Non - Metallic Materials

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Module - 10
Measurement of the mechanical electrical, thermal, magnetic and optical properties of non - metallic materials
Lecture - 52
Thermal analysis techniques: Differential scanning calorimetry and thermogravimetry

Welcome to my course Non-Metallic Materials and this is module number 10, Measurement of the mechanical, electrical, thermal, magnetic and optical properties of non-metallic materials. And, we are in lecture number 52 – Thermal analysis technique, particularly differential scanning calorimetry and thermo-gravimetry. This I will be discussing.

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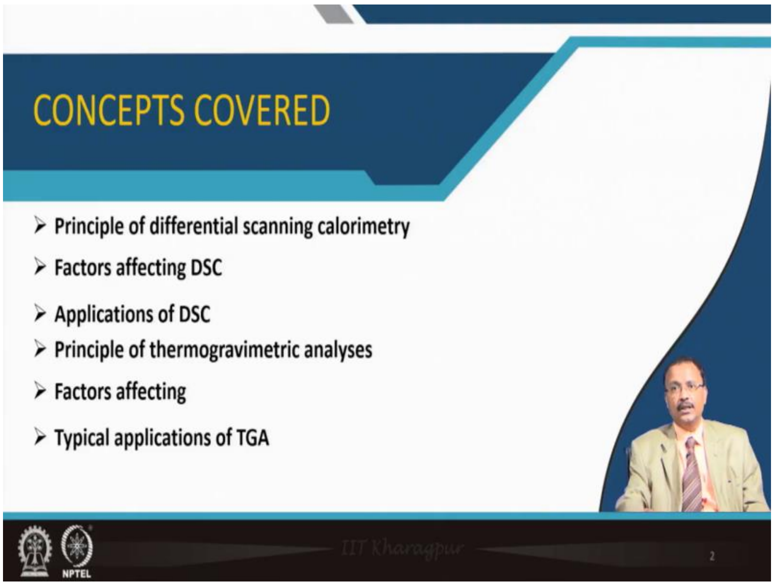

CONCEPTS COVERED

- Principle of differential scanning calorimetry
- Factors affecting DSC
- Applications of DSC
- Principle of thermogravimetric analyses
- Factors affecting
- Typical applications of TGA

So, principle of the differential scanning calorimetry will introduce and in one of my earlier lectures we already talked about differential thermal analysis as well as TGA we have introduced the concept and now, in this lecture we will see that what are the factors that affect the DSC or thermogravimetry analysis of a non-metallic materials.

What are their typical applications and what are the principles of thermogravimetric analysis, what are the factors that affect TGA analysis, and typical applications of TGA.

So, this lecture is devoted to what to do, how to do this measurement and what information that you get out of this measurement and what are the factors that influence your measurement result and this is a elaboration in this particular lecture.

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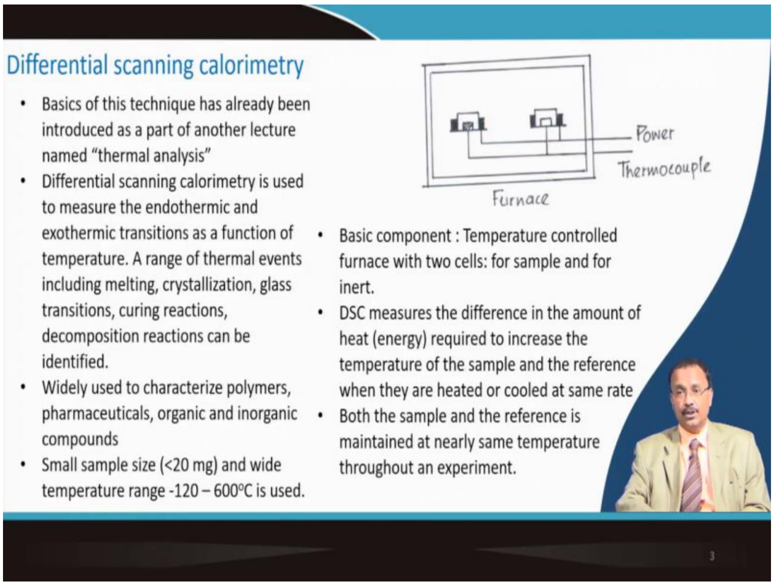

Differential scanning calorimetry

- Basics of this technique has already been introduced as a part of another lecture named "thermal analysis"
- Differential scanning calorimetry is used to measure the endothermic and exothermic transitions as a function of temperature. A range of thermal events including melting, crystallization, glass transitions, curing reactions, decomposition reactions can be identified.
- Widely used to characterize polymers, pharmaceuticals, organic and inorganic compounds
- Small sample size (<20 mg) and wide temperature range -120 – 600°C is used.
- Basic component : Temperature controlled furnace with two cells: for sample and for inert.
- DSC measures the difference in the amount of heat (energy) required to increase the temperature of the sample and the reference when they are heated or cooled at same rate
- Both the sample and the reference is maintained at nearly same temperature throughout an experiment.

So, differential scanning calorimetry the basics techniques the basics of this techniques have already introduced as a part of another lecture. So, the DSC, that is used to measure the endothermic and exothermic transition as a function of temperature.

So, a range of thermal events that is possible melting, crystallization, glass transition, curing reactions, decomposition reactions all these reactions can be identified and this technique is widely used in case of polymer pharmaceuticals organic as well as inorganic compounds and very small sample size is required may be less than 20 milligram.

And, a wide temperature range is available in the furnace it can run this measurement starting from minus 120 to 600 and in some instrument it is about 900 degree Celsius also you can go as a part of the high temperature measurement.

So, basic component of the DSC is a temperature control furnace with two cells for the sample one is for the sample another is one for the inert and why the inert and is required along with the sample that already I have described as a part of my earlier lecture.

So, DSC measures the difference in the amount of heat in the form of an energy that is required to increase the temperature of the sample and the reference when they are heated and cooled at the same rate. So, both the sample and reference maintained nearly similar type of temperature throughout the experiment. So, it is a measurement of basically the flow of heat.

