energy required maintaining zero temperature different between the sample and the reference is measured.
- By calibrating the standard material(reference material), the unknown sample quantitative measurement is achievable.

2. TYPES

There are four different types of DSC instrument

- Heat flux DSC
- Power compensated DSC
- Modulated DSC
- Hyper DSC
- Pressure DSC

The most common methods are Heat flux DSC and Power compensated DSC

3. POWER COMPENSATION DSC

- A technique in which difference of thermal energy that is applied to the sample and the reference material separately per unit of time is measured as a function of the temperature.

(a) Components

- Separate sensors and heaters are used for the sample and reference
- **Sample holder:** Al or Platinum pans
- **Sensors:** Platinum resistance thermocouples
- **Furnace:** Separate blocks for sample and reference cells

- **Temperature controller:** Differential thermal power is supplied to the heaters to maintain the temperature of the sample and reference at the program value

(b) Working

- The power needed to maintain the sample temperature equal to the reference temperature is measured.
- In power compensation DSC two independent heating units are employed.
- These heating units are quite small, allowing for rapid rates of heating, cooling and equilibration. The heating units are embedded in a large temperature- controlled heat sink.
- The sample and reference holders have platinum resistance thermometers to continuously monitor the temperature of the materials.
- The instrument records the power difference needed to maintain the sample and reference at the same temperature as a function of the programmed temperatures.
- Power compensated DSC has lower sensitivity than heat flux DSC, but its response time is more rapid. It is also capable of higher resolution then heat flux DSC .
- This makes power compensated DSC well suited for kinetics studies in which fast equilibrations to new temperature settings are needed.

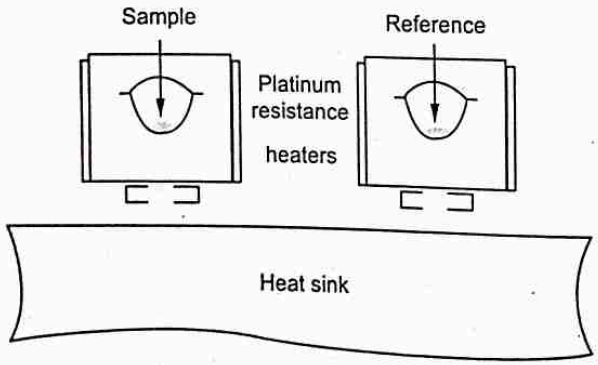


Fig. 5.1. Typical arrangement of Power compensated DSC

4. HEAT FLUX DSC

- In heat flux DSC, the difference in heat flow into the sample and reference is measured while the sample temperature is changed at the constant rate
- Sample and reference are connected by a low resistance heat flow path (a metal disc).The assembly is enclosed in a single furnace.

(a) Components

One blocks for both sample and reference cells

- **Sample holder:** Sample and reference are connected by a low- resistance heat flow path.Al or Platinum pans placed on constantan disc.
- **Sensors:** Chromel alumel thermocouples Furnace are used.

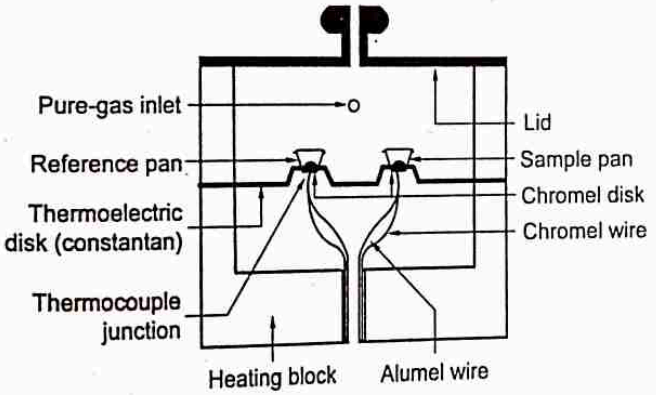


Fig. 5.2. Typical arrangement of heat flux DSC

(b) Working

- The main assembly of the DSC cell is enclosed in a cylindrical, silver heating black, which dissipates heat to the specimens via a constantan disc which is attached to the silver block.
- The disk has two raised platforms on which the sample and reference pans are placed.
- A chromel disk and connecting wire are attached to the underside of each platform, and the resulting chromel-constantan thermocouples are used to determine the differential temperatures of interest.

- Alumel wires attached to the chrome discs provide the chromel-alumel junctions for independently measuring the sample and reference temperature.

5. DSC MEASURES

- Glass transitions
- Melting and boiling points
- Crystallization time and temperature
- Percent crystallinity
- Heats of fusion and reactions
- Specific heat capacity
- Oxidative/thermal stability
- Reaction kinetics
- Purity

6. DSC Curve

- DSC Curve is plot between heat flow and temperature. It shows various peaks of measurement

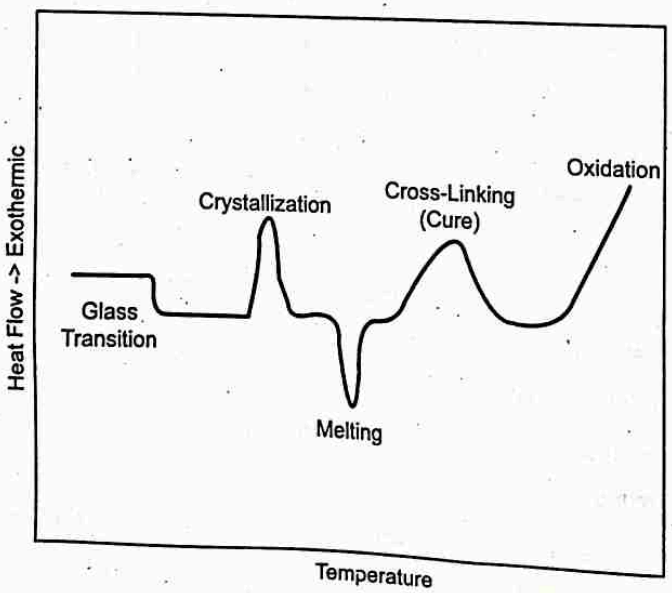


Fig. 5.3. DSC Curve

Factors Affecting DSC Curve

Table 5.3. Factors Affecting DSC Curve

Instrumental Factors	Sample Characteristic Factors
❖ Furnace heating rate ❖ Recording or chart speed ❖ Furnace atmosphere ❖ Geometry of sample holder/location of sensors ❖ Sensitivity of the recording system ❖ Composition of sample containers	❖ Amount of sample ❖ Nature of sample ❖ Sample packing ❖ Solubility of evolved gases in the sample ❖ Particle size ❖ Heat of reaction ❖ Thermal conductivity

7. APPLICATION OF DSC

- To observe fusion and crystallization events as well glass transition temperature
- To study oxidation, as well as other chemical reactions
- The transition from amorphous to crystalline is known.
- The ability to determine transition temperature and enthalpies.
- Rapid optimization of purification and manufacturing conditions

8. SOURCES OF ERRORS

- Calibration
- Contamination
- Sample preparation - how sample is loaded into a pan
- Residual solvents and moisture.
- Thermal lag
- Heating/Cooling rates
- Sample mass

9. ADVANTAGES OF DSC

- Instruments can be used at very high temperatures

- Instruments are highly sensitive
- Flexibility in sample volume/form
- Characteristic transition or reaction temperatures can be determined
- High resolution obtained
- High sensitivity
- Stability of the material.

10. LIMITATIONS OF DSC

- DSC generally unsuitable for two-phase mixtures
- Difficulties in test cell preparation in avoiding evaporation of volatile Solvents
- DSC is generally only used for thermal screening of isolated intermediates and products
- Does not detect gas generation
- Uncertainty of heats of fusion and transition temperatures

5.2.5. DIFFERENTIAL THERMAL ANALYSIS

- Differential thermal analysis (DTA) is a thermo-analytical technique which is used for thermal analysis where thermal changes can be studied. It is used to determine the oxidation process, decomposition, and loss of water or solvent.

1. PRINCIPLE

- In DTA, the sample material and an reference material are made to undergo identical thermal cycles, (i.e., same cooling or heating programme) while recording any temperature difference between sample and reference. This differential temperature is then plotted against time, or against temperature (DTA curve, or thermogram). Changes in the sample, either exothermic or endothermic, can be detected relative to the inert reference.

2. COMPONENTS

- **Furnace** - This is device for heating the sample(Nickel and chromium alloy furnace)

- **Sample holder** - This is used to contain the sample as well as the reference material (Platinum alloy holder)
- **DC amplifier** - Generally a low level DC amplifier is employed.
- **Differential Temperature Detector (Thermogram)** - The function of this detector is to measure differential temperature.
- **Furnace Temperature Programme** - The main function of this is to increase the temperature of the furnace at a steady rate.
- **Recorder** - This is to record the DTA curve(automatic electronic recorder)
- **Control Equipment**- Its function is to maintain a suitable atmosphere in the furnace & sample holder.

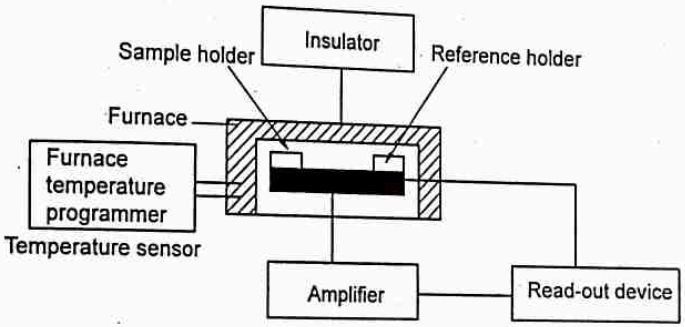


Fig. 5.4. Cross section of DTA

3. WORKING

- The sample under investigation is loaded into a container.
- This container is then placed onto the sample pan and it is marked as S (means sample). Same quantity of reference sample is placed in another container which is then placed onto the reference pan and it is marked as R (means reference).
- In order to heat the sample pan and the reference pan at an identical rate, the dimensions of these two pans should be nearly identical; moreover, the sample and the reference should have equal weights, thermally matched and should be arranged symmetrically with the furnace.
- The metal block which surrounds the pans acts as a heat sink whose temperature is increased slowly by using an internal heater.
- The sink then heats the sample and reference material simultaneously.

