## UNIT V APPLICATIONS AND USES

Selection of Materials for Biomedical Applications, Medical Products, Materials in Electronic Packaging, Advanced Materials in Sports Equipment, Materials Selection for Wear Resistance, Advanced Materials in Telecommunications, Using Composites Manufacture and Assembly with Plastics, fiber and Diamond Films.

### Selection of Materials for Biomedical Applications

Because of its primary role in mechanical support, the stem of a femoral prosthesis can realistically be manufactured from a metal, a ceramic, or a composite material.

### Load Support

Because of its primary role in mechanical support, the stem of a femoral prosthesis can realistically be manufactured from a metal, a ceramic, or a composite material. For this discussion, composite materials will not be included, as they have not yet been utilized in commercially available total joint replacements (they are available for bone plate applications).

### Strength

The first property to be considered for a load-bearing implant is its mechanical strength. The loading of the femur is dynamic and, while estimated to range up to 8 times body

weight, is difficult to determine precisely. Therefore, as the implant will be loaded in essentially the same way as the natural bone, it is reasonable to assume that a material that will provide the same or greater load-bearing capacity as bone will meet the necessary mechanical requirements. Based on the cyclic loading that a material is likely to undergo when implanted in the body, it is reasonable when evaluating selections to compare the endurance limit of the materials under consideration to the experimentally determined strength values for bone.

**Joint Motion**

***Friction.*** Frictional forces between the articulating surfaces of a joint have two primary effects: (1) to increase the muscle force required to overcome the internal friction and allow motion to occur and (2) to increase the torque experienced by the implant and/or bone, such as at the location of the femoral neck. Large internal bending moments due to high frictional forces may lead to failure of the implant, and therefore should be avoided.

**Wear**

Whenever contact surfaces and motion are combined, material wear must be taken into consideration. Wear is the process whereby one object, through motion, removes material from the surface of the contacting object. Generally, the harder material will cause wear to occur on the softer material. Three basic types of wear can occur: abrasive wear, adhesive wear, and third body wear. Abrasive wear exists when a hard material, such as a metal, moves cyclically against a soft material, such as a polymer. Adhesive wear involves the sliding motion of two similar materials, where molecular bonds can be formed at the interface of the structures. In rough materials, the surfaces appear as a series of peaks and valleys. The two articulating surfaces typically come into contact at the peaks of the surface roughness, concentrating the contact load over a much smaller area and increasing the contact stress. As the molecular bonds between the objects are broken through motion, they also break off particles of the underlying material. Third-body wear

includes the effect of particles between the articulating surfaces that tend to accelerate wear. The wear rate, or volume of wear particles produced ($V$), can be approximated for adhesive wear by the equation

$$V = \frac{kF_n x}{3p}$$

**Biocompatibility**

Once a material is selected for an implant application based on the functional requirements, it must be evaluated in terms of material–body interactions. An implant material will react chemically with the local environment, with the type of reaction dependent on the class of material. Metals are susceptible to corrosion, polymers experience leaching and absorption, while ceramics are generally considered to be chemically inert—unless designed to be bioactive. The effects of chemical degradation may affect both the tissue and the material itself, especially its mechanical properties, and so both aspects must be considered. In addition, degradation products can affect the physiology locally, at a remote location, or systemically.

**Corrosion**

Metallic materials are susceptible to corrosion, particularly in the ionic fluid environment of the body. To assess the corrosion potential of a metal, it is necessary to examine the half-cell potential of that metal—which will act as an anode when it releases electrons—with respect to the material acting as the cathode. This cathode may be another metal or the ionic environment itself. An electrochemical series lists the half-cell potentials of metals in order from the most noble (or cathodic) to the most anodic. When two materials are in contact with each other directly or through an ionic solution, the metal listed first in the list will act as the cathode while the other will behave as the anode. Practical electrochemical series typically relate half-cell potentials as measured in an application-specific environment and may include alloys. This contrasts with ideal series, which list only pure metals as measured with respect to a hydrogen half cell reaction.

**Leaching and Absorption**

Polymers placed in a fluid environment can experience two opposite phenomena. In leaching, unreacted monomer molecules, fillers, or small chains of polymers can diffuse from the bulk of the polymer to the surrounding fluid. As in corrosion products, these released molecules may have a negative effect on the local physiology or, if transported through the bloodstream or lymphatic system, on systemic or remote processes. In addition, significant leaching may reduce the density of the polymer and consequently have an adverse effect on the properties of the structure.