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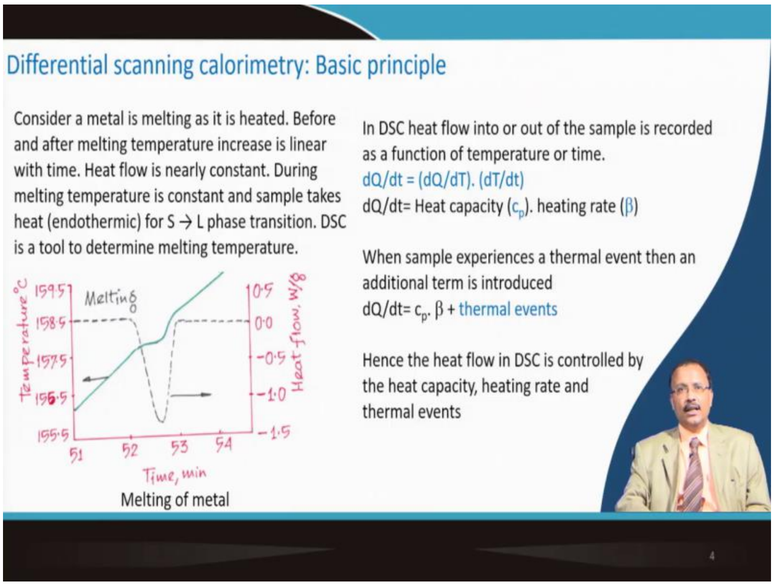

Differential scanning calorimetry: Basic principle

Consider a metal is melting as it is heated. Before and after melting temperature increase is linear with time. Heat flow is nearly constant. During melting temperature is constant and sample takes heat (endothermic) for S → L phase transition. DSC is a tool to determine melting temperature.

Melting of metal

In DSC heat flow into or out of the sample is recorded as a function of temperature or time.

dQ/dt = (dQ/dT). (dT/dt)

dQ/dt= Heat capacity (c_p). heating rate (β)

When sample experiences a thermal event then an additional term is introduced

dQ/dt= c_p. β + thermal events

Hence the heat flow in DSC is controlled by the heat capacity, heating rate and thermal events

So, if you consider a metal is melting as it is heated so, before and after the melting transition solid to liquid transition it increases the temperature if you see it is linear linearly varies with time whenever the melting takes place the temperature remains almost constant then again it increases and follow this.

So, during melting the temperature is constant and the sample takes heat which is endothermic for this solid to liquid phase transition. So, DSC is a tool to determine the melting temperature. So, in case of DSC the heat flows into either into the sample or out of the sample.

So, that heat flow is recorded as a function of temperature or time. So, you are measuring dQ by dt. So, you can write dQ by d capital T as a function of temperature and the rate of temperature dT by dt. So, dQ by dt that is heat capacity and heating rate is given by d capital T by d small t.

So, this relation is valid in a normal case and when sample experiences say any kind of thermal event an additional term is introduced. So, it will be c p and heating rate plus that thermal event that could be melting that could be glass transition or various other thermal event. Since the heat flow in DSC is controlled by the heat capacity, it is controlled by the heating rate and also the thermal event that is taking place.

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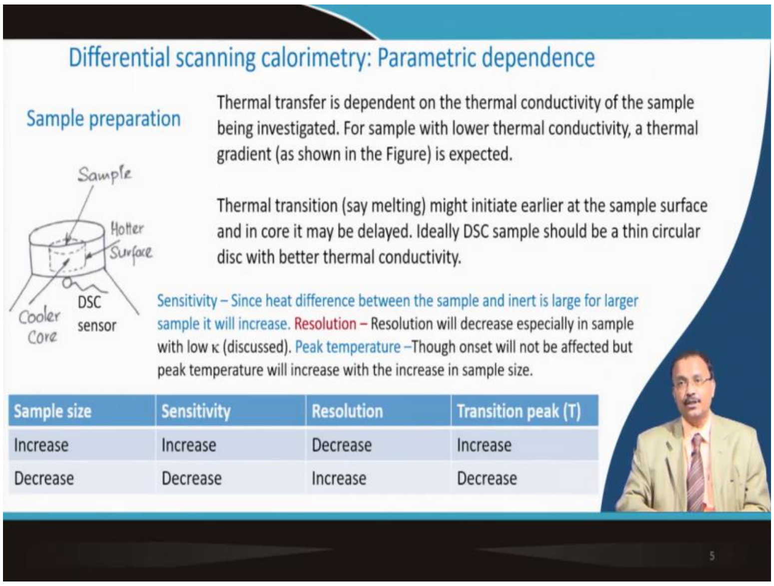

Differential scanning calorimetry: Parametric dependence

Sample preparation

Thermal transfer is dependent on the thermal conductivity of the sample being investigated. For sample with lower thermal conductivity, a thermal gradient (as shown in the Figure) is expected.

Thermal transition (say melting) might initiate earlier at the sample surface and in core it may be delayed. Ideally DSC sample should be a thin circular disc with better thermal conductivity.

Sensitivity – Since heat difference between the sample and inert is large for larger sample it will increase. Resolution – Resolution will decrease especially in sample with low κ (discussed). Peak temperature –Though onset will not be affected but peak temperature will increase with the increase in sample size.

Sample size	Sensitivity	Resolution	Transition peak (T)
Increase	Increase	Decrease	Increase
Decrease	Decrease	Increase	Decrease

So, sample preparation is important. So, thermal transfer is dependent on the thermal conductivity of the sample being investigated. So, they use a small pan where the sample is put, so, if the sample is having lower thermal conductivity a thermal gradient of course, will be there. So, the surface which will face the furnace part that will be hot and this will be in the cooler region.

So, in case of a thermal transition say melting that can initiate earlier at the sample surface because the temperature is high and it is less thermal thermally conductive and core may not be seeing the transition. So, ideally DSC sample should be a thin circular disc not a massive one, so that this thermal gradient is avoided.

Now, you can think of that the parameter like sensitivity, resolution of the measurement, transition peak etcetera how they are dependent on the sample size. So, first let us talk about the sensitivity. So, the heat difference between the sample and inert is large for larger sample. So, of course, it will increase. So, sample size if it is increases, the sensitivity will increase.

But, at the same time resolution will decrease in the sample because particularly if it is having lower thermal conductivity then resolution suddenly will decrease because this part will not get melt melted as compared to the surface having a large sample size. Peak temperature onset will not be affected, but the peak temperature will increase with the increase in sample size because it will take time to melt the whole of it.

So, for larger sample size sensitivity will increase resolution will decrease and transition peak will increase to a higher temperature and same thing will happen if you decrease it then sensitivity will also decrease resolution will increase and for a decrease sample size your transition peak temperature will be correctly predicted.

