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Study of the Molecular Stress Response
of Solid Polymers by Photoelastic Methods
(Unclassified Title)

By

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Chief Investigator

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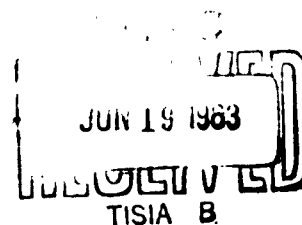
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Feltman Research and Engineering Laboratories
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The work carried out in the second quarter included a continuation of stress-optical measurements, a number of measurements of elastic modulus, and also some equipment developments of interest. The results will be described under these respective headings.

Stress-Optical Measurements

1. A second sample of methyl acrylate/methyl methacrylate copolymer (monomer ratio 50/50, byweight) was obtained from the Rohm and Haas Co. This sample, unlike the sample described in the first quarterly report, seemed satisfactorily polymerized (hardly any monomer odor could be noted), and measurements of stress-optical coefficient (SOC) of this sample as a function of temperature were begun.
2. Samples of two copolymers of ethyl acrylate/ methyl methacrylate were also obtained from Rohm and Haas; one contained 9% ethyl acrylate and the other 13%. Some measurements were made on these samples from room temperature (27°) to +100° C., and at liquid nitrogen temperature (-198° C.). The SOC values are negative throughout, and the curve is generally similar to that for PMMA, except that upward bumps (in the positive direction) are observed at about +65° for the 9% sample and at about +40° for the 13% sample. These must be due to the ethyl acrylate; the SOC of polyethyl acrylate is positive in the glassy state. But the sudden onset of this contribution is surprising. This interesting behavior deserves further study.
3. A sample of poly (methyl alpha-chlor acrylate) cast sheet was obtained from Rohm and Haas. This was actually material manufactured by General Aniline and Film Corp. under the name "Gafite". Measurements of the SOC of this material were carried out at temperatures from dry ice (-78° C.) to +100° C. (in the glassy state throughout). The curve obtained is very similar to that for PMMA, but is essentially a straight line over the whole range. This is not surprising, since the molecular structures of the two polymers are so similar; the only difference is in the presence of a chlorine atom or a methyl group in the alpha position, and most of the birefringence effect is undoubtedly due to the ester group, rather than to the alpha substituent.
4. Measurements on the series of PMMA samples of different molecular weight were continued. Samples of each of these polymers were sent back to Rohm and Haas for determination of residual monomer content; this analysis was carried out in solution, using a sensitive and accurate polarographic technique.
5. Measurements on the samples of two different molecular weights undergoing humidity treatment (at 0% and 100% R.H., exposure at room temperature) were also continued. Loss or gain of absorbed moisture is determined by weight changes of the samples. The effects of absorbed water on the SOC seem to be small.

Elastic (Young's) Modulus

It is very desirable to have data on elastic modulus to accompany stress-optical data, since this allows values of the strain-optical coefficient (ratio of birefringence to strain) to be calculated from the directly measured stress-optical coefficient (ratio of birefringence to stress).

Our equipment for measuring elastic modulus is very simple; it involves the measurement of the flexure of a beam-shaped sample which is supported near the ends and loaded at the center. The equipment is designed to fit into a large-diameter Dewar flask, so that temperatures down to liquid nitrogen can be attained, by direct immersion of the sample in the cooling medium. The equipment is very similar to that described by Rudd and Gurnee [J. Appl Phys. 28, 1096 (1957)], except that the deflection is measured by a mirror-and-scale arrangement viewed through a telescope (optical lever). This equipment was accurately calibrated for absolute deflection by use of a stack of Johansson blocks in the position where the center of the sample would normally be.

The modulus measurements carried out here were measurements at room temperature (27°C.) of:

- (1) PMMA molecular weight series
- (2) PMMA humidity treatment samples

Some scatter was encountered with these modulus data, just as with the SOC data. It will therefore apparently be necessary to make a series of duplicate measurements and take an average in order to obtain the most accurate values in any particular case.

Equipment

1. It was noted in the report for the first quarter that some difficulties were being experienced with the insulated chamber unit used for making SOC measurements at low temperatures. This has been modified for more convenient use, including the construction of a connection which allows the use of a Tenney control unit (containing dry ice) to circulate cold air through the metal jacket surrounding the sample. This should operate from room temperature down to perhaps -50°C. over a continuous range; available temperatures will therefore no longer be restricted to the freezing points of liquids as fixed points, in this range.

An improved technique for use of the freezing pentane fixed point (-132°C.) has been developed. Some possible effects of acetone vapor have been noticed in the use of an acetone-dry ice mixture; therefore future measurements at -78°C. will be carried out with crushed dry ice alone in the sample jacket, omitting the acetone.

2. From the small effects of stereospecific structure on the SOC of PMMA noted in the first quarter report, and because it would be desirable in general to be able to measure small birefringence effects with high accuracy, it was decided to construct a high precision electronic instrument

for the measurement of birefringence which would give higher accuracy than the visual method normally used.

The visual method ordinarily used is an arrangement called a Sénarmont Compensator, in which measurements are made by rotating a polarizer to the position of minimum light transmission. The high-precision instrument will be simply an electronic version of the same device, with a photo-cell in place of the eye. A Faraday cell (solenoid, with some transparent material in the core) with A.C. current passing through it, will be placed before the rotating polarizer. This wobbles the plane of polarization of the light passing through its axis and gives an output signal to the photo-cell which can be used to set the extinction point with extremely high precision. A rod of annealed high-grade optical glass was obtained for the core material, but the residual birefringence in the glass was still too large to allow it to be used for this purpose. A tube containing water, with annealed glass end plates, will be used instead.

This instrument will require rotation angles to be measured with extremely high precision. Special rotating mounts are being constructed, using transit heads with vernier scales as the rotating element. In addition, the rotators will be equipped with a long arm which can be rotated with a micrometer screw out at the end, to give very fine control. Monochromatic mercury green light (5461A) will be used in this instrument, as in the visual method. By use of suitable electronic circuitry, read-out can either be on an oscilloscope screen or a vacuum-tube voltmeter.

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