All materials, including metals and ceramics, can absorb molecules particularly water—from the surrounding environment. However, this occurs much more readily in the relatively loosely bonded polymers. Absorption in polymers can also result in swelling, due to their low elastic modulus, which may cause geometric changes that interfere with the performance of an implant. The strain that a polymeric object experiences due to swelling may induce cracks and may also reduce the ultimate strength of the object. This latter phenomenon occurs because, due to the new baseline strain in the material, less stress is needed to reach the material's ultimate strain. If the absorbed molecules are small, such as water, they will act as plasticizers and weaken the bonds between the polymer chains, thus reducing the Young modulus of the material. If a polymer is hydrophobic in nature, it is less likely to absorb water. However, absorption of nonpolar molecules such as lipids may still occur.

**BLOOD-CONTACTING BIOMATERIALS: VASCULAR PROSTHESES**

When blood vessels are damaged through injury or disease, they often must be replaced or bypassed in order to maintain adequate blood flow to and from the regions of the body. Disease-induced damage, such as atherosclerosis and aneurisms, occurs more often in arteries than in veins, due in large part to the higher working pressure of the blood within these vessels. Injury can occur to any blood vessel; however, collateral circulation typically eliminates the need to replace small veins, and the low venous return pressure

provides an environment in the larger veins that is much more conducive to traditional surgical repair or auto graft use. As a result, this section will focus on the selection of materials for the development of arterial prostheses.

**Biocompatibility**

The primary functional need of a blood vessel—transport of blood—can easily be met through general implant design. However, due to the delicate nature of blood cells and the ease at which the clotting cascade can be initiated, biocompatibility issues place substantial limitations on material selection for this application. The natural vessel provides an optimal environment for blood flow, and the mimicking or replacing of its intimal layer is one of the underlying ideas in work to improve biocompatibility in vascular grafts.

There are few, if any, current implants that can be described as perfectly meeting their design goals and constraints so that no further investigation of design or material selection is warranted. As materials continue to be developed, whether specifically for biomedical applications or in some different discipline, the selection of biomaterials for implants will remain a challenge in the design of the optimum implant. The evolution of tissue engineering from a bench-top science to a clinically workable tool for new implant design will also open new doors for the development and use of biomaterials. In all of these cases, however, the same principles apply to the selection of a material for a biomedical application.

The selection process can be summarized in the following way:

1. Determine the functional requirements of the material for the particular application (preferably with an idea of the overall design in hand).
2. Select a group of materials that appear to meet those functional requirements and ensure that all confirming tests are conducted in an environment that simulates human physiology.
3. Determine the biocompatibility of the materials in terms of material degradation, tissue effects, blood compatibility, implant fixation, and long term physiologic consequences.

4. Complete the design and approval process, with mechanisms in place to obtain data on functional or material complications for many years after clinical use is initiated.

## 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 properties 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. |
| 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 (e) 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 Materials of | Certain amount of energy is involved in this of change of phase, When a material changes phase from solid to liquid or from liquid to gas. |

## 5.2.2THERMAL TESTING

- Thermal Testing involves testing a product at the extremes of its intended use thermal environment for heating rate, temperature and airflow on gaseous atmosphere or vacuum with measuring case temperatures of individual components to determine the effect on product performance an long-term reliability.
- It measures based on dynamic relationships between temperature, Mas 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 |

## 5.2.3THERMOGRAVIMETRIC 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 in response time is more rapid. It is also capable of higher resolution the heat flux DSC.
- This makes power compensated DSC well suited for kinetics studies which fast equilibrations to new temperature settings are needed.