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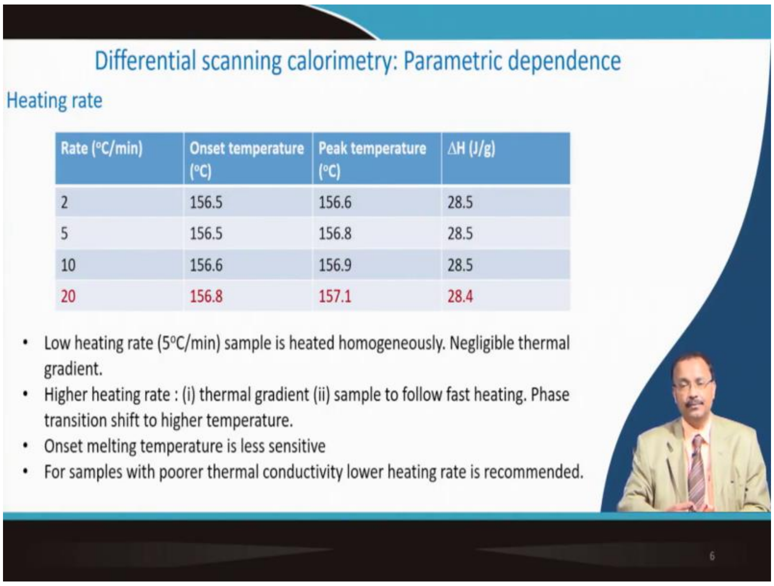

Differential scanning calorimetry: Parametric dependence

Heating rate

Rate (°C/min)	Onset temperature (°C)	Peak temperature (°C)	ΔH (J/g)
2	156.5	156.6	28.5
5	156.5	156.8	28.5
10	156.6	156.9	28.5
20	156.8	157.1	28.4

- Low heating rate (5°C/min) sample is heated homogeneously. Negligible thermal gradient.
- Higher heating rate : (i) thermal gradient (ii) sample to follow fast heating. Phase transition shift to higher temperature.
- Onset melting temperature is less sensitive
- For samples with poorer thermal conductivity lower heating rate is recommended.

Heating rate is also affect the correctness of the measurement. So, lower heating rate is about 5 degree Celsius per minute. So, sample is heated homogeneously and there is negligible thermal gradient in case of heating rate if it is high then there is a thermal gradient. So, sample to follow the first heating rate is problematic.

So, the phase transition is usually shift to high temperature onset of melting will be less affected for the sample which are poorer in thermal conductivity having poor thermal conductivity, lower heating rate is always recommended. So, it is a typical example for a particular metal and this metal indium is used as one of the standard for DSC calibration.

You can see the heating rate is increase 2, 5, 10 and 20 onset temperature it remains more or less constant peak temperature it varies. You see that almost with the heating rate there is a one degree difference it takes place and the heat flow they are normally not very that much.

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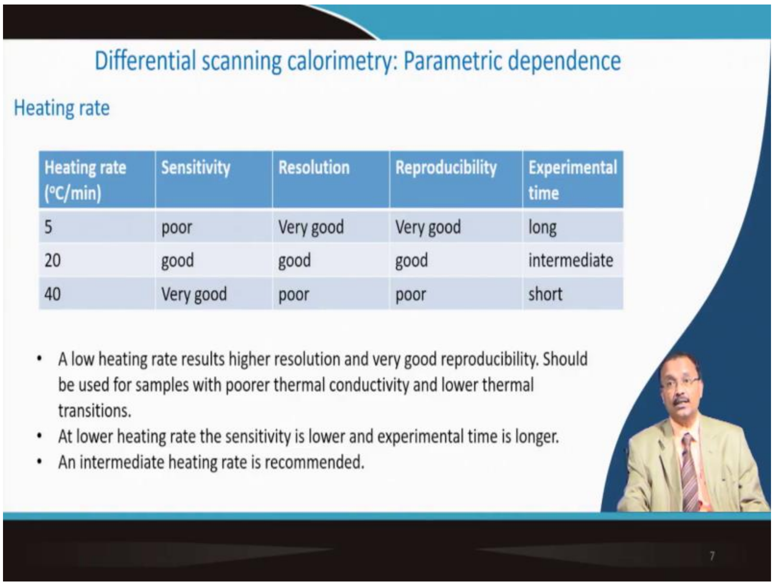

Differential scanning calorimetry: Parametric dependence

Heating rate

Heating rate (°C/min)	Sensitivity	Resolution	Reproducibility	Experimental time
5	poor	Very good	Very good	long
20	good	good	good	intermediate
40	Very good	poor	poor	short

- A low heating rate results higher resolution and very good reproducibility. Should be used for samples with poorer thermal conductivity and lower thermal transitions.
- At lower heating rate the sensitivity is lower and experimental time is longer.
- An intermediate heating rate is recommended.

So, a lower heating rate results a higher resolution and very good reproducibility and this should be used for samples which are poor thermal conductivity and lower thermal transition at lower heating rate the sensitivity as you can see is low and the experimental time of course, is longer, it takes long time to complete the experiment.

So, usually a intermediate heating is preferred which is typically 20 degree Celsius per minute which gives you a reasonable good sensitivity, resolution is also good experimental time requirement is not that high. So, depending on the type of sample you may have to choose the right kind of heating rate.

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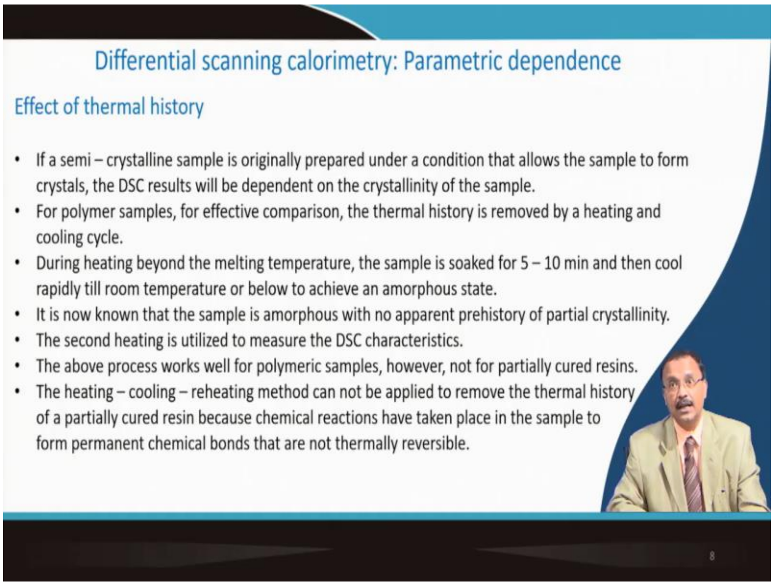

Differential scanning calorimetry: Parametric dependence

Effect of thermal history

- If a semi – crystalline sample is originally prepared under a condition that allows the sample to form crystals, the DSC results will be dependent on the crystallinity of the sample.
- For polymer samples, for effective comparison, the thermal history is removed by a heating and cooling cycle.
- During heating beyond the melting temperature, the sample is soaked for 5 – 10 min and then cool rapidly till room temperature or below to achieve an amorphous state.
- It is now known that the sample is amorphous with no apparent prehistory of partial crystallinity.
- The second heating is utilized to measure the DSC characteristics.
- The above process works well for polymeric samples, however, not for partially cured resins.
- The heating – cooling – reheating method can not be applied to remove the thermal history of a partially cured resin because chemical reactions have taken place in the sample to form permanent chemical bonds that are not thermally reversible.