- Two pairs of thermocouples are used, one pair is in contact with the sample and the second pair is in contact with the reference.
- Thermocouple is attached with an amplifier which amplifies the result of differential thermocouple and sent this result to the read-out device which displays the results in the form of DTA curve or thermogram as a function of the sample temperature, reference temperature or time.
- No signal is generated if no temperature difference is observed even though the actual temperatures of both the sample and reference are increasing.
- When there is a physical change in the sample then heat is absorbed or released. For example, when a metal carbonate is decomposed then carbon dioxide is released. This is an endothermic reaction where the heat is absorbed and the temperature of the sample is decreased. Now the sample is at a lower temperature than that of the reference. This temperature difference between sample and reference produces a net signal, which is then recorded.

4. DTA Curve

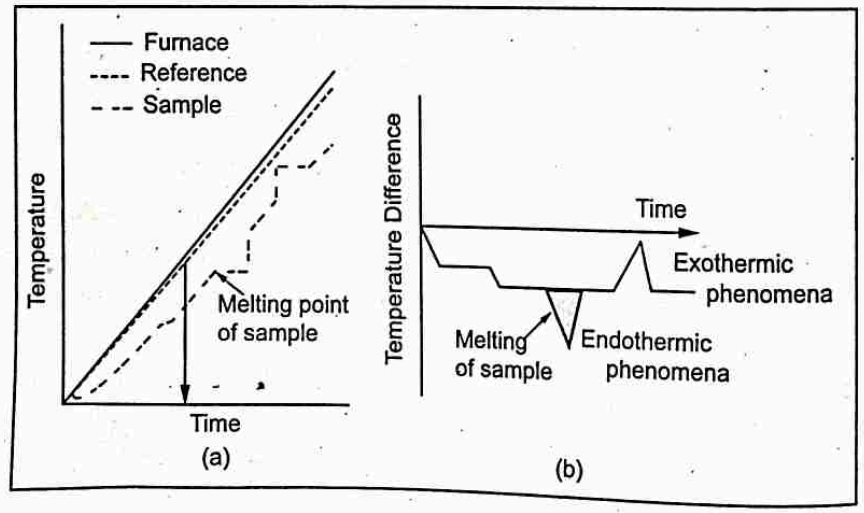


Fig. 5.5. (a) The DTA curve or thermo gram is a plot between differential temperature and Time. (b) DTA curve may be endothermic (downward plot) or exothermic (upward plot).

Factors affecting DTA curve

Table 5.4. Factors affecting DTA curve

Sample factors	Instrumental factors	Physical factors
❖ Amount of the sample. ❖ Packing density. ❖ Particle size of the sample material. ❖ Degree of crystallinity. ❖ Heat capacity. ❖ Thermal conductivity. ❖ Dilutes of the diluents. ❖ Swelling of the sample. ❖ Shrinkage of the sample.	❖ Size or shape of the holders. ❖ Material of the sample holder. ❖ Recording system sensitivity. ❖ Rate of heating of the sample. ❖ Atmosphere around the sample. ❖ Thermocouple location in the sample. ❖ Instrumental design.	❖ Adsorption. ❖ Change in the crystal structure. ❖ Crystallization. ❖ Desorption. ❖ Change in the crystal structure. ❖ Vaporization. ❖ Sublimation. ❖ Melting.

5. ADVANTAGES

- It can be operated at very high temperature ranges.
- Highly sensitive technique.
- Flexibility in crucible volume
- Both exothermic and endothermic reactions can be determined accurately.

6. DISADVANTAGES

- There is lot of uncertainty in transition reactions and heat of fusions upto 20-50%
- Destructive limited range of samples time consuming usually not qualitative.

7. APPLICATIONS

- Used to identify the minerals both qualitatively and quantitatively.
- Rapid identification of the compositions of mixed clays
- Polymers characteristics can be easily characterized.
- Degree of crystallinity can be measured.
- Degree of polymerization can be assessed.
- Many of the biological materials can be analyzed.
- DTA offers a wide spectrum of useful investigations related to reaction kinetics, polymerization, solvent retention, phase-transformations, solid-phase reactions and curing or drying properties of a product
- Melting point, boiling point, and temperatures of decomposition of organic compounds can be determined.
- Have wide applications for the quality control (QC) of many substances such as soil, cement, glass, etc.
- Also used to determine the thermal stability of many inorganic compound s and complexes.

5.2.6. THERMO- MECHANICAL ANALYSIS

- A technique in which a deformation of the sample under non-oscillating stress is monitored against time or temperature while the temperature of the sample, in a specified atmosphere, is programmed. Thermo mechanical analysis (TMA) easily and rapidly measures sample displacement (growth, shrinkage, movement, etc.) as a function of temperature, time and applied force.

1. PRINCIPLE

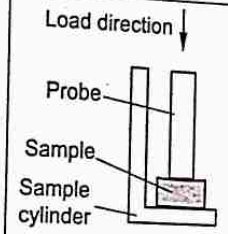
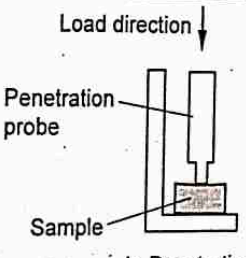
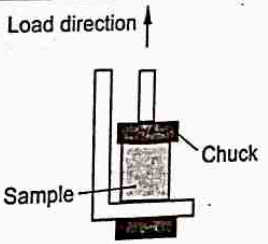
- Thermo mechanical analysis (TMA) is used to measure the dimensional changes of a material as a function of temperature by applying stress. The stress may be compression, tension, flexure or torsion.

2. COMPONENTS

- Transducer (Linear Variable Displacement Transducer (LVDT) , laser , optoelectronic etc.,

- Probe (made up of quartz glass)
- Thermocouple Furnace
- Force generator

3. PROBES ON DIFFERENT LOADING CONDITION

Loading Condition	Load Application	Purpose
(a) Expansion / Compression Probe	 a. Expansion/Compression	It is used for the measurement of the deformation by the thermal expansion and the transition of the sample under the compressed force is applied.
(b) Penetration Probe	 b. Penetration	It is used for the measurement of the softening temperature.
(c) Tension Probe	 c. Tension	It is used for the measurement of the thermal expansion and the thermal shrinkage of the sample such as the film and the fiber.

4. CONSTRUCTION AND WORKING

- The sample is inserted into the furnace and is touched by the probe which is connected with the Length Detector and the Force Generator. The construction of the pushrod and sample holder depends on the mode of the measurements.

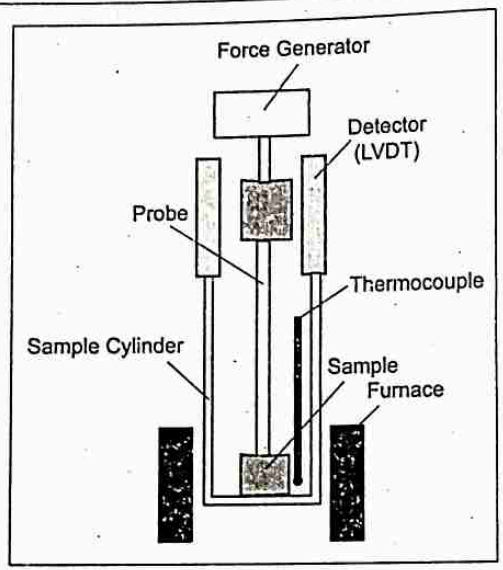


Fig. 5.6. Working of Thermo mechanical analyser

- The thermocouple for temperature measurement is located near the sample. The rate of 5° C/min is usually the maximum recommended value for good temperature equilibration across the specimen
- The sample temperature is changed in the furnace by applying the force onto the sample from the Force Generator via probe.
- The sample deformation such as Thermal Expansion and Softening with changing temperature is measured as the probe displacement by the Length Detector. Linear Variable Differential Transformer (LVDT) is used for Length Detection sensor.
- Every displacement of the pushrod is trans-formed into an analog signal by the LVDT, converted to digital form and then recorded in the computer system, and finally presented by the software as a dimensional change versus time or temperature.

5. APPLICATION

- Measurement of Dimensional Change
- Coefficient of Linear Thermal Expansion
- Determination of Material Anisotropy

- Softening Temperatures and Glass Transition
- Linear Thermal Expansion

6. ADVANTAGES

- Compactness and lightness
- Low operation voltage
- Measures large deformation
- Large actuation force
- Measures measure relaxation effects

7. LIMITATION

- Used only for solid samples.
- Creep occurring concurrently with normal dimensional changes.
- Usage of proper probe.
- Low operational speed

5.2.7. THERMO MECHANICAL DYNAMIC ANALYSIS

- **Thermo mechanical dynamic analysis**, otherwise known as Dynamic Mechanical Analysis (DMA), is a technique where a small deformation is applied to a sample in a cyclic manner. This allows the materials response to stress, temperature, frequency and other values to be studied. The term is also used to refer to the analyzer that performs the test.
- Dynamic mechanical analysis (DMA) is an important technique used to measure the mechanical and viscoelastic properties of materials such as thermoplastics, thermosets, elastomers, ceramics and metals.

1. PRINCIPLE

- A sinusoidal stress is applied and the strain in the material is measured, allowing one to determine the complex modulus. The temperature of the sample or the frequency of the stress are often varied, leading to variations in the complex modulus; this approach can be used to locate the temperature of the material, as well as to identify transitions corresponding to other molecular motions.