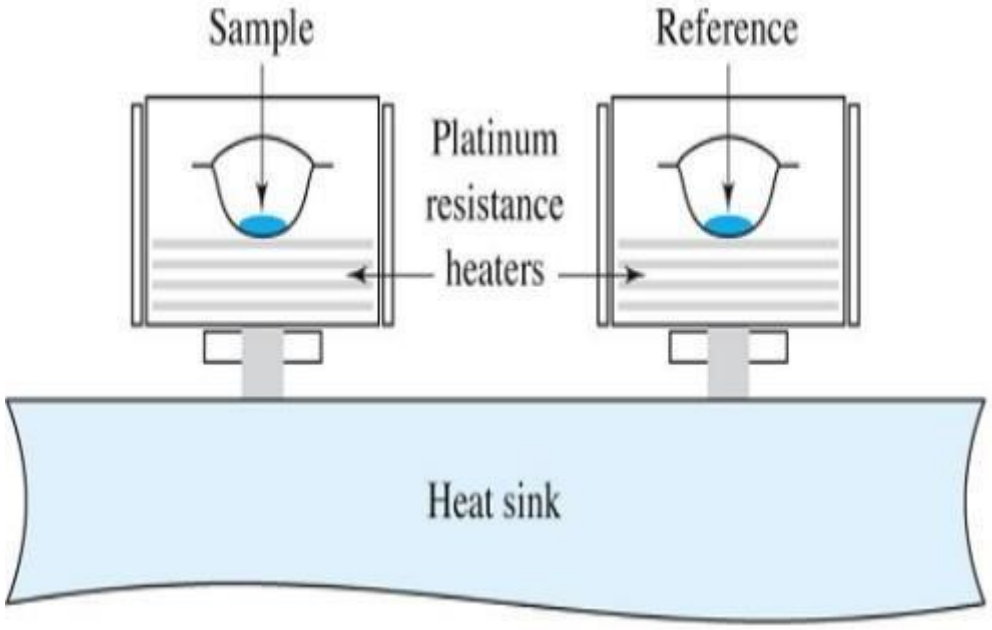



***Fig. 5.1. Typical arrangement of Power compensated DSC***

**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.

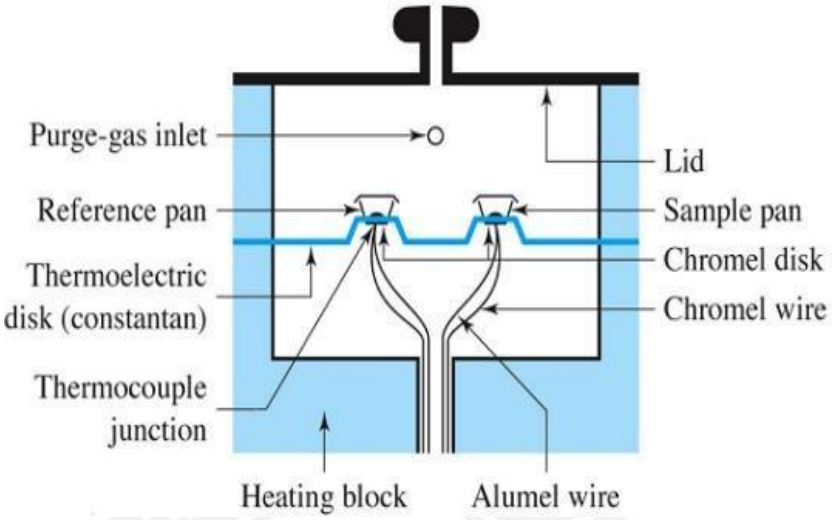



***Fig. 5.2. Typical arrangement of heat flux DSC***

**(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

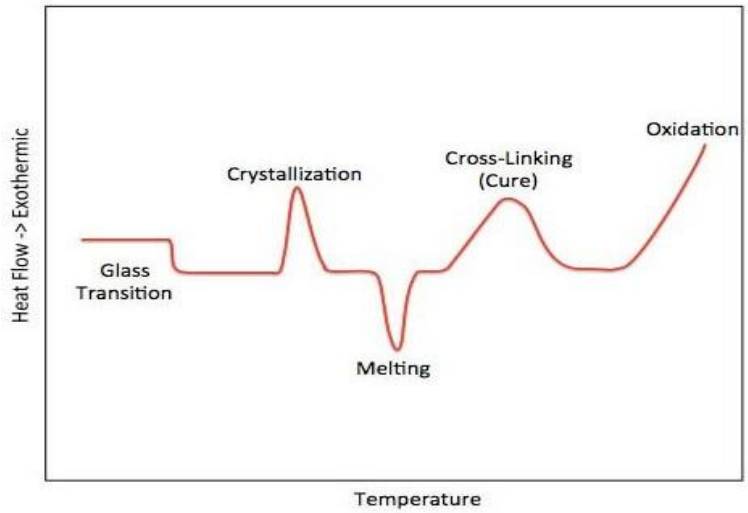



***Fig. 5.3. DSC Curve***

## Factors Affecting DSC Curve

***Table 5.3. Factors Affecting DSC Curve***

| Instrumental Factors | Sample Characteristic Factors |
|---|---|
| ❖ Furnace heating rate<br>❖ Recording or chart speed<br>❖ Furnace atmosphere<br>❖ Geometry of sample holder/location of sensors<br>❖ Sensitivity of the recording System<br>❖ Composition of sample containers | ❖ Amount of sample<br>❖ Nature of sample<br>❖ Sample packing<br>❖ Solubility of evolved gases in the sample<br>❖ Particle size<br>❖ Heat of reaction<br>❖ 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.

