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DIELECTRIC DETERMINATION OF THE GLASS TRANSITION TEMPERATURE (T_g)

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OBJECTIVE

To determine the glass transition temperature of a polymer using a dielectric dissipation technique.

EQUIPMENT

Polymer specimen, aluminum foil electrodes, optically flat pyrex discs, brass weight, thermocouple, thermal joint compound, furnace, minicomputer, plotter, LCR meter (inductance, capacitance, resistance meter)

BACKGROUND

The "glass transition" occurs in materials which can be cooled from a liquid phase to a solid phase without crystallizing. As a viscous liquid is cooled, its viscosity increases with decreasing temperature. The point at which the viscosity becomes so large that the molecules are rigidly fixed in place, although no crystallization has occurred, is considered to be the glass transition temperature (T_g). Thus, below T_g the molecules are "frozen" in place, whereas above T_g the molecules are relatively free to move. A large variety of materials exhibiting a glass transition exist, including organic polymers, inorganic oxides, molecular liquids, fused salts and some metallic systems [1].

The glass transition between the solid phase and the liquid phase results in changes in a variety of properties of the material. In this experiment, the change in the dielectric dissipation factor of the material will be measured in order to determine T_g.

The dielectric dissipation factor is a measure of how much energy is dissipated when an alternating electric field is applied across a capacitor. Although the dissipation factor has several components, the contribution from the dipolar molecules in the material is the piece which varies significantly with temperature.

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*This experiment was developed at NASA Langley Research Center. The author worked with this equipment under NASA Cooperative Agreement NCC1-90.

In a dielectric material below T_g, the molecules are "frozen" in place, cannot respond to the applied field, and therefore do not contribute to the dissipation of energy. Consequently, the dissipation factor is relatively constant for all temperatures below T_g.

As the temperature is increased above T_g, however, the viscosity decreases, so the molecules may gradually begin to move in response to the applied field. Since the molecules are not completely free to move at this point, they will lag somewhat behind the applied field, resulting in the dissipation of energy. Therefore, the dissipation factor will increase significantly at T_g.

At temperatures well above T_g, the viscosity will decrease so much that the molecules will be free enough to move in phase with the electric field. When the molecules are in step with the electric field, energy will not be dissipated by this means, so the dissipation factor will decrease.

SUMMARY

In summary, a peak in the dissipation factor versus temperature curve is expected near the glass transition temperature T_g, as shown in Figure 1. It should be noted that the glass transition is gradual rather than abrupt, so that the glass transition temperature T_g is not clearly identifiable. In this case, the glass transition temperature is defined to be the temperature at the intersection point of the tangent lines to the dissipation factor versus temperature curve above and below the transition region, as illustrated in Figure 1.

In this experiment, the dielectric dissipation factor technique will be applied to polymers.

PROCEDURE

1) Stack the apparatus as shown in Figure 2.

   Typically, the specimen will be 5.5 cm in diameter and the other components are 5.0 cm in diameter.

   The electrodes may be cut from aluminum foil, with "tails" attached for leads to the LCR meter. Be sure that they are not wrinkled.

   The disks and brass weight are present to insure uniform pressure at the electrode-specimen interface.

   Thermal joint compound is used at the tip of the thermocouple to provide contact with the quartz disk.

2) Place the assembly in the furnace. Connect the leads to the LCR meter, and begin data collection. (The total temperature range and temperature intervals for data collection will depend upon the sample you are using.)

CAUTION! DO NOT BURN YOURSELF!

3) Analyze the data. Determine the best line fits to the curve above and below the transition region, as shown in Figure 1. The glass transition temperature is the temperature at the intersection of these two lines.

REFERENCES

1. M. Goldstein J. Chem. Phys. 51, 3728 (1969)

INSTRUCTOR'S NOTES

When this experiment was performed at NASA Langley Research Center, the data acquisition system was controlled by an HP-9830A computer. The data acquisition program reads data from the thermocouple to determine the specimen temperature, and then reads the dissipation factor measurements from an HP-4275A multifrequency LCR meter at temperature intervals and frequencies specified by the operator. The furnace was adjusted to provide a heating rate of 6 ^{0}C/min. It was found that this heating rate and a frequency of 10 kHz yields Tg measurements which are in agreement with measurements obtained from other techniques.

The data was analyzed using a linear regression program to determine the best line fits. The computer was then used to calculate the temperature at the intersection of the two lines.

If a computer is not available to control the experiment, the student could record the temperature and dissipation factor readings manually and perform the data analysis by hand. Variations of only two or three degrees are typically introduced when the data is analyzed manually.

ADDITIONAL NOTES

As discussed previously, the glass transition temperature occurs when the molecules become free to move in the material. Thus, Tg is dependent upon the length of the polymeric chain, and therefore upon the processing techniques used.

Measurement of the glass transition temperature is therefore useful in comparing the degree of crosslinking induced by various processing techniques. For example, irradiation of the polymer film may result in crosslinking or chain scissioning, with a corresponding increase or decrease in Tg.