Now, in case of DSC the thermal history is important particularly, in case of a semi crystalline sample which is prepared originally under various other condition. So, the crystallinity may be different, it may have different types of crystals. So, the DSC that will be dependent on the initial crystallinity of that polymer sample if it is a semi-crystalline polymer.

So, for effective comparison the thermal history should be removed. So, initially the sample is heated and then again pulled back. So, a heating and cooling cycle is required. So, usually heating is done beyond the melting temperature and then the sample is soaked for 5 to 10 minutes and then cool rapidly at room temperature, so that it achieve a amorphous state.

So, it is now known that the sample is in originally an amorphous state and it does not have any prehistory for partial crystallinity. Then you do the second heating and measured the DSC characteristics. So, for polymeric sample it works, but not for all samples this criteria will work. For example, if you take a partially cured resin.

So, heating and cooling and again reheating method that cannot be applied here to remove the thermal history because it is partially cured a chemical reaction has been taking place in the sample to form a permanent chemical change and that is not thermally reversible. So, depending on the sample the thermal history is important for you to know before performing a DSC measurement.

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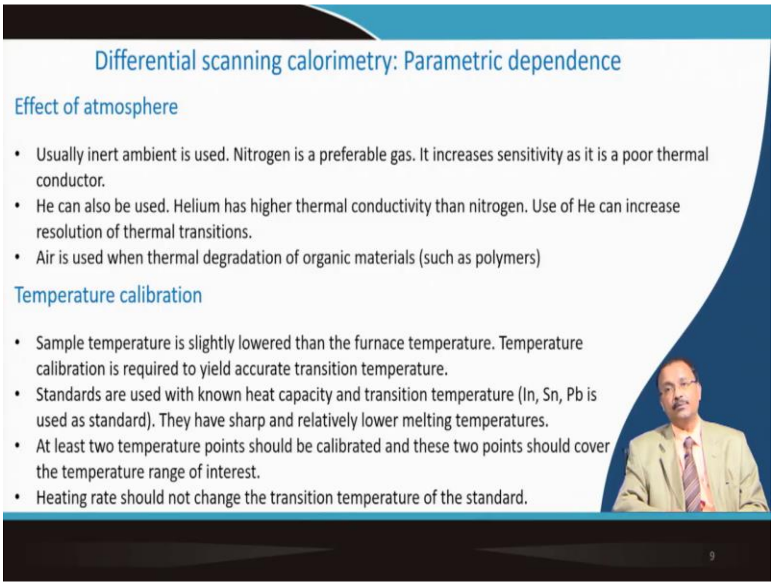

Differential scanning calorimetry: Parametric dependence

Effect of atmosphere

- Usually inert ambient is used. Nitrogen is a preferable gas. It increases sensitivity as it is a poor thermal conductor.
- He can also be used. Helium has higher thermal conductivity than nitrogen. Use of He can increase resolution of thermal transitions.
- Air is used when thermal degradation of organic materials (such as polymers)

Temperature calibration

- Sample temperature is slightly lowered than the furnace temperature. Temperature calibration is required to yield accurate transition temperature.
- Standards are used with known heat capacity and transition temperature (In, Sn, Pb is used as standard). They have sharp and relatively lower melting temperatures.
- At least two temperature points should be calibrated and these two points should cover the temperature range of interest.
- Heating rate should not change the transition temperature of the standard.

Usually inert ambient is used in case of DSC. Nitrogen is a preferable gas because it increases the sensitivity and it is a poor thermal conductor. Sometimes helium also can be used because helium is having a higher thermal conductivity than nitrogen, but helium is expensive, but it increases the resolution of the thermal transition very accurately thermal transition you can achieve by using helium.

When decomposition reaction is taking place, then one can use air when you want to know the thermal degradation of organic materials then the air ambient is used. Temperature calibration is required. Sample temperature as you can understand that is slightly lowered than the furnace temperature.

So, for accurate measurement of any transition temperature you need to have a temperature calibration and for this purpose the standard are used which has very well known heat capacity and transition temperature. Usually metals are used indium tin lead that is used as standard they have a very sharp. In fact, I have shown you the melting transition of indium metal is very sharp temperature melting temperature is there at relatively low melting temperature, low temperature it melts.

So, at least in the whole temperature regime where the experiment is done at least two temperature point should be calibrated to know exactly the temperature of the equipment what instrument whatever it is the whatever it is showing it is correct.

And, heating rate should not change the transition temperature of the standard. So, that you have seen that even if you change the heating rate transition temperature of a standard metal indium that do not change much.

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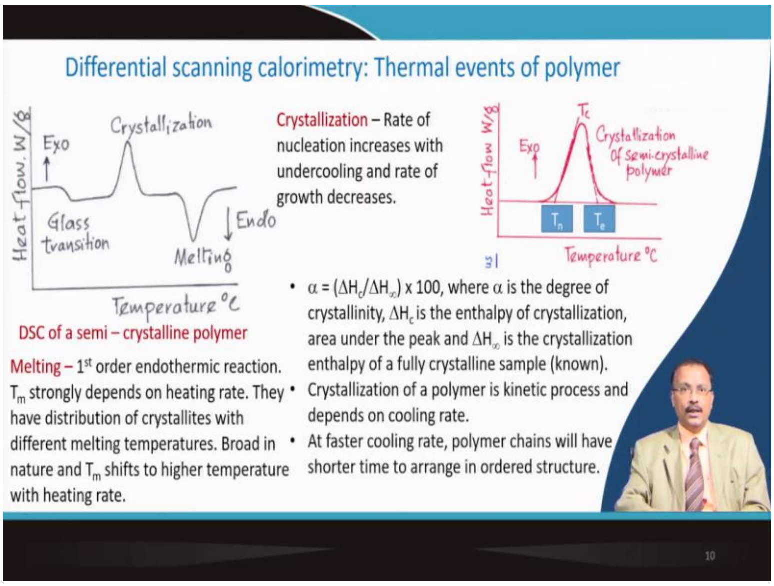

Differential scanning calorimetry: Thermal events of polymer

DSC of a semi – crystalline polymer

Melting – 1st order endothermic reaction. T_m strongly depends on heating rate. They have distribution of crystallites with different melting temperatures. Broad in nature and T_m shifts to higher temperature with heating rate.