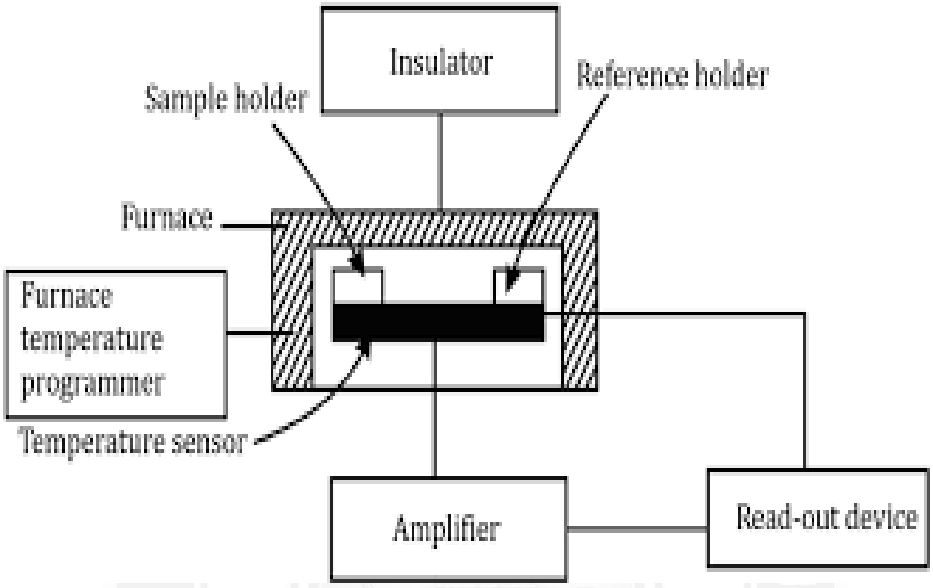



***Fig. 5.4. Cross section of DTA***

## 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 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

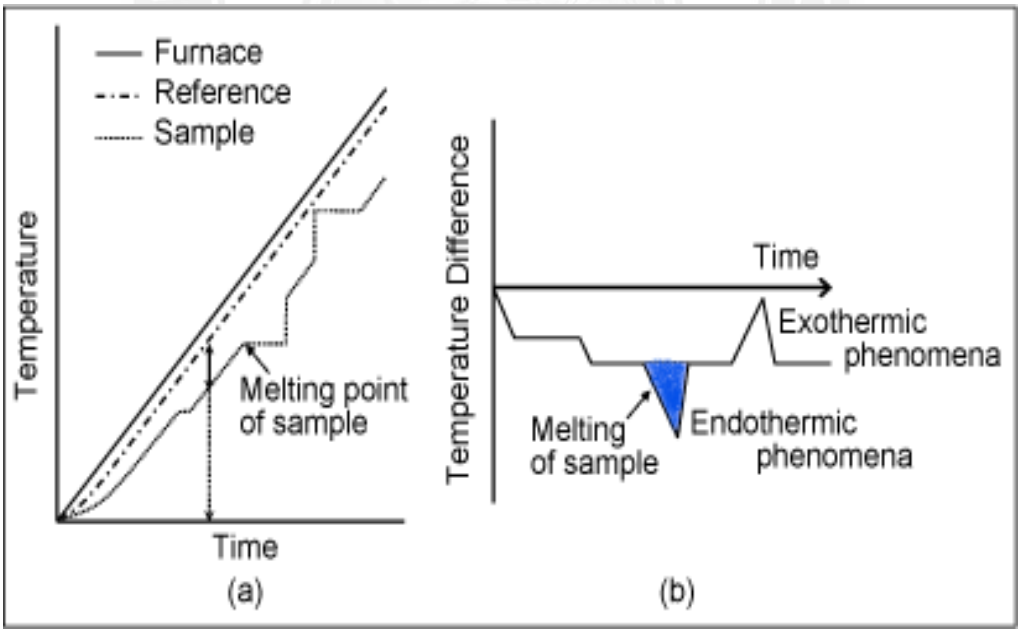



***Fig. 5.5 (a) The DTA curve or thermo gram is a plot between differential temperature and time. (b) DTA curve may be endothermic (downward plot) orexothermic (upward plot)***