2. TYPES OF THERMO MECHANICAL DYNAMIC ANALYZER

- Forced resonance analyzers - Analyzers force the sample to oscillate at a certain frequency and are reliable for performing a temperature sweep.
- Free resonance analyzers- Free resonance analyzers measure the free oscillations of damping of the sample being tested by suspending and swinging the sample

3. MODE OF ANALYZER

- Stress (force) control - The structure of the sample is less likely to be destroyed and longer relaxation times/ longer creep studies can be done.
- Strain (displacement) control-The better short time response for materials of low viscosity and experiments of stress relaxation are done with relative ease

4. COMPONENTS

- **Transducer Sensor (Linear Variable Displacement Transducer (LVDT)** – It is which measures a change in voltage.
- **Drive shaft or probe** - It is a support and guidance system to act as a guide for the force from the motor to the sample.
- **Drive motor** - A linear motor for probe loading which provides load for the applied force.
- **Stepper motor** - It controls the specimen dimension and measurement.

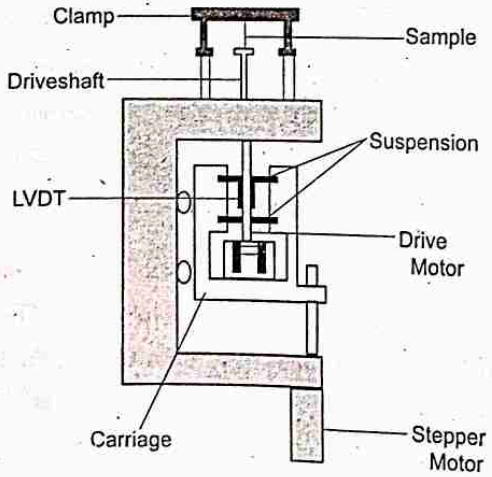
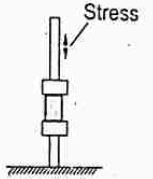
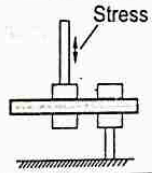
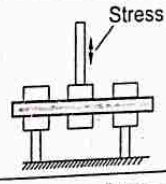
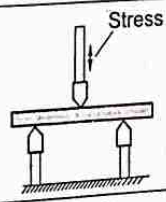
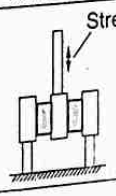


Fig. 5.7. Cross section of Thermo mechanical dynamic analyser

5. WORKING

- The sample is clamped in the measurement head of the DMA instrument. During measurement, sinusoidal force is applied to the sample via the probe or driving shaft.
- Deformation caused by the sinusoidal force is detected and the relation between the deformation and the applied force is measured.
- Properties such as elasticity and viscosity are calculated from the applied stress and strain plotted as a function of temperature or time.

Table 5.5. different loading condition

Modes	Stress application	Purpose
Shear mode		Used for evaluation of thin fibers or films or bundle of single fiber
3- point bending mode		Best for medium to high modulus materials.
Dual cantilever mode		Highly damped materials can be measured.
Single cantilever mode		Suited best for thermoplastics
Tension or compression mode		Used for low to medium moduius materials

6. DIFFERENT LOADING MODES

- The most suitable type should be selected depending on the sample shape, modulus and measurement purpose.

7. ADVANTAGES

- It is an essential analytical technique to determining the viscoelastic properties of polymers.
- Very soft and hard samples are measured.
- Allows accurate temperature measurement.
- It can provide major and minor transitions of materials
- It is also more sensitive.
- It is able to quickly scan and calculate the modulus for a range of temperatures.
- It is the only technique that can determine the basic structure of a polymer system
- This analytical method is able to accurately predict the performance of materials in use.

8. LIMITATIONS

- It leads to calculation inaccuracies.
- The large inaccuracies are introduced if dimensional measurements of samples are slightly inaccurate.
- The oscillating stress converts mechanical energy to heat and changes the temperature of the sample.
- The maintaining an exact temperature is important in temperature scans, this also introduces inaccuracies.
- The final source of measurement uncertainty comes from computer error.

9. DMA MEASURES

- Displacement and force
- Wide range of force 1mN to 40N
- Wide range of frequency 0.001 to 1000Hz.
- Wide stiffness range.

- Coefficient of Thermal Expansion (CTE)
- Glass Transition Temperature
- Compression Modulus of Polymeric Materials
- Viscoelastic properties such as:
  - Storage modulus (purely elastic component)
  - Loss modulus (purely viscous component)
  - Loss tangent

10. APPLICATION

- Measurement of the glass transition temperature of polymers
- Varying the composition of monomers
- Effectively evaluate the miscibility of polymers
- To characterize the glass transition temperature of a material.
- Mechanical properties in the relevant frequency range
- Modulus information
- Measurement of different relaxations
- Molecular interaction
- Nonlinear properties
- Damping behaviour

5.3. CHEMICAL ANALYSIS

- Chemical analysis is used to identify the contents, composition and quality of the materials used in product development, manufacturing and testing.

5.3.1. CHEMICAL PROPERTY

- A chemical property is a characteristic or behavior of a substance that may be observed when it undergoes a chemical change or reaction.

S.No	Property	Description
1.	**Toxicity**	Toxicity is a very important chemical property because it gives the harm of a substance can bring to other organisms.

S.No	Property	Description
2.	**Reactivity**	Reactivity is the tendency of a substance to undergo chemical reaction, either by itself or with other materials, and to release energy.
3.	**Types of chemical bonds formed**	Chemical bonds include covalent, polar covalent and ionic bonds.
4.	**Coordination number**	The coordination number of an atom in a molecule is the number of atoms bonded to the atom, the coordination number describes the number of neighbor atoms with respect to a central atom
5.	**Oxidation states**	The oxidation state is the charge of an atom if all bonds it formed were ionic bonds.
6.	**Flammability**	Flammable is a property of a material relating how easily the material ignites or sustains a combustion reaction.
7.	**Heat of combustion**	A combustion reaction involves oxygen and releases energy as heat.
8.	**Enthalpy of formation**	The heat of formation is the heat released or absorbed (enthalpy change) during the formation of a pure substance from its elements at constant pressure (in their standard states). Heat of formation is also called enthalpy of formation kilojoules per mole (kJ/mol).
9.	**Chemical stability under specific conditions**	Stability occurs when a system is in its lowest energy state, or chemical equilibrium with its environment

S.No	Property	Description
10.	**Acidity or basicity**	Acidity is the extent to which a substance will donate a proton/hydrogen ion. Basicity is the extent to which a substance will accept a proton/hydrogen ion.
11.	**Radioactivity**	Radioactive decay is a property of several naturally occurring elements as well as of artificially produced isotopes of the elements

5.3.2. CHEMICAL TESTING

- Chemical Testing provides a variety of quantitative and qualitative services for verification, identification and component analysis of ferrous and non-ferrous metals.

5.3.3. PURPOSE OF CHEMICAL TESTING

- Chemical Trace Analysis
- Elemental Trace Analysis
- Failure Analysis
- Contamination Analysis
- Materials Analysis and Testing
- Material Verification
- Material Identification
- Chemical Composition Analysis

5.3.4. CHEMICAL COMPOSITION TECHNIQUE AND TESTS

(a) Chromatography Technique

- Gas Chromatography
- Ion Chromatography
- Liquid Chromatography

(b) Mass Spectroscopy Technique

- Gas Chromatography Mass Spectroscopy
- Inductively Coupled Plasma

(c) Spectroscopy Technique

- Fourier-transform infrared spectroscopy (Solution & Pellet)
- X-Ray - EDS & XRF Analysis
- Inductively Coupled Plasma (ICP-AES)
- Atomic Absorption Graphite Furnace (GF-AAS)
- Spark Atomic Emissions (Spark-AES)

(d) Wet Chemistry Technique

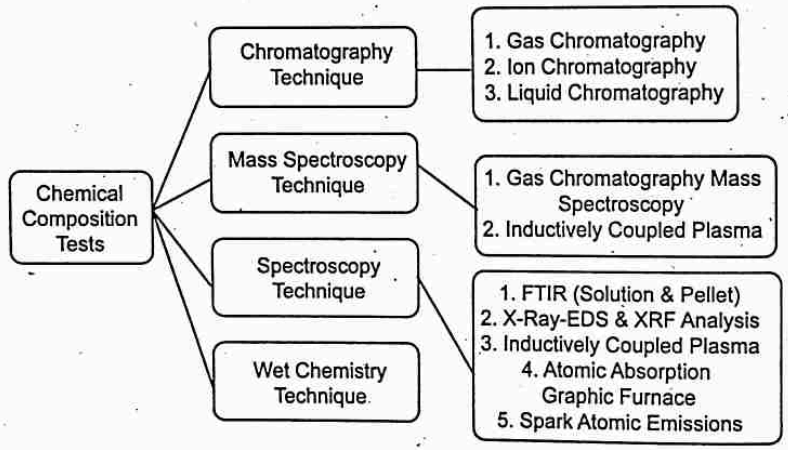


Fig. 5.8. Types of chemical composition test

5.3.5. X-RAY FLUORESCENCE

- XRF (X-ray fluorescence) is a non-destructive analytical technique used to determine the elemental composition of materials. XRF analyzers determine the chemistry of a sample by measuring the fluorescent X-ray emitted from a sample when it is excited by a primary X-ray source.
- The phenomenon is widely used for elemental analysis and chemical analysis, particularly in the investigation of metals, glass, ceramics and building materials, and for research in geochemistry.

1. PRINCIPLE

- **X-ray fluorescence (XRF)** is the emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by being bombarded with high-energy X-rays or gamma rays.

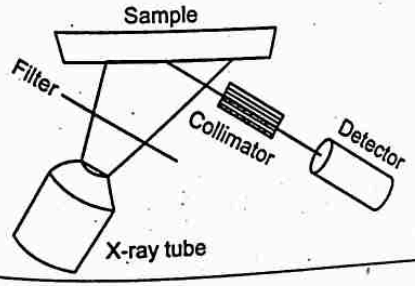
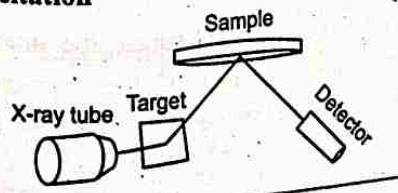
2. COMPONENTS OF A TYPICAL XRF SPECTROMETER

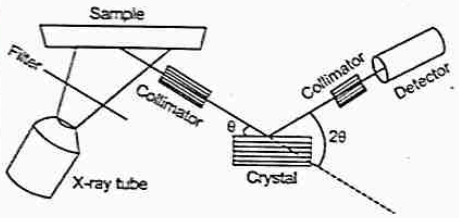
- Source of X-rays used to irradiate the sample. Wavelengths are typically in the range 0.01 to 10 nm, which is equivalent to energies of 125 keV to 0.125 keV.
- Detection equipped by Gas-filled detectors, semiconductor detector, scintillation detector, a photographic plate.