Crystallization – Rate of nucleation increases with undercooling and rate of growth decreases.

- $\alpha = (\Delta H_c/\Delta H_\infty) \times 100$, where α is the degree of crystallinity, ΔH_c is the enthalpy of crystallization, area under the peak and ΔH_∞ is the crystallization enthalpy of a fully crystalline sample (known).
- Crystallization of a polymer is kinetic process and depends on cooling rate.
- At faster cooling rate, polymer chains will have shorter time to arrange in ordered structure.

So, here we can show one example a thermal event of a polymer. So, as you can see melting is an endothermic reaction and this is a first order transition and this melting point that strongly depends on the heating rate because they have distribution of the crystallite in the polymer sample and each of them have different melting temperature. So, usually this transition in broad in nature and with heating rate the melting point shifts to higher temperature with the increase of the heating rate.

Crystallization as you know that the crystallization that basically will depend on the nucleation and from the melting if you cool it down it depends on the under cooling under cooling this crystallization will be dependent on.

So, we can define the value of alpha which is the degree of crystallinity and you can measure the value of del H c which is the enthalpy of crystallization and which is nothing, but the area under the peak and if you know the crystallization enthalpy for a fully crystalline sample, then one can calculate the value of alpha.

And, crystallization of a polymer is a kinetic process and suddenly it will depend on the cooling rate faster cooling rate usually the polymer chain will have shorter time to arrange in a ordered structure. So, it will be amorphous in nature.

So, typically from the exothermic peak corresponding to this crystallization this onset temperature is the nucleation temperature and this temperature if you have a tangent here and this is your end temperature. So, this will be determined by both melting and crystallization of a semi-crystalline polymer that can be identified by a DSC measurement.

Apart from that you have a small deviation which is the indication of a glass transition temperature.

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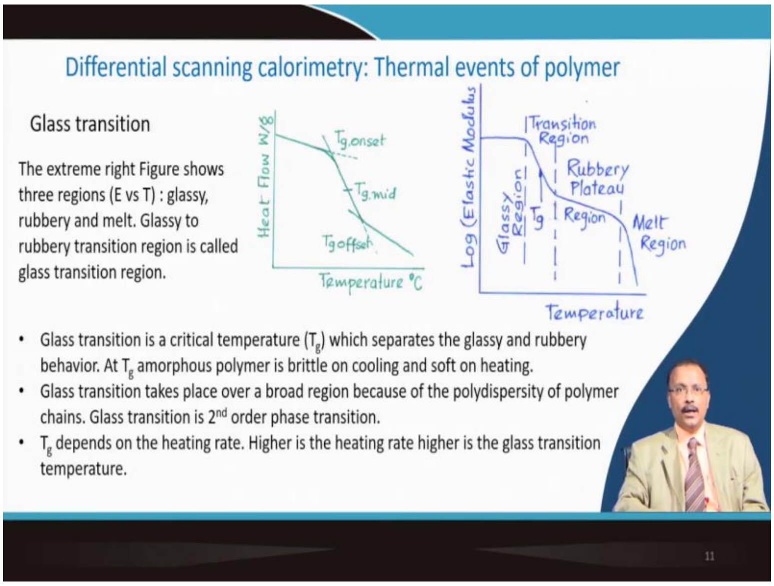

Differential scanning calorimetry: Thermal events of polymer

Glass transition

The extreme right Figure shows three regions (E vs T) : glassy, rubbery and melt. Glassy to rubbery transition region is called glass transition region.

- Glass transition is a critical temperature (T_g) which separates the glassy and rubbery behavior. At T_g amorphous polymer is brittle on cooling and soft on heating.
- Glass transition takes place over a broad region because of the polydispersity of polymer chains. Glass transition is 2nd order phase transition.
- T_g depends on the heating rate. Higher is the heating rate higher is the glass transition temperature.

So, the figure here it shows the glass transition is eventually if you consider the elastic modulus versus temperature plot of a polymer, then initially this is a glassy region where elastic modulus remain constant and then there is a glass to rubbery region, this is called a glass transition region. It is having a plateau also and followed by this melting transition.

So, usually the elastic modulus changes when the polymer is heated and you can also get the same information by measuring its elastic modulus. So, glass transition is a critical temperature which separates the glassy region with rubbery behavior. So, at T_g

amorphous polymer they becomes brittle on cooling and soft on heating. So, if you go to the this side, then it is brittle in nature you go to this side; then it is a rubbery region.

So, glass transition usually takes place over a broad region because mainly of the polydispersity of polymer chain and it is a second order transition and T g actually depends on the heating rate if the heating rate is increase, then the glass transition temperature is also increased.

So, this is in DSC it is just a small kind of variation like this. So, onset temperature can be identified and also this temperature can be identified. So, in between in fact, if you differentiate this plot this in between temperature that is taken as exact T_g of the polymer that is being experimented.

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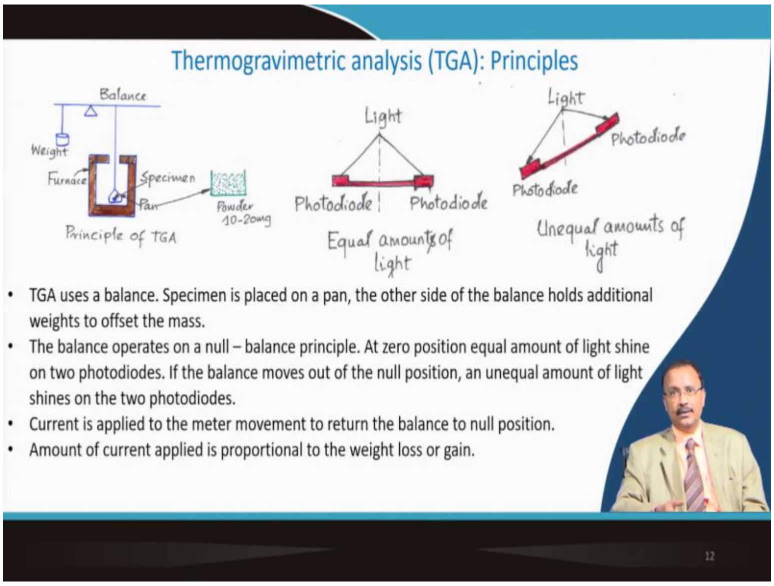

Thermogravimetric analysis (TGA): Principles

- TGA uses a balance. Specimen is placed on a pan, the other side of the balance holds additional weights to offset the mass.
- The balance operates on a null – balance principle. At zero position equal amount of light shine on two photodiodes. If the balance moves out of the null position, an unequal amount of light shines on the two photodiodes.
- Current is applied to the meter movement to return the balance to null position.
- Amount of current applied is proportional to the weight loss or gain.