**Factors affecting DTA curve**

*Table 5.4. Factors affecting DTA curve*

| Sample factors | Instrumental factors | Physical factors |
|---|---|---|
| ❖ Amount of the sample.<br>❖ Packing density.<br>❖ Particle size of the sample material.<br>❖ Degree of crystallinity.<br>❖ Heat capacity.<br>❖ Thermal conductivity.<br>❖ Dilutes of the diluents.<br>❖ Swelling of the sample.<br>❖ Shrinkage of the sample | ❖ Size or shape of the holders.<br>❖ Material of the sample holder.<br>❖ Recording system sensitivity.<br>❖ Rate of heating of the sample.<br>❖ Atmosphere around the sample.<br>❖ Thermocouple location in the sample.<br>❖ Instrumental design. | ❖ Adsorption.<br>❖ Change in the crystal structure.<br>❖ Crystallization.<br>❖ Desorption.<br>❖ Change in the crystal structure.<br>❖ Vaporization.<br>❖ Sublimation.<br>❖ 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
- Polymer's 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

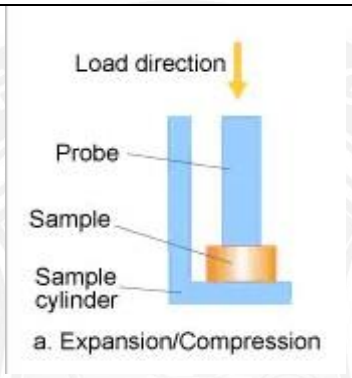
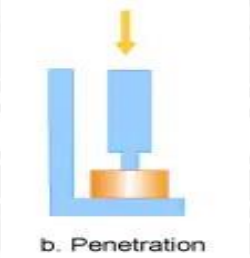
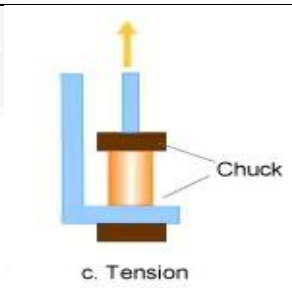
- Thermo mechanical analysis (TMA) is used to measure the dimensional changes of a material as a function of temperature by applying stress. The stress may be compression, tension, flexure or torsion.

## 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/<br><br>Compression<br><br>Probe |  | 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 |  | It is used for the measurement softening temperature. |
| (c) Tension Probe |  | It is used for the measurement of the thermal expansion and the thermal shrinkage of the sample such as the film and the fibre. |

## 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.

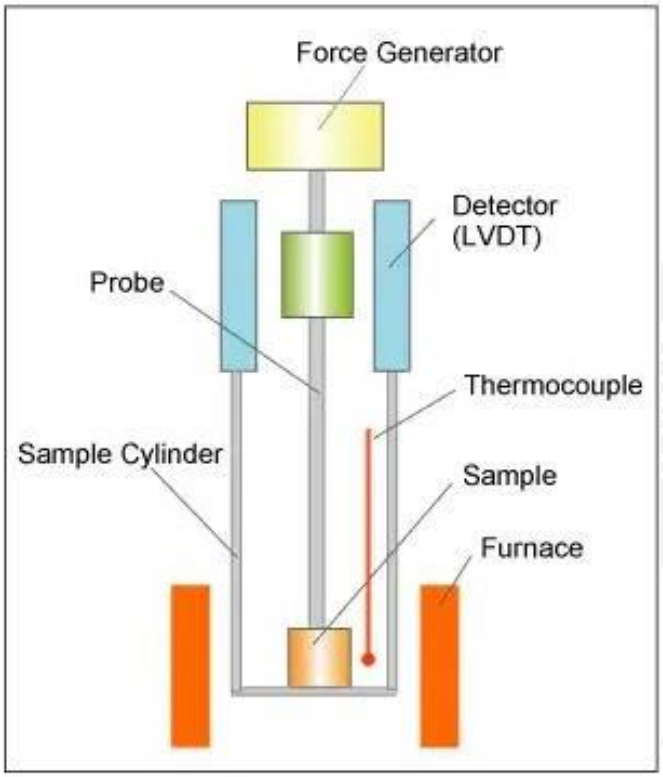



***Fig. 5.6. Working of Thermo mechanical analyser***

- The thermocouple for temperature measurement is located near the sample. The rate of 5 degrees 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 Temperature 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 analuzers 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.