3. TYPES OF XRF SPECTROSCOPY.

The XRF spectroscopy differs primarily by detection and analyzing.

- Energy Dispersive XRF (Direct and polarized excitation)
- Wavelength Dispersive XRF

Methods	Description
Energy Dispersive X-ray fluorescence with direct excitation 	An energy dispersive detection system directly measures the different energies of the emitted X-rays from the sample. By counting and plotting the relative numbers of X-rays at each energy an XRF spectrum is generated
Energy Dispersive X-ray fluorescence with polarized excitation 	The detector must be perpendicular to the plane determined by the tube, target and sample. The most important effect is that by deflecting the X-ray radiation by 90°, the radiation is polarized and the spectral background in the spectrum is reduced.

Methods	Description
Wavelength Dispersive X-ray fluorescence 	The X-rays are directed to a crystal, which diffracts the X-rays in different directions according to their wavelengths (energies).On a sequential system a detector is placed at a fixed position, and the crystal is rotated so that different wavelengths are picked up by the detector.

4. WORKING OF X-RAY FLUORESCENCE

1. A solid or a liquid sample is irradiated with high energy X-rays from a controlled X-ray tube.
2. When an atom in the sample is struck with an X-ray of sufficient energy, an electron from one of the atom's inner orbital shells is removed.
3. The atom regains stability, filling the vacancy left in the inner orbital shell with an electron from one of the atom's higher energy orbital shells.
4. The electron drops to the lower energy state by releasing a fluorescent X-ray. The energy of this X-ray is equal to the specific difference in energy between two quantum states of the electron. The measurement of this energy is the basis of XRF analysis.
5. The intensity of each characteristic radiation is directly related to the amount of each element in the material.

5. APPLICATIONS

- It is a method of elemental (metal and Nonmetal) analysis with atomic number greater than 12.
- Quantitative analysis can be carried out by measuring the intensity of fluorescence at the wavelength characteristics of the element being determined, especially applicable to most of the element in the periodic table.

- Research in igneous, sedimentary, and metamorphic petrology, Soil surveys, Mining (e.g., measuring the grade of ore),Cement production, Ceramic and glass manufacturing
- Metallurgy (e.g., quality control)
- Environmental studies (e.g., analyses of particulate matter on air filters)
- Petroleum industry (e.g., sulfur content of crude oils and petroleum products)
- Field analysis in geological and environmental studies (using portable, hand-held XRF spectrometers)
- Bulk chemical analyses of major elements and trace elements

6. ADVANTAGES

- Simple spectra analysis
- XRF is a versatile and rapid technique
- Easily analysis of the element among the same family elements
- It is non-destructive method of chemical analysis
- Important as in case of samples in limited amounts, or valuable or irreplaceable
- It is precise and with skilled operations it is accurate
- Applicable to a wide variety of samples from powders to liquids
- It is convenient and economical to use
- The instruments have few moving parts, tend to be low-maintenance, and on a regular basis consume only liquid nitrogen and electricity
- Spectral positions are almost independent of the chemical state of the analyses
- Applicable over a wide range of concentrations

7. ADVANTAGES

- It fairly high limits of detection when compared to other methods
- Possibility of matrix effects, although these can usually be accounted for using software-based correction procedures

- It is limited in their ability to precisely and accurately measure the abundances of elements with Atomic number <11 in most natural earth materials
- XRF analyses cannot distinguish variations among isotopes of an element
- XRF analyses cannot distinguish ions of the same element in different valence states
- Instrumentation is fairly expensive

5.4. ELEMENTAL ANALYSIS BY INDUCTIVELY COUPLED PLASMA

5.4.1. ELEMENTAL ANALYSIS

- **Elemental analysis** is a process where a sample of some material (e.g., soil, waste or drinking water, bodily fluids, minerals, chemical compounds) is analyzed for its elemental and sometimes isotopic composition. Elemental analysis can be qualitative (determining what elements are present), and it can be quantitative (determining how much of each are present). Elemental analysis falls within the ambit of analytical chemistry, the set of instruments involved in deciphering the chemical nature of our world.

5.4.2. METHODS OF ELEMENTAL ANALYSIS

- CHNX analysis
- Quantitative analysis
- Qualitative analysis

1. **CHNX analysis** - The determination of the mass fractions of carbon, hydrogen, nitrogen and heteroatoms (X) (halogens, sulfur) of a sample.
   - The varies CHNX analysis are
     - NMR (Nuclear Magnetic Resonance)
     - Mass spectrometry chromatographic
     - Combustion analysis
2. **Quantitative analysis** - Quantitative analysis is the determination of the mass of each element or compound present. The quantitative analysis are
   - Gravimetery analysis

   - Optical atomic spectroscopy(Flame atomic absorption, Graphite furnace atomic absorption, and Inductively coupled plasma atomic emission spectroscopy)
   - Neutron activation analysis
3. **Qualitative analysis** - To qualitatively determine which elements exist in a sample
   - Mass spectrometric (atomic spectroscopy)
   - Inductively coupled plasma mass spectrometry
   - X-ray fluorescence
   - Particle-induced X-ray emission
   - X-ray photoelectron spectroscopy
   - Auger electron spectroscopy
   - Sodium fusion test

5.4.3. EXCITING SOURCE OF MASS AND EMISSION SPECTROMETRY

- The excitation source must desolvate, atomize, and excite the analyte atoms. A variety of excitation sources are flame, arc/spark and plasma.

5.4.3.1. PLASMA

- Plasma is an electrical conducting gaseous mixture containing significant amounts of cations and electrons (net charge approaches zero).

1. ADVANTAGES OF PLASMA

- Increased atomization/excitation
- Wider range of elements
- Simultaneous multi element analysis
- Wide dynamic range

2. TYPES OF PLASMA

- **Direct-current plasma (DCP)** - In a DCP, a dc current passing between two electrodes heats the plasma gas, again typically argon, and produces a discharge. The most common version is the three- electrode system.

- **Microwave-induced plasma (MIP)** - A MIP is an electrode less discharge generated in a glass or quartz capillary discharge tube, often in a resonant cavity.
- **Capacitively Coupled Microwave Plasmas (CMP)** - A CMP is formed using a magnetron to produce microwave energy at 2.45 GHz.
- **Inductively-coupled plasma (ICP)**

5.4.3.2. Inductively-Coupled Plasma (ICP)

- An **inductively coupled plasma (ICP)** or **transformer coupled plasma (TCP)** is a type of plasma source in which the energy is supplied by electric currents which are produced by electromagnetic induction, that is, by time-varying magnetic fields.
- The most commonly used ion source for plasma spectrometry, the ICP, is produced by flowing an inert gas, typically argon, through a water-cooled induction coil which has a high-frequency field (typically 27 MHz) running through it .
- An inductively coupled plasma (ICP) is a very high temperature (7000-8000K) excitation source. ICP sources are used to excite atoms for atomic-emission spectroscopy and to ionize atoms for mass spectrometry.

1. PRODUCTION OF PLASMA

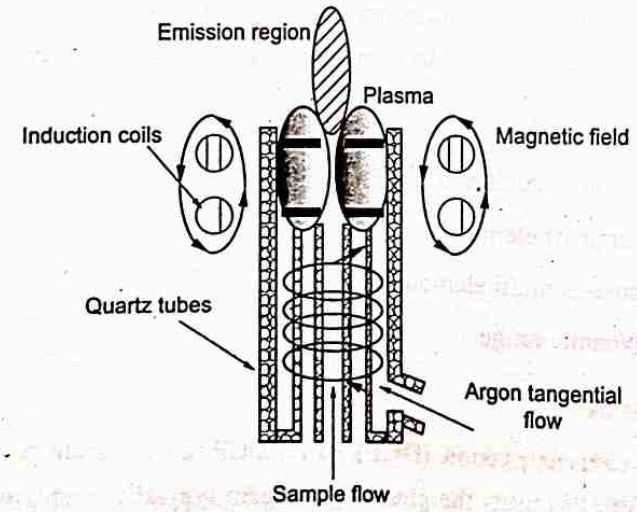


Fig. 5.9. Production process of plasma

- Inductively coupled discharge also uses RF power supply like capacitively coupled discharge.
- A radiofrequency (RF) generator (typically 1-5 kW @ 27 MHz) produces an oscillating current in an induction coil that wraps around the tubes.
- For a commonly used cylindrical plasma chamber shown below, antenna is usually wrapped around the electrically insulating chamber wall.
- RF generator drives high alternating current through coil antenna, which creates an alternating magnetic field within the plasma chamber.
- Oscillating magnetic field will generate an oscillating electric field in the plasma chamber.
- Eventually, the electric field will accelerate the electrons and generate plasma.
- The magnetic field in turn sets up an oscillating current in the ions and electrons of the support gas (argon). As the ions and electrons collide with other atoms in the support gas.
- Since the excitation force is delivered through magnetic field, inductively coupled discharge is also called "H-discharge". For some applications, it is described as "electrode less discharge" because there is no cathode or anode required for inductively coupled discharge.

2. CHARACTERISTICS OF OPTICALLY COUPLED PLASMA

- High temperature (7000 – 8000 K)
- High electron density (1014–1016cm)
- Appreciable degree of ionization for many elements
- Simultaneous multielement capability (over 70 elements including P and S)
- Low background emission and relatively low chemical interference
- High stability leading to excellent accuracy and precision
- Excellent detection limits for most elements (0.1 –100 ng mL-1)
- Wide linear dynamic range (LDR) (four to six orders of magnitude)
- Applicable to the refractory elements cost-effective analyses

5.4.4. OPTICAL EMISSION SPECTROSCOPY

- Optical Emission Spectroscopy, or OES analysis, is a rapid method for determining the elemental composition of a variety of metals and alloys.