TGA thermogravimetric analysis that uses a balance, so, specimen is placed on a pan the other side of the balance holds additional weight to offset the mass change because of the reaction that might have taken place in the sample during heating the.

So, balance operates in a null balance principle. A zero position equal amount of light shines on two photodiodes and if the balance move out of the null position and unequal amount of light shines this two photodiode. So, a current is applied to the meter. So, that the movement return to the balance to the null position.

So, amount of this applied current is in fact, proportional to the weight loss and weight gain. So, that is the principle that is followed in commercial thermogravimetric instrument.

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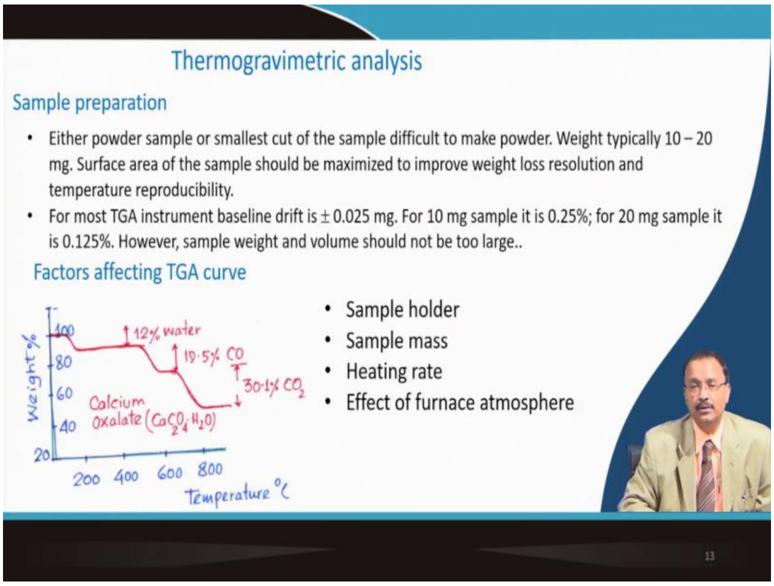

Thermogravimetric analysis

Sample preparation

- Either powder sample or smallest cut of the sample difficult to make powder. Weight typically 10 – 20 mg. Surface area of the sample should be maximized to improve weight loss resolution and temperature reproducibility.
- For most TGA instrument baseline drift is ± 0.025 mg. For 10 mg sample it is 0.25%; for 20 mg sample it is 0.125%. However, sample weight and volume should not be too large..

Factors affecting TGA curve

- Sample holder
- Sample mass
- Heating rate
- Effect of furnace atmosphere

So, sample preparation either you can use a powder sample or a very smallest cut sample if it is difficult to make a powder. Weight is typically taken 10 to 20 milligram. The surface area should be as high as possible. It should be maximized to improve the weight loss resolution.

So, for most of this TGA you have a baseline drift which is typically plus minus 0.025 milligram. So, if you have a 10 milligram sample it is 0.25 percent; if you increase it 20 milligram sample this is 0.125 percent the. So, larger sample will help in terms of the baseline, but the sample weight and volume should not be too large.

So, the factors that affect a typical TG curve which is shown here which is a weight loss as a function of temperature. So, sample holder is important, sample mass is important, heating rate is important and also the sample furnace temperature is important.

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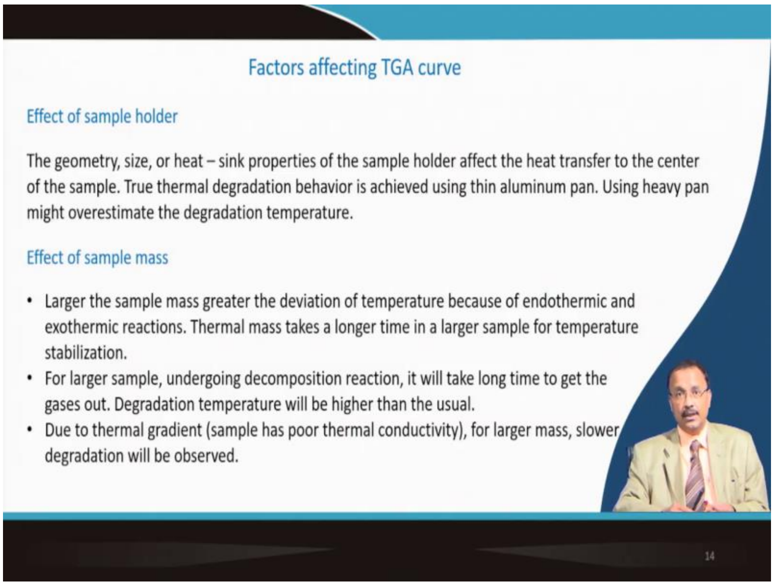

Factors affecting TGA curve

Effect of sample holder

The geometry, size, or heat – sink properties of the sample holder affect the heat transfer to the center of the sample. True thermal degradation behavior is achieved using thin aluminum pan. Using heavy pan might overestimate the degradation temperature.

Effect of sample mass

- Larger the sample mass greater the deviation of temperature because of endothermic and exothermic reactions. Thermal mass takes a longer time in a larger sample for temperature stabilization.
- For larger sample, undergoing decomposition reaction, it will take long time to get the gases out. Degradation temperature will be higher than the usual.
- Due to thermal gradient (sample has poor thermal conductivity), for larger mass, slower degradation will be observed.

So, the geometry and size heat sink properties of the sample holder affects the heat transfer to the center of the sample where inside the crucible. So, true thermal degradation behavior is achieved using a very thin aluminum pan using a heavy pan that might overestimate the degradation temperature.