Thus, this Tg experiment may be used in conjunction with the study of a variety of polymer processing techniques.

It should also be noted that some polymers which possess pendant groups may exhibit more that one transition. This occurs because the pendant group may become free to move at a temperature lower than the temperature at which the entire chain is freed.

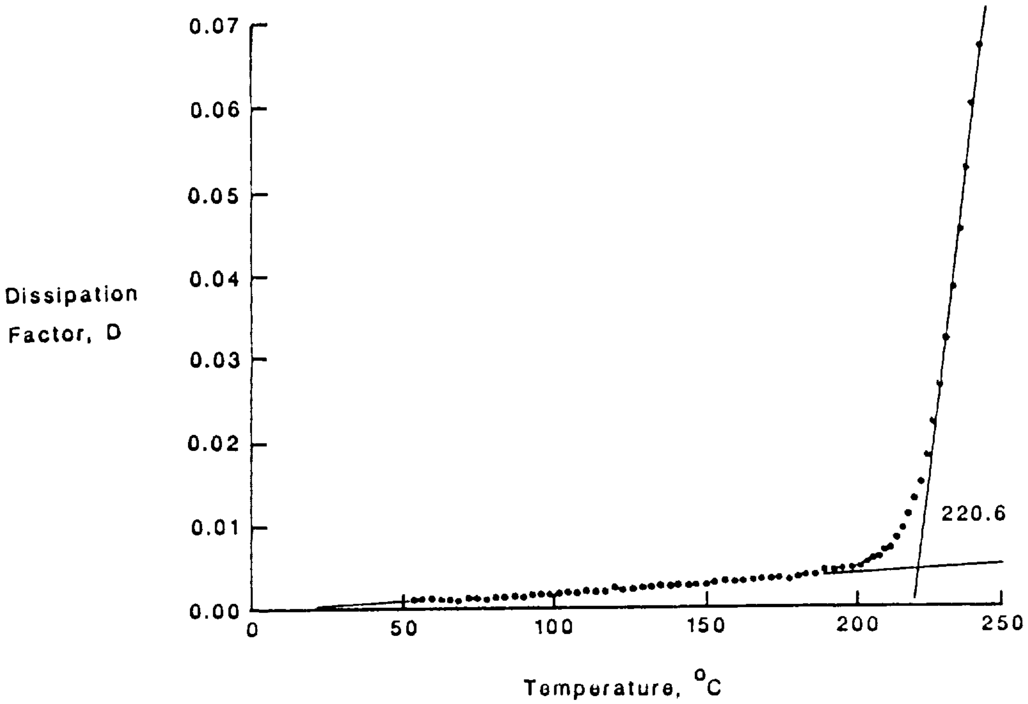

Figure 1. Determination of the glass transition temperature of a polymer from AC electrical dissipation data.

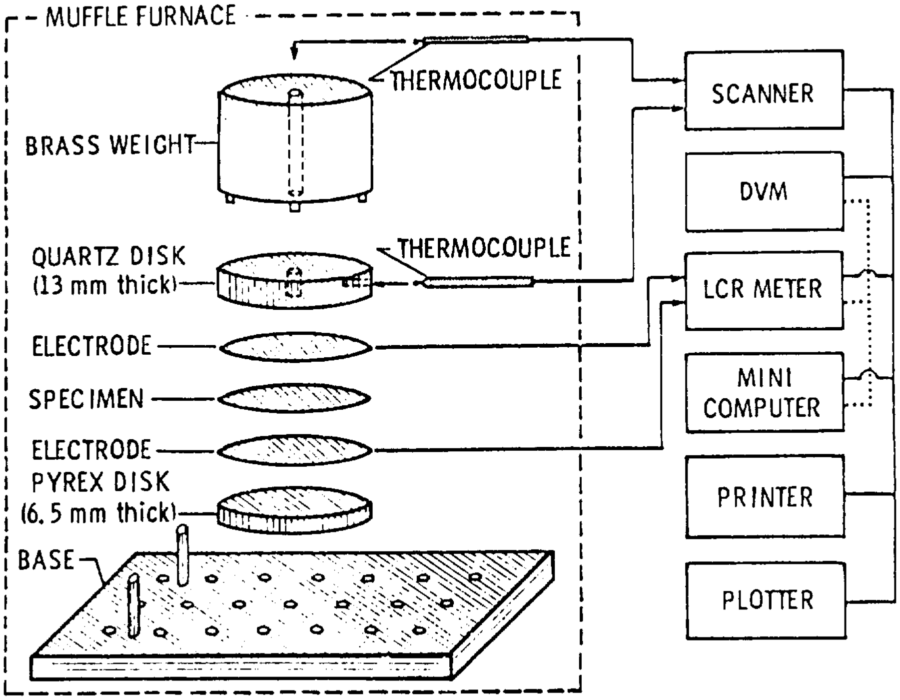

Diagram by: E. R. Long, Jr.
NASA-LRC

Figure 2. Experimental arrangement for glass transition temperature measurements.