Based on excitation source Optical Emission Spectroscopy is classified as,

- Inductively Coupled Optical Emission Spectroscopy
- Glow Discharge Optical Emission Spectrometry (GD-OES) or Glow Discharge MS (GD-MS)
- Arc spark Optical Emission Spectroscopy
- Flame emission spectroscopy

5.4.5. INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY

- The Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) analysis method uses high-frequency inductively coupled plasma as the light source, and is ideal for the element analysis of sample solutions.

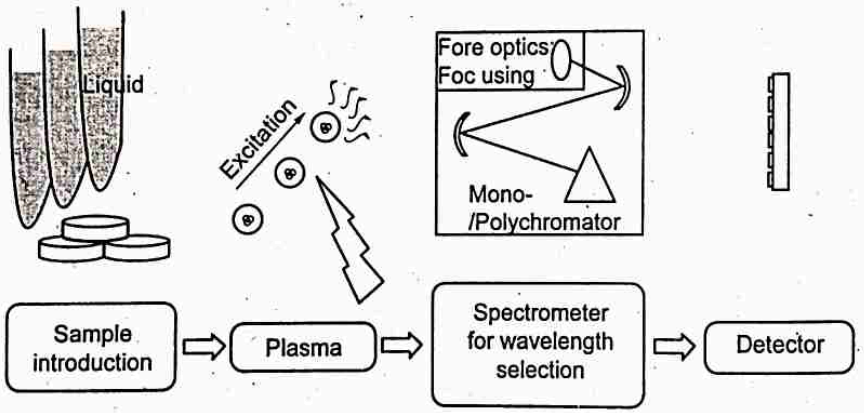


Fig. 5.10. Flow diagram of ICP-OES

1. PRINCIPLE

- When plasma energy is given to an analysis sample from outside, the component elements (atoms) is excited. When the excited atoms return to low energy position, emission rays (spectrum rays) are released and the emission rays that correspond to the photon wavelength are measured.

2. COMPONENTS

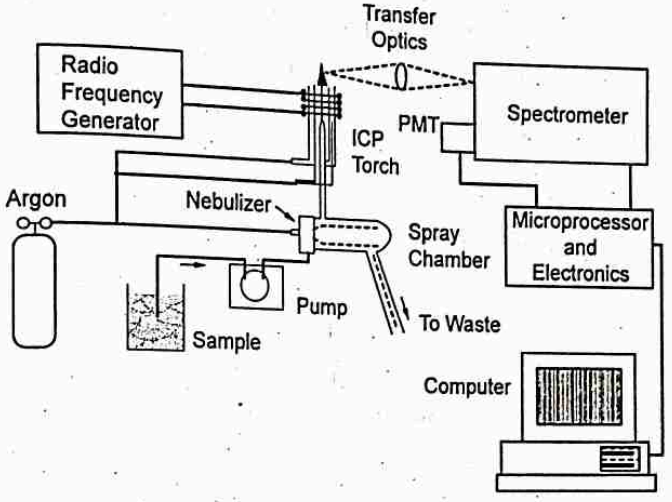


Fig. 5.11. A representation of the layout of a typical ICP-OES instrument.

3. CONSTRUCTION

- Sample introduction
- Production of emission
- Collection and detection of emission
- Signal processing and instrument control

(a) SAMPLE INTRODUCTION

1. Nebulizer

- With a nebulizer, the sample liquid is converted into an aerosol and transported to the plasma.

2. Pump

- It require for the solution to be pumped into the nebulizer, the Peristaltic pumps are almost exclusively the choice for ICP-OES applications. These pumps utilize a series of rollers that push the sample solution through the tubing using a process known as peristalsis.

3. Spray chambers

- A spray chamber is placed between the nebulizer and the torch. Only very small droplets in the aerosol are suitable for injection into the plasma, it can be injected into the plasma by spray chamber.

4. Drains

- The drain carries excess sample from the spray Chamber to a waste container can have an impact on the performance of the ICP instrument.

(b) Production of Emission

1. Torches

- It contains a ring-shaped toroidal plasma is formed, where the sample aerosol passes centrally through the hot plasma.
- The burner consists of three concentric quartz tubes. The aerosol is led with its carrier gas through the central tube.
- Between the outer and the intermediate tube a gas flow is introduced tangentially. It takes up the high-frequency energy and also prevents the torch from melting.

2. Radio Frequency Generators

- The radio frequency (RF) Generator is the device that provides the power for the generation and sustainment of the plasma discharge.
- The plasma with the energy of a high-frequency generator (in the frequency range of around 6 –100 MHz) is transferred to a gas flow at atmospheric pressure (mostly argon) in a quartz tube system with the aid of a working coil.
- The electrons take up energy and collide with atoms, by which a plasma with a temperature of up to 6000 K is formed.

(c) Collection and Detection of Emission

1. Transfer Optics

- The emission radiation from the region of the plasma known as the normal analytical zone (NAZ) is sampled for the spectrometric measurement.

2. Wavelength Dispersive Devices

- The next step in ICP-OES is the differentiation of the emission radiation from one element from the radiation emitted by other elements and molecules. The physical dispersion of the different wavelengths is done by

- Diffraction gratings
- Prisms
- Filters

- To separate polychromatic light the grating is incorporated in an optical instrument called a spectrometer. The spectrometer receives white light or polychromatic radiation and disperses it into monochromatic radiation. One or more exit slits on the exit plane or circle are then used to allow certain wavelengths to pass to the detector while blocking out other Wavelengths.
- The monochromatic radiation which is diffracted from the grating is composed primarily of wavelengths representative of the light emitted by a particular elemental or molecular species in the ICP.

3. Detectors

- Once the proper emission line has been isolated by the Spectrometer, the detector and its associated electronics are used to measure the intensity of the emission line. The most common detector is photomultiplier tube.
- A photomultiplier tube (PMT) consists of a photosensitive cathode, several dynodes and a collection anode. The dynodes are responsible for the increase in signal by electron multiplication.

(d) Signal Processing and Instrument Control

1. Signal Processing

- The electrical current measured at the anode of the PMT is converted into information that can be used by a computer.

2. Computers and Processors

- The computer to control the spectrometer and to collect, manipulate, and report analytical data, the amount of computer control over other functions of the instrument varies widely from model to model.

3. Software

- ICP-OES instrument would be that it could prepare the standards and samples, develop the analytical method, analyze the samples, report the

results, and make decisions based on those results all from a single keystroke.

4. WORKING OF ICP-OES

- The first step in an analysis is to prepare the samples and Standards for introduction to the ICP. This step depends on the physical and chemical characteristics of the samples and from simple dilution to a complex series of chemical reactions and other preparation steps.
- The next step in the analysis concerns the sample introduction method and hardware to be used. For most ICP-OES analyses, the standard sample introduction system provided with the instrument will be sufficient.
- In inductively coupled plasma-optical emission spectrometry, the sample is usually transported into the instrument as a stream of liquid sample. Inside the instrument, the liquid is converted into an aerosol through a process known as nebulization.
- The sample aerosol is then transported to the plasma where it is desolvated, vaporized, atomized, and excited and/or ionized by the plasma.
- The excited atoms and ions emit their characteristic radiation which is collected by a device that sorts the radiation by wavelength.
- The radiation is detected and turned into electronic signals that are converted into concentration information for the analyst.
- The next step in the development of an analysis methodology is to program the instrument, using the computer software provided with the instrument, to perform the data collection and processing steps.
- To do this, decisions must be made concerning the operating conditions, wavelength selection, instrument calibration, emission measurement, and the actual sample analysis.

5. Advantages

- Extremely high sensitivity
- Almost full elemental coverage without need for specific excitation sources
- Linear range of several orders of magnitude

- Very accurate quantification at low concentrations
- By using bulk samples a true bulk analysis is obtained (this is often difficult or impossible for many other methods)
- The ability to analyze most any sample type even with limited availability (most commonly samples are about 0.1-1 g but can be as small as a few milligrams)
- Even gases may be analyzed when introduced into the torch using methods such as gas chromatography.
- High sample throughput enabling the efficient analysis of large batches
- Simultaneous determination of multiple elements in each sample
- Complementary analysis to techniques like XRF
- Large dynamic linear range
- Low chemical and matrix interference effects

6. DISADVANTAGES

- Cumbersome sample preparation
- The need to generate calibration curves from samples as similar in all respects as the samples under investigation
- Initial progress is often time consuming and tedious
- In the case of failure analyses method development will often be necessary each time a new sample type is encountered
- Relatively long analysis times
- The method is inherently destructive

7. APPLICATIONS

- Trace analysis of environmental soil and water samples
- Assessment of metal ores for mass balances and process control
- Trace metal analysis of any material that can be digested into an aqueous matrix
- Boron and Lithia in glasses
- Forensic analysis

- Trace analysis of food and drink samples such as; metals in wine; and elements bound to proteins
- Metal release testing of tableware.
- Determination of toxic, trace and major constituents in coal and slags
- Analysis of low alloy steels for As, B, Bi, Ce, La, P, Sn and Ta; High-precision determination of Si in steels.
- Determination of contaminants in high-purity Al.
- Analysis of superconducting materials for trace.

5.4.6. INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY

- Inductively coupled plasma mass spectrometry (ICP-MS) is an instrumental analytical technique based on the use of a high temperature ionisation source (ICP) coupled to a mass spectrometer.
- It is an elemental analysis technology capable of detecting most of the periodic table of elements at milligram to Nano gram levels per liter.
- It is used in a variety of industries including, but not limited to, environmental monitoring, geochemical analysis, metallurgy, pharmaceutical analysis, and clinical research.

1. PRINCIPLE

- It is a type of mass spectrometry that uses an inductively coupled plasma to ionize the sample. It atomizes the sample and creates atomic and small polyatomic ions, which are then detected.
- It is known and used for its ability to detect metals and several non-metals in liquid samples at very low concentrations. It can detect different isotopes of the same element, which makes it a versatile tool in isotopic labeling.