Sample mass, as I told that larger the sample mass greater is the deviation of temperature because of the endothermic or exothermic reaction taking place. So, the deviation of temperature will be more. So, thermal mass actually takes a long time for a larger sample for temperature stabilization.

If you take a large sample weight that is undergoing a decomposition reaction, so it will take long time for the gases to come out of the sample. So, degradation temperature in fact, they will be estimated higher because the weight loss will simultaneously be taking place. So, it will be higher in case of larger sample.

And, of course, during thermal gradient due to the thermal gradient sample particularly those samples which are having poor thermal conductivity, for larger mass slower degradation will be observed.

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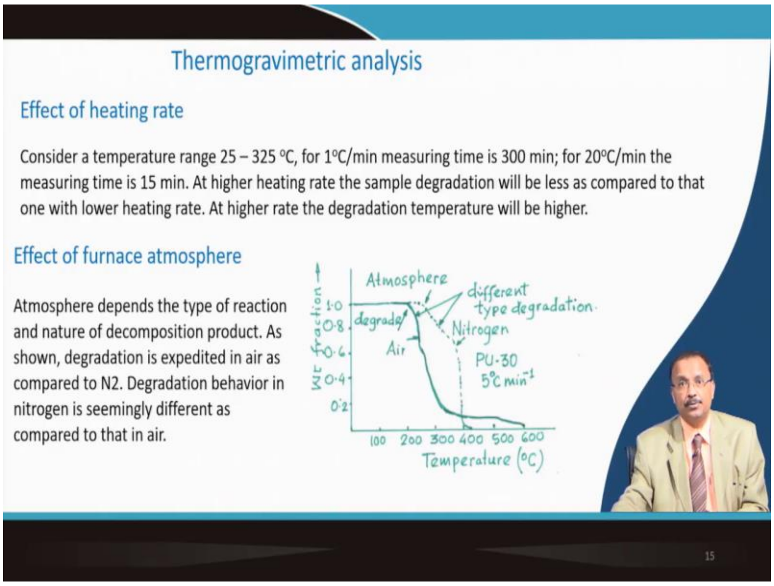

Thermogravimetric analysis

Effect of heating rate

Consider a temperature range 25 – 325 °C, for 1°C/min measuring time is 300 min; for 20°C/min the measuring time is 15 min. At higher heating rate the sample degradation will be less as compared to that one with lower heating rate. At higher rate the degradation temperature will be higher.

Effect of furnace atmosphere

Atmosphere depends the type of reaction and nature of decomposition product. As shown, degradation is expedited in air as compared to N2. Degradation behavior in nitrogen is seemingly different as compared to that in air.

In the temperature range if you consider 25 to 325 degree Celsius, heating rate 1 degree Celsius per minute, measuring time is 300 minute, if you change the heating rate to 20 degree Celsius per minute measuring time is only 15 minutes. So, at higher heating rate the sample degradation will be less as compared to one with slower heating rate because the degradation temperature at higher heating rate that will be estimated larger because the sample is seeing very small time the temperature change.

So, that affects the degradation temperature furnace atmosphere is also important and as you can see that the same sample when you are doing the measurement in air ambient it depends on the type of degradation in the sample. So, nature of degradation product that is important.

So, in case of air the degradation is expedite it occurs at relatively lower temperature as compared to the same in nitrogen also the degradation behavior in nitrogen is very different as compared to that of in air. So, the type of degradation it is important and therefore, the furnace and atmosphere will also have to be judiciously select.

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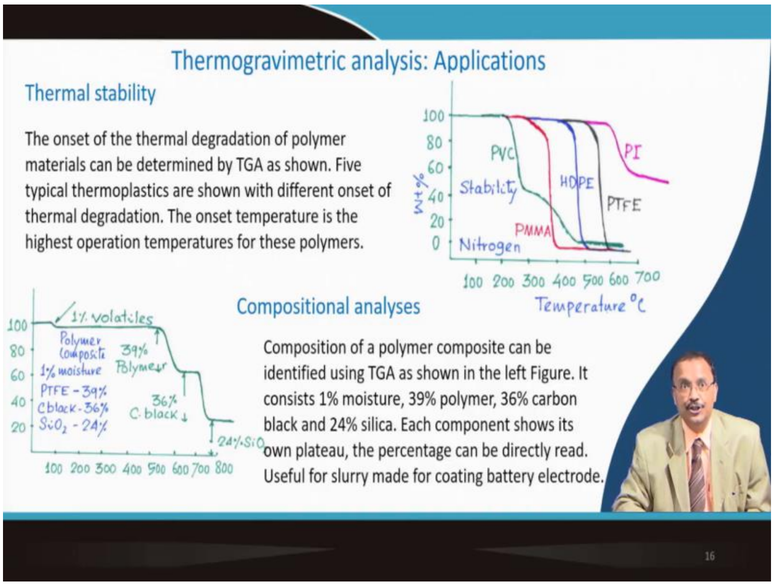

Thermogravimetric analysis: Applications

Thermal stability

The onset of the thermal degradation of polymer materials can be determined by TGA as shown. Five typical thermoplastics are shown with different onset of thermal degradation. The onset temperature is the highest operation temperatures for these polymers.

Compositional analyses

Composition of a polymer composite can be identified using TGA as shown in the left Figure. It consists 1% moisture, 39% polymer, 36% carbon black and 24% silica. Each component shows its own plateau, the percentage can be directly read. Useful for slurry made for coating battery electrode.

Thermal stability can be determined by TGA analysis the onset for the thermal degradation of the polymer material that can be determined by TGA as you can see here for PVC, for PMMA, HDPE, PTFE and PI progressively the thermal degradation temperature you can see the onset of the weight loss that is taking place it is done in nitrogen ambient. So, this is a good way to check the thermal stability of the sample.

Another thing what is done by TGA is composition analysis. So, here typically we have say mixed silicon dioxide with carbon black with a polymer and it is absorbing about 1 percent of moisture and do the thermogravimetric analysis you get the weight loss. And, eventually from the weight loss you can get the composition of the mixture.

So, it can be seen that it consist of 1 percent of moisture, about 39 percent polymer it decomposes, then 36 percent of carbon and whatever is remaining about 24 percent that is the silica. So, each component they usually show its own plateau and the percentage can be directly read.

So, this is very useful for the slurry preparation for example, for battery rechargeable battery we prepared the slurry to coat the electrode material current collectors. So, the composition of the slurry that can be identified by thermogravimetric analysis.