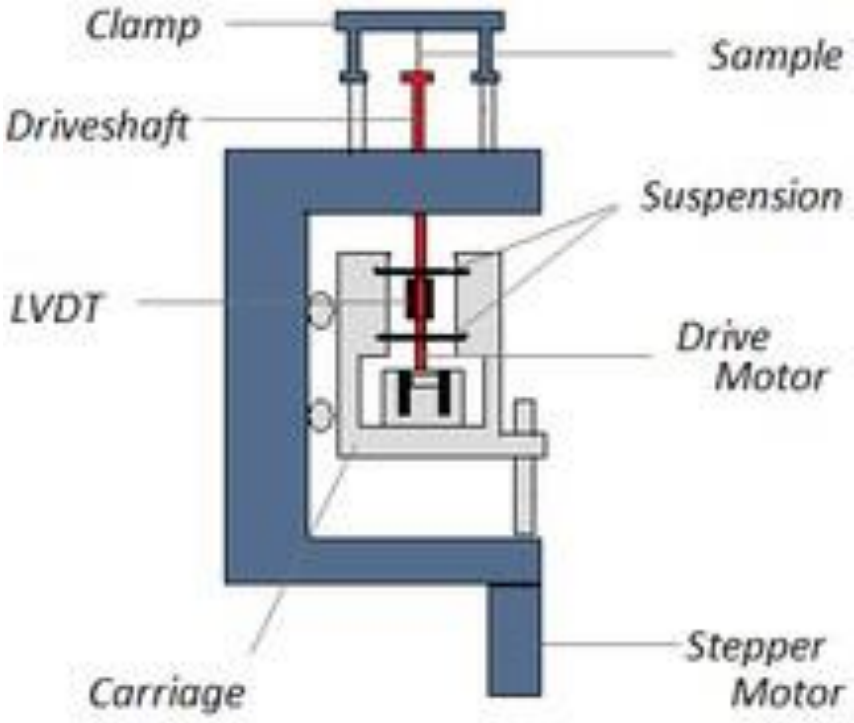
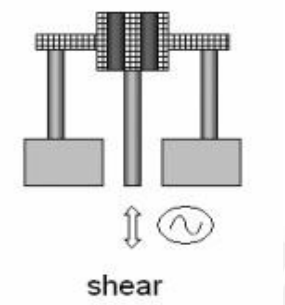
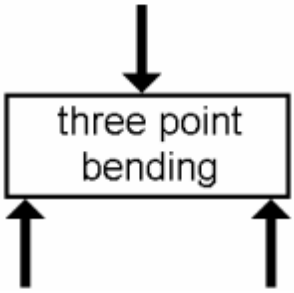
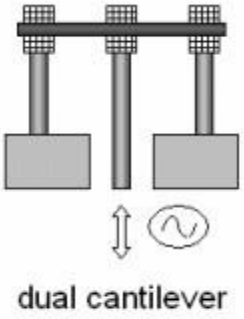


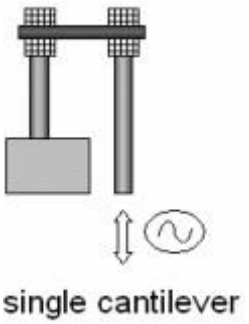
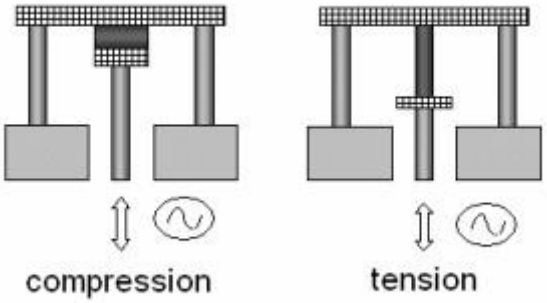

***Fig. 5.7. Cross section of Thermo mechanical dynamic analyser***

## 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 modulus materials |

## 6. DIFFERENT LOADING MODES

- The most suitable type should be selected depending on the sample shapes, 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 1 mN 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