2. SAMPLE PREPARATION

- This internal standard consists primarily of deionized water, with nitric or hydrochloric acid, and Indium and/or Gallium. Depending on the sample type, usually 5 mL of the internal standard is added to a test tube along with 10 - 500 microliters of sample.

3. COMPONENTS

- **Peristaltic Pump** - Ensures constant flow of liquid irrespective of differences in viscosity between samples, standards and blanks. Sample pumped at 1ml/min
- **Nebulizer and spray chamber** - It uses supersonic expansion of gas to turn the liquid into a fine mist, and the spray chamber then removes any droplets that are too large to be processed in the plasma. This occurs at the sample interface of the instrument.
- **Torch** - The plasma torch consists of three concentric quartz tubes through which streams of argon flow. The nebulizer gas which carries the analyte into the plasma flows in the central tube. The auxiliary gas flows around the central tube and adjusts the position of the plasma relative to the torch. The coolant gas streams tangentially through the outer tube, serving to cool the inside walls and center of the torch, and stabilizes the plasma.
- **Plasma Ionization Source** - Inductively coupled plasmas are formed by coupling energy produced by a Radio Frequency generator to the plasma support gas with an electromagnetic field. The field is produced by applying an RF power (typically 700-1500 W) to a load coil.
- **Interface Region (Skimmer cone & Sampler cone)** - A section that connects the ionizing section at ambient pressure to the mass spectrometer at high vacuum. Function is to export the ions produced in argon plasma and transport them to the mass spectrometer.
- **Ion Focusing Region** - One or more electrostatically controlled lens component made up of series of metallic plates or cylinders having a voltage placed on them Ions are separated from Photons & Neutrals
- **Mass Analyzer (Mass spectroscopy)** - Quadrupole is a sequential mass filter, which separates ions based on their m/z. Measurement of the m/z of the ion allows qualitative identification of the isotope or molecule. Magnitude of the ion current is used to provide quantitation of the amount of the analyze in the original sample.
- **Spectral Interferences** - Polyatomic or molecular Spectral Interferences severely compromise detection capability of certain elements by ICP-MS

using the Quadrupole mass analyzer technology. Generated by combination of Plasma/nebulizer Gas, solvent and matrix derived ions

- **Collision Reaction Cell (CRC)** - The CRC devices in commercial instruments have been designed to remove polyatomic species.
- **Ion Detectors**-Detector is an Electron Multiplier Device which can generate a measurable signal pulse from the impact of a single ion. Each electron which strikes a dynode releases several electrons from that surface and hence the device is called "electron multiplier".

4. WORKING OF ICP-MS

- The sample solution is introduced into the device by means of a peristaltic pump.
- There it becomes nebulized in a spray chamber.
- The resulting aerosol is injected into an argon-plasma that has a temperature of 6000-8000 K.
- Inside the plasma torch, solution is removed from the sample and also atomization and ionization occur.

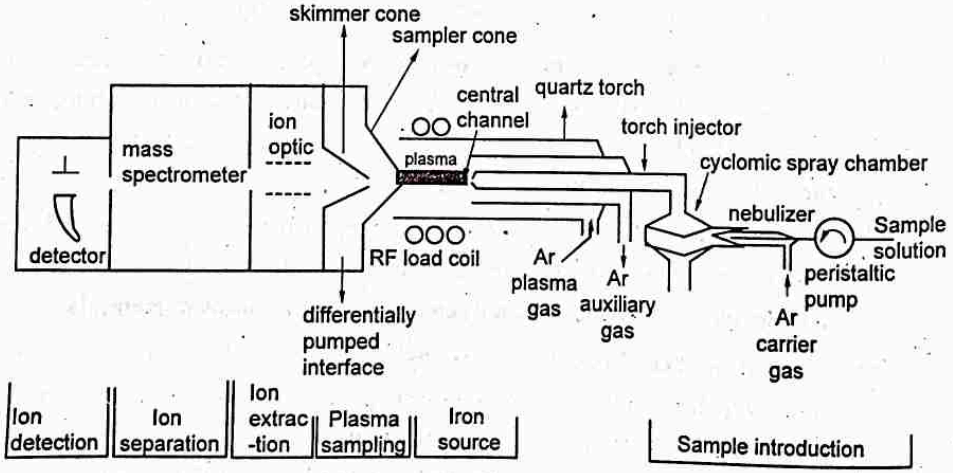


Fig. 5.12. Working flow of ICP-MS

- To be processed efficiently in the plasma, samples must be in either gas or vapor (aerosol) form. So, while gases can be analyzed directly by the plasma (e.g., when separated by gas chromatography), solids and liquids

have to be converted to aerosol form using either a nebulizer (for liquids) or an ablation device (for solids).

- Only a small amount part of the ions produced in the plasma further penetrate to the mass-spectrometer part.
- After mass separation, ions must be detected and amplified in order to determine their intensities.
- Electron multipliers (also known as secondary electron multiplier, or SEM, detectors) can detect extremely small ion currents, including even single ions, coming from the mass analyzer. They operate on the principle of secondary electron emission, in which charged particles with sufficient energy incident on a 'dynode' stimulate the emission of electrons from the surface.

5. MAINTENANCE OF ICP-MS

- Pump tubing has the tendency to stretch, which changes the amount of sample being delivered to the nebulizer
- Tip of the nebulizer should not get blocked.
- Microscopic particles can build up on the tip of the nebulizer without the operator noticing, which, over time, can cause a loss of sensitivity, imprecision, and poor long-term stability.
- Drain of spray chamber must function properly. Malfunctioning or leaking drain can produce changes in the spray chamber backpressure, producing fluctuations in the analyte signal, resulting in erratic and imprecise data
- Staining and discoloration of the outer tube of the quartz torch because of heat and the corrosiveness of the liquid sample can cause electrical arcing
- The most common types of problems associated with the interface are blocking or corrosion of the sampler cone & skimmer cone

6. ADVANTAGES

- Quantitative analysis is the fundamental tool used to determine analyte concentrations in unknown samples.
- Increased sensitivity and wide dynamic range
- Extremely low detection limits

- A large linear range
- Possibilities to detect isotope composition of elements
- Wide Elemental Coverage
- Extremely Low Detection Limits (ppt/ppm) or (ng/L to mg/L)
- Fast Analysis times (all elements at once)
- Simple Spectra
- Isotopic Information
- High Throughput & Productivity

7. APPLICATION

- Simple metal analysis during metal based drug development
- Impurity limit tests
- Metals present in Active Pharmaceutical Ingredients
- Quality Control Tests of natural products for toxic impurities testing
- Monitoring metabolites of an administered drug
- Detection of metal impurities from leachable packaging material
- For elemental speciation
- Pharmaceutical Waste Water monitoring

8. DISADVANTAGE

- The high capital cost of the instrumentation.
- Lower precision compared with atomic absorption spectrometry (AAS)
- The total dissolved salts should be less than 1000 ppm
- Severe matrix effects
- Heavier elements, such as lead, are well-suited for ICP-MS analysis, whereas lighter elements are prone to more interference.

TWO MARK QUESTIONS WITH ANSWERS

1. ***Define thermal analysis***

Thermal analysis is a form of analytical technique most commonly used in the branch of materials science where changes in the properties of materials are examined with respect to temperature.

2. ***List out various thermal properties***

- Thermo-Elastic Effect
- Specific heat
- Thermal expansion
- Thermal stress
- Thermal conductivity

3. ***What is meant by Dilatometer?***

A dilatometer is a scientific instrument that measures volume changes caused by a physical or chemical process. A familiar application of a dilatometer is the mercury-in-glass thermometer, in which the change in volume of the liquid column is read from a graduated scale.

4. ***Define Thermogravimetric analysis***

The Thermogravimetric analysis (TGA) is a type of thermo analytical testing performed on materials to determine changes in weight in relation to changes in temperature.

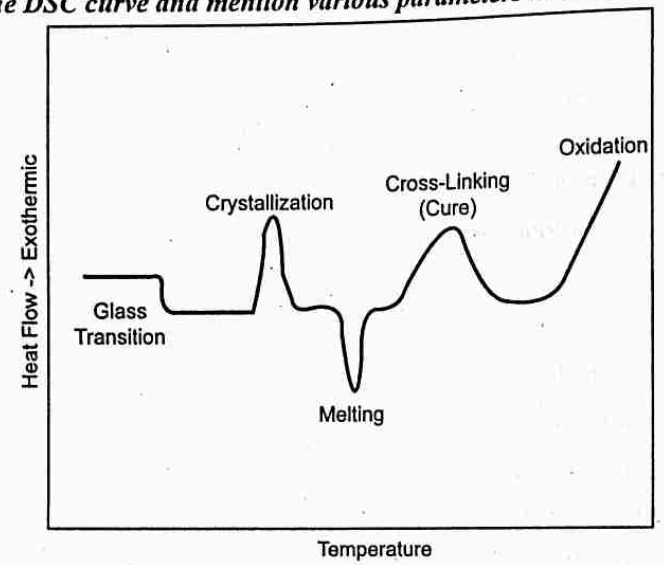
5. ***What is principle of working in Differential scanning calorimetry?***

Differential scanning calorimetry (DSC) is based on the principle; sample and reference are maintained at the same temperature, even during a thermal event (in the sample). The energy required maintaining zero temperature different between the sample and the reference is measured.

6. ***Write the methods of Differential scanning calorimetry***

- Heat flux DSC
- Power compensated DSC
- Modulated DSC
- Hyper DSC
- Pressure DSC

7. ***Draw the DSC curve and mention various parameters in curve.***



8. ***Define Differential thermal analysis.***

Differential thermal analysis (DTA) is a thermo-analytical technique which is used for thermal analysis where thermal changes can be studied. It is used to determine the oxidation process, decomposition, and loss of water or solvent.

9. ***Write about detector used in Differential thermal analysis.***

Differential Temperature Detector (Thermogram), the main function of this detector is to measure differential temperature.

10. ***Write limitation of Differential Temperature Detector***

1. There is lot of uncertainty in transition reactions and heat of fusions upto 20-50%
2. Destructive limited range of samples time consuming usually not qualitative.