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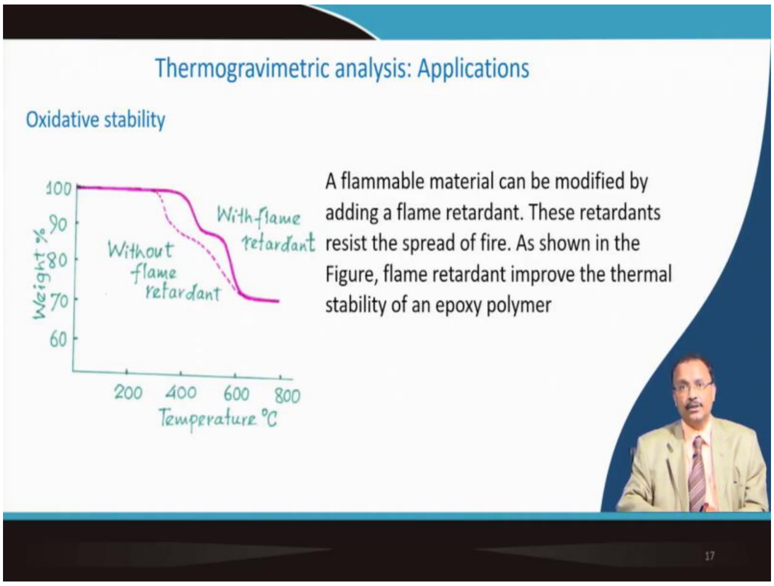
Thermogravimetric analysis: Applications

Oxidative stability

A flammable material can be modified by adding a flame retardant. These retardants resist the spread of fire. As shown in the Figure, flame retardant improve the thermal stability of an epoxy polymer

Oxidative stability also can be identified by the TGA. So, here is one example a polymer with a flame retardant and without a flame retardant. So, a flammable material can be modified by adding a flame retardant. So, this retardant resists the spread of the fire.

So, this figure shows the flame retardant improve the thermal stability of an epoxy polymer because its degradation temperature is expedite is moved to high temperature about 150 degree Celsius. So, that is the usefulness of thermogravimetric analysis.

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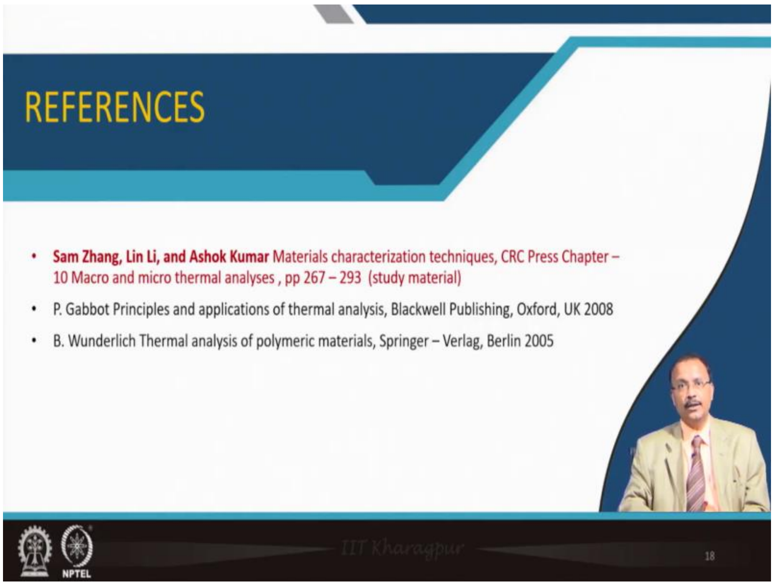
REFERENCES

- **Sam Zhang, Lin Li, and Ashok Kumar** Materials characterization techniques, CRC Press Chapter – 10 Macro and micro thermal analyses , pp 267 – 293 (study material)
- P. Gabbot Principles and applications of thermal analysis, Blackwell Publishing, Oxford, UK 2008
- B. Wunderlich Thermal analysis of polymeric materials, Springer – Verlag, Berlin 2005

So, this part the study material is from the book by Zhang Li and Kumar, Chapter number 10, Macro and micro thermal analysis and apart from that the book by Gabbot and Wunderlich that is also helpful for clarifying some of the idea taught in this lecture.

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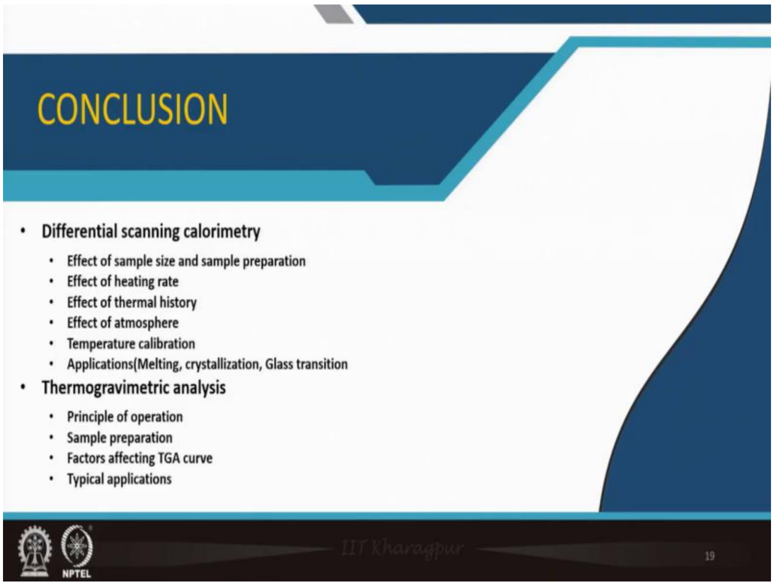

CONCLUSION

- Differential scanning calorimetry
  - Effect of sample size and sample preparation
  - Effect of heating rate
  - Effect of thermal history
  - Effect of atmosphere
  - Temperature calibration
  - Applications(Melting, crystallization, Glass transition
- Thermogravimetric analysis
  - Principle of operation
  - Sample preparation
  - Factors affecting TGA curve
  - Typical applications

So, in this lecture we have mainly focused on differential scaling calorimetry, talked about the effect of sample size and sample preparation, effect of heating rate, thermal history, effect of atmosphere temperature calibration and finally, how and what are the stages and how this DSC can be used as a material characterization tool.

Next we took thermogravimetric analysis – first we identified the principle of operation, then sample preparation, factors affecting the thermogravimetric curve and then we cited some typical applications.

Thank you for your attention.