

Fine Structure of Acetal Resins and Its Effect on Mechanical Properties

C. F. HAMMER, T. A. KOCH, and J. F. WHITNEY

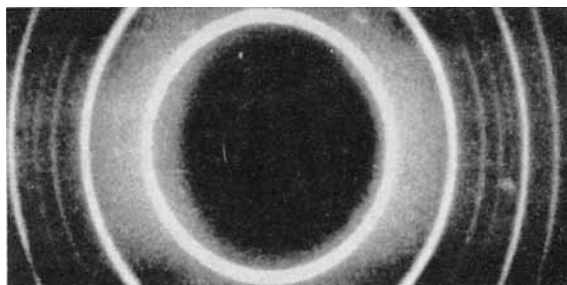
*Polychemicals Department, E. I. du Pont de Nemours and Company, Inc.,
Wilmington, Delaware*

INTRODUCTION

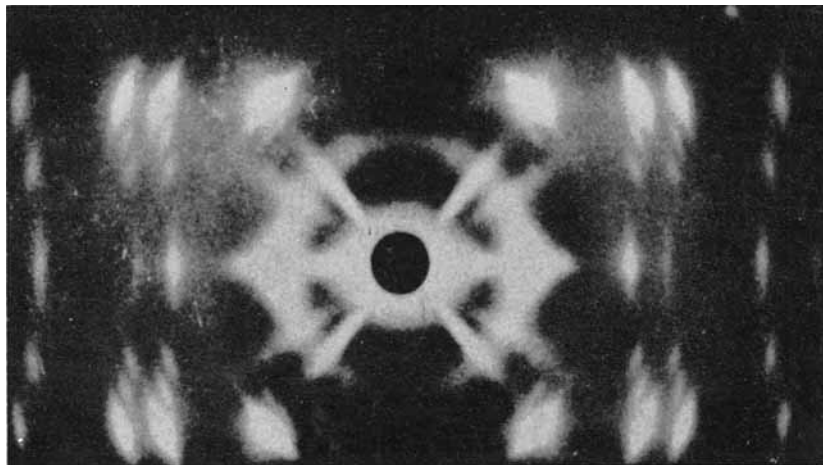
The crystalline linear polymers of formaldehyde known as polyoxymethylenes have been studied for many years. The older methods of polymerization yield polymers that are unstable. They degrade rapidly to the monomer when heated. For this reason these products are known mainly for their use in the preparation of formaldehyde solutions. The polyoxymethylenes that are the principal subject of this discussion are stable, high molecular weight resins and can be molded and extruded without serious degradation into products with excellent physical properties.¹⁻³

These resins, which may be described as containing acetal backbone linkages, are characterized by unusually high crystallinity. This results in very sharp x-ray diffraction patterns, as shown in Figure 1, giving (a) the Debye-Scherrer pattern of unoriented resin and (b) the fiber diagram of highly oriented resin. These patterns are essentially identical with patterns given by those polyoxymethylenes, the structures and properties of which were extensively studied by the Staudinger school in the 1920's.⁴⁻⁶

It was shown by Hengstenberg^{5,7} and Sauter⁸ that the high molecular weight polyoxymethylenes



(a)



(b)

Fig. 1. X-ray diffraction patterns of polyoxymethylene resin: (a) Debye-Scherrer photograph; (b) fiber diagram.

have a hexagonal unit cell with space group symmetry C_3^2 or C_3^3 and with lattice constants, $a = 4.46$ Å. and $c = 17.30$ Å. One helical chain molecule passes through each unit cell, with nine —HCHO— units in the identity period. The crystalline density calculated for this cell, 1.506 g./cc., is consistent with observed values of bulk density that lie in the range 1.40 to 1.45 for resins of moderate to high crystallinity. Our results agree with these investigations.

Since the discovery that stable high polymeric resins, suitable for molding and other thermal manipulation can be