11. ***Define LVDT.***

Linear Variable Differential Transformer is a common type of electromechanical transducer that can convert the rectilinear motion of an object to which it is coupled mechanically into a corresponding electrical signal.

12. ***Write the usage of LVDT in Differential thermal analysis***

Every displacement in pushrod of Differential thermal analysis is transformed into an analog signal by the LVDT, converted to digital form and then recorded in the computer system, and finally presented by the software as a dimensional change versus time or temperature.

13. ***Compare the merits and demerits of TMA, DSC, TGA.***

Technique	Employed for	Merits	Demerits
Thermomechanical analysis	Glass transition temperature, softening point, coefficient of linear thermal expansion	Straightforward method with high accuracy and applicable for all polymers	High cost
Differential scanning calorimetry	Crystalllinity, oxidation time, glass transition temperature	Limited for chlorinated polymers	High cost, qualitative result
Thermogravimetric analysis	Polymer additives, ash content, carbon black content, decomposition temperature	Straightforward method with high accuracy for all polymers	Qualitative

14. ***Write various advantages in Differential thermal analysis***

- Compactness and lightness
- Low operation voltage
- Measures large deformation
- Large actuation force
- Measures measure relaxation effects

15. ***Mention the various components of Dynamic Mechanical Analyser***

- Transducer Sensor (Linear Variable Displacement Transducer (LVDT)
- Drive shaft or probe

- Drive motor
- Stepper motor

16. *Difference between Drive motor and Stepper motor of DMA*

- Drive motor is a linear motor for probe loading which provides load for the applied force
- Stepper motor is controls the specimen dimension and measurement

17. *What are benefits of using DMA?*

- Very soft and hard samples are measured.
- Allows accurate temperature measurement.
- It can provide major and minor transitions of materials
- It is also more sensitive.
- It is able to quickly scan and calculate the modulus for a range of temperatures.

18. *Differentiate between TGA, DTA, DSC*

TGA	DTA	DSC
TGA is Thermogravimetric analysis	DTA is Differential thermal analysis	DSC is Differential scanning calorimetry
The change mass with change of temperature is analysed.	Temperature difference developed between the sample and reference is measured identically.	Heat flow is measured against temperature at particular time
Sample can be used as solid substance.	Sample can be used as solid substance.	Sample can be used as liquid substance.

19. *What are purposes of chemical analysis*

- Chemical Trace Analysis
- Elemental Trace Analysis
- Failure Analysis
- Contamination Analysis
- Materials Analysis and Testing
- Material Verification

20. *Define chromatography technique*

Chromatography is a technique for the separation of a mixture. The mixture is dissolved in a fluid called the mobile phase, which carries it through a structure holding another material called the stationary phase. The various constituents of the mixture travel at different speeds, causing them to separate.

21. *Define Wet Chemistry*

Wet Chemistry, also called wet chemical analysis, generally refers to chemistry performed on samples in the liquid phase. Since wet chemistry analysis is performed on liquid samples, this type of element analysis can often be performed on samples too small for other instrumental methods.

22. *How X Ray is utilized in XRF spectroscopy?*

X-ray fluorescence (XRF) is the emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by being bombarded with high-energy X-rays or gamma rays.

23. *What are the types of XRF spectroscopy?*

- Energy Dispersive XRF (Direct and polarized excitation)
- Wavelength Dispersive XRF

24. *What are the limitations of XRF spectroscopy?*

- XRF analyses cannot distinguish variations among isotopes of an element.
- XRF analyses cannot distinguish ions of the same element in different valence states.
- Instrumentation is fairly expensive

25. *Define inductively coupled plasma.*

An inductively coupled plasma (ICP) or transformer coupled plasma (TCP) is a type of plasma source in which the energy is supplied by electric currents which are produced by electromagnetic induction, that is, by time-varying magnetic fields.

26. *Define Nebulizer*

Nebulizers are devices that convert a liquid into an aerosol that can be transported to the plasma. This process is one of the critical steps in ICP-OES. The ideal sample introduction system would be one that delivers the

entire sample to the plasma in a form that the plasma could reproducibly desolvate, vaporize, atomize and ionize, and excite. Types of Nebulizers,

- Pneumatic nebulizer
- Babington nebulizer
- Ultrasonic nebulizer

27. ***Compare contrast between ICP OES and ICP MS***

ICP OES	ICP MS
Inductively coupled plasma optical emission spectroscopy (ICP-OES)	Inductively coupled plasma mass spectrometry (ICP-MS)
Measurement of excited atoms and ions at the wavelength characteristics for the specific elements being measured	Measures an atom's mass by mass spectrometry
Detection limit for ICP-MS can extend to parts per trillion (ppt)	Detection limit for ICP-OES is parts per billion (ppb)
ICP-OES has much higher tolerance for TDS (up to 30%)	ICP-MS has much lower tolerance for TDS (about 0.2%) although there are ways to increase the tolerance.

28. ***Define Torches in ICP OES***

- It contains a ring-shaped toroidal plasma is formed, where the sample aerosol passes centrally through the hot plasma.
- The burner consists of three con- centric quartz tubes. The aerosol is led with its carrier gas through the central tube.

29. ***List out types of dispersing unit.***

- **Prism or diffraction gratings** - The grating provides dispersion of the wavelength range of interest over a given angular range.
- **Monochromators** - Multi-element determinations using a monochromator must be sequential, as the monochromator can observe only one line at a time owing to single secondary slit.

- **Polychromators** - Polychromators have a permanently fixed secondary slit for certain individual wavelengths (individual elements).

REVIEW QUESTIONS

1. Explain differential scanning calorimetry with working principle and write its applications.

   Ans: Section No. 5.2.4 **Page No:** 5.5

2. Explain the types of differential scanning calorimetry with neat sketch.

   Ans: Section No. 5.2.4 **Page No:** 5.5

3. Describe the various components of differential thermal analysis with working.

   Ans: Section No. 5.2.5 **Page No:** 5.10

4. What are the benefits and limitation of differential thermal analysis?

   Ans: Section No. 5.25 **Page No:** 5.13

5. Compare the curve of DTA and DSC.

   Ans: Section No. 5.2.4, 5.2.5 **Page No:** 5.8, 5.12

6. Explain the various loading condition in thermo mechanical analysis.

   Ans: Section No. 5.26 **Page No:** 5.15

7. Write short note on
   - DTA
   - DSC
   - DMA
   - TMA

   Ans: Section No. 5.2.4 to 5.2.7 **Page No:** 5.5 to 5.17

8. Explain in detail about thermo mechanical dynamic analysis.

   Ans: Section No. 5.2.7 **Page No:** 5.17

9. What are the various applications, advantages, disadvantages of x-ray fluorescence?

Ans: Section No. 5.3.5 **Page No:** 5.24

10. Describe the process of production of plasma in ICP.

Ans: Section No. 5.4.3 **Page No:** 5.30

11. Explain the various components working in Inductively Coupled Plasma Optical Emission Spectroscopy.

Ans: Section No. 5.4.5 **Page No:** 5.32

12. Write comparison between various features of ICP OES and ICP MS.

Ans: Section No. 5.4.5, 5.4.6 **Page No:** 5.32, 5.38

13. Write short note on

- ICP
- ICP OES
- ICP MS

Ans: Section No. 5.4.3.2, 5.4.5, 5.4.6 **Page No:** 5.30, 5.32, 5.38

14. Explain the working condition, advantages and disadvantages of ICP MS.

Ans: Section No. 5.4.6 **Page No:** 5.38

MODEL QUESTION PAPER - 1

B.E./B.Tech., DEGREE EXAMINATIONS,

Electronics and Communication Engineering,

OML751 - TESTING OF MATERIALS

(Regulation 2017)

Time: Three Hours **Maximum: 100 marks**

Answer ALL questions

PART A (2 × 10 = 20 Marks)

1. ***Why more concentration is needed for selection of materials?***

Material selection is one of the foremost functions of effective engineering design as it determines the reliability of the design in terms of industrial and economical aspects.

Iterative in nature, there is a strong element of trial and error where an initial design is done and then analyzed, tested, and subjected to trial production. Changes may be made at any stage of the process to satisfy requirements not previously considered or problems just discovered.

2. ***Define prototype.***

A proto type, or trial model, is often made and subjected to simulated service testing to demonstrate whether or not a machine or vehicle functions properly.

3. ***Define strain hardening and proof stress.***

Strain hardening: This increase in the tensile strength of the material is due to strain hardening which is due to the increased dislocations interactions during the deformation of the tensile test. This is called Strain-hardening.

Proof Stress: The stress that causes a percentage increase in gauge length. It can be found by drawing a line parallel to the straight part of the graph. A value can be taken from the vertical axis.

4. ***What are advantages made the choice of Brinell hardness test?***

- A choice can be made between a large numbers of test forces.
- The influence of surface scratches and roughness will be less in the Brinell test than other hardness tests.
- The specimen surface can be rough.

- Suitable for hardness tests on large blanks such as forged pieces, castings and hot-rolled etc
- Measurement is usually not affected by movement of the specimen

5. ***How densities of material influence the radiographic testing?***

(*a*) If an object has a high density, i.e., a thicker object, it absorbs more radiation causing less radiation to hit the film, which produces a lighter image.

(*b*) If an object has a low density, i.e., when the through section is reduced or there is a lower-density material such as slag (compared to the surrounding material), it will absorb less radiation causing more radiation to hit the film, producing a darker image.

6. ***What aids used for visual testing?***

(*i*) Magnifying glasses
(*ii*) Fillet weld gauge
(*iii*) Microscopes
(*iv*) Computer equipment (remote viewing)
(*v*) Illuminated magnifier
(*vi*) Holography

7. ***Why specimen preparation is important in microscopic technique?***

- Specimen preparation is important in any microscopic technique with proper preparation methods facilitating examination and interpretation of micro structural features.
- Improper preparation methods may obscure features, and even create artifacts that may be misinterpreted.

8. ***Difference between Raman and IR spectroscopy.***

Raman spectroscopy	Infrared spectroscopy
It is due to the scattering of light by vibrating molecules	It is the result of absorption of light by vibrating molecules.
The vibration is active if it causes to change in polarizability	The vibration is active if it causes to change in dipole moment
The molecule need not possess a permanent dipole moment	The vibration concerned change in dipole moment due to vibration

9. ***Define inductively coupled plasma.***

An inductively coupled plasma (ICP) or transformer coupled plasma (TCP) is a type of plasma source in which the energy is supplied by electric currents which are produced by electromagnetic induction, that is, by time-varying magnetic fields.

10. ***How x ray is utilized in XRF spectroscopy?***

X-ray fluorescence (XRF) is the emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by being bombarded with high-energy X-rays or gamma rays.

PART B (5 × 13 = 65 Marks)

11. (*a*) (*i*) What are the advantages and disadvantages encountered by various material testing? (7)

Ans: Refer Section No. 1.2 Page No.1.10

(*ii*) What are criteria that affect the selection of materials? (6)

Ans: Refer Section No. 1.4 Page No. 1.19

[OR]

(*b*) What are steps to be followed during selection of materials? Explain each step in detail. (13)

Ans: Refer Section No. 1.4 Page No. 1.17

12. (*a*) Explain the working principles of machines used to conduct Charpy and Izod impact test. How specimens are put–up in both the tests? (13)

Ans: Refer Section No. 2.12 Page No. 2.32

[OR]

(*b*) Write short note following terms

(*i*) True stress - strain and engineering stress - strain. (3)
(*ii*) Stages of creep (5)
(*iii*) S-N curve (5)

Ans: Refer Page No.2.53, 2.59

13. (*a*) What do you understand by NDT test? Explain the role of Nondestructive testing in manufacturing process. (13)

Ans: Refer Section No. 3.1 Page No. 3.1

[OR]

(*b*) Write short note on

(*i*) Eddy current testing. (5)

(*ii*) Acoustic emission testing. (3)

(*iii*) Liquid penetration Test. (5)

Ans: Refer Section No. 3.2.8, 3.2.11 & 3.2.2 Page No. 3.34, 3.47 & 3.10

14. (*a*) Explain SEM with principle of working, advantages, limitation and various applications.

Ans: Refer Section No. 4.6.1 Page No. 4.14

[OR]

(*b*) (*i*) Write short note on various method of sample preparation in SEM and TEM. (5)

Ans: Refer Section No. 4.6 Page No. 4.17 & 4.21

(*ii*) What is optical microscope and how it is working? (8)

Ans: Refer Section No. 4.5 Page No. 4.9

15. (*a*) Describe the various components of differential thermal analysis with working. (13)

Ans: Refer Section No. 5.2.5 Page No. 5.10

[OR]

(*b*) Write short note on (13)

- ❖ DTA
- ❖ DSC
- ❖ DMA
- ❖ TMA

Ans: Refer Section No. 5.2.4 to 5.2.7 Page No. 5.5 to 5.17

PART C (1 × 15 = 15 Marks)

16. (*a*) The steel deck truss bridge in Trichy is 90-year-old, constructed across Kollidam River that connects Srirangam with mainland Tiruchirapalli. Use proper NDT test for monitoring bridge condition and explain step procedure of using NDT in bridge.

[OR]

(*b*) Write an case study on NDT test used in inspection on welding works.

MODEL QUESTION PAPER - 2

B.E./B.Tech., DEGREE EXAMINATIONS,

Electronics and Communication Engineering,

OML751 - TESTING OF MATERIALS

(Regulation 2017)

Time: Three Hours **Maximum: 100 marks**

Answer ALL questions

PART A (2 × 10 = 20 Marks)

1. ***What are the benefits of testing?***
   - Safety issues can be identified
   - It provides reliability
   - It is cost effective
   - It offers reassurance
2. ***What are the tests used to testing metals?***

   Major test used for testing metals are destructive one i.e., Test, Shear (Torsion test),Test, Creep Test, Bending test etc.
3. ***Define Endurance limit.***

   Endurance limit (fatigue limit) is the maximum fatigue stress applied to material without failure. It is 50% of fatigue failure load which is preferred for design criteria
4. ***What is role of SN curve in fatigue mechanism?***

   S-N curves are derived from tests on samples of the material to be characterized, where a regular sinusoidal stress is applied by a testing machine which also counts the number of cycles to failure.
5. ***Define NDT.***

   Non-destructive testing (NDT) is a testing and analysis technique used by industry to evaluate the properties of a material, component, structure or system

for characteristic differences or welding defects and discontinuities without causing damage to the original part.

6. ***What is purpose penetrant in liquid penetrant inspection?***

The liquid, by capillary action, will penetrate the discontinuities and the excess remaining on the surface will be removed by a suitable cleaning system. It will be highly visible or fluoresce brightly to produce easy to see indications.

7. ***State Diffraction Principle.***

Bragg's law is which determines the angles of coherent and incoherent scattering from a crystal lattice. When X-rays are incident on a particular atom, they make an electronic cloud move just like an electromagnetic wave.

8. ***What are the methods of Spectroscopy?***

- Ultraviolet-visible spectroscopy (UV-vis)
- Electron Spin Resonance spectroscopy
- Atomic spectroscopy
- infrared spectroscopy and Raman spectroscopy
- Mass spectrometry
- Nuclear spectroscopy(nuclear magnetic resonance)

9. ***Define Nebulizer.***

- Nebulizers are devices that convert a liquid into an aerosol that can be transported to the plasma. This process is one of the critical steps in ICP-OES. The ideal sample introduction system would be one that delivers the entire sample to the plasma in a form that the plasma could reproducibly desolvate, vaporize, atomize and ionize, and excite

10. ***What is meant by Dilatometer?***

A dilatometer is a scientific instrument that measures volume changes caused by a physical or chemical process. A familiar application of a dilatometer is the mercury-in-glass thermometer, in which the change in volume of the liquid column is read from a graduated scale.

PART B (5 × 13 = 65 Marks)

11. (*a*) Explain various stages in development of testing in detail. What is the purpose of developing a test? Explain with few examples.

Ans: Refer Section No. 1.5 Page No. 1.22

[OR]

(*b*) (*i*) How will you represent the result analysis of testing? (8)

Ans: Refer Section No. 1.8 Page No. 1.32

(*ii*) Differentiate between NDT and destructive testing. (5)

Ans: Refer Section No. 1.2 Page No. 1.14

12. (*a*) What are the various destructive tests available and which is more suitable to the hardness of material? (13)

Ans: Refer Section No. 2.2 Page No. 2.4, 2.6

[OR]

(*b*) What are properties arrived from the bending test? How do you relate with failure of section? (13)

Ans: Refer Section No. 2.36 Page No. 2.38

13. (*a*) (*i*) Differentiate between Radiography, Eddy current and Ultrasonic testing. (7)

Ans: Refer Page No. 3.61

(*ii*) What is ultrasonic testing? Explain types of transducer. (6)

Ans: Refer Section No. 3.2.10 Page No. 3.43

[OR]

(*b*) Explain the penetration test with step process and its application. What are the various advantages and disadvantages of penetration test. (13)

Ans: Refer Section No. 3.2.2 Page No. 3.10

14. (*a*) (*i*) Difference between optical and electron microscope.

(*ii*) Write about advantages and limitation of TEM.

Ans: Refer Section No. 4.6.2 Page No. 4.25, 4.55

[OR]

(*b*) (*i*) Write short note on Mass spectroscopy. (5)

Ans: Refer Section No. 4.8 Page No. 4.45

(*ii*) Write major contrast between SEM and TEM (8)

Ans: Refer Section No. 4.6.3 Page No. 4.26

15. (*a*) Explain the types of differential scanning calorimetry with neat sketch. (13)

Ans: Refer Section No. 5.2.4 Page No. 5.5

[OR]

(*b*) Write short note on (13)

- ICP
- ICP OES
- ICP MS

Ans: Refer Section No. 5.4.3.2, 5.4.5 & 5.4.6 Page No. 5.30, 5.32 & 5.38

PART C (1 × 15 = 15 Marks)

16. (*a*) In the steel industry, iron rod is manufactured. Now, the iron rod is need to quality check. What is the quickest test available for testing various properties?

Ans: Refer Section No. 2.10 Page No. 2.25

[OR]

(*b*) In the construction site of steel cell phone tower, the quality engineer need to the check bold quality used for connection purpose. What is better test used for checking bold that used in connection? Explain with experimental procedure with advantages and limitation.

Ans: Refer Section No. 2.15 Page No. 2.40

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REFERENCES

1. Fundamentals of Materials Science for Technologists: Properties, Testing By Larry Horath
2. Testing of Metals By AlokNayar
3. Material science and engineering : an introduction by william D. callister
4. Mechanical behavior of material by marc andremayers and Krishna kumarchawla
5. Mechanical behavior of material by Norman E.Dowling
6. Mechanical behavior of Engineering material by JoachinRoesler, HaraldHarders and Martin Baeker
7. NDT Guidance-Document: An introduction to common NDT methods.
8. Inspection methods-overview and comparison by ASM international
9. ICP - OPTICAL emission spectroscopy : a review by Somsubhra Ghosh and david banji
10. Atomic emission methods and instrumentation by Sandra L Bonchi, Grace K zoorob and Joesph A Caruso.
11. ICP- Mass spectroscopy methods by D Beauchemin
12. Thermo mechanical , dynamic mechanical and dielectric methods by Duncan M price
13. Essentials of pharmaceutical analysis by Muhammad sajid hamid akash and kanwal rehman
14. Impedance spectroscopy theory , experimental and application by Evgenij barsoukov and J.Ross Macdonald
15. Hand book of plastic testing and failure analysis by Vishnu shah
16. IS 1121-4(1974):Method of determining strength properties
17. IS 1608 (2005): Mechanical testing of metals - Tensile testing.
18. Testing of materials by Vernon John
19. Building and Construction Materials By Gambhir
20. Mechanical Properties and Working of Metals and Alloys by A. Bhaduri
21. Materials Science and Engineering by Freudenberger.J
22. www.wikepedia.org (general introduction to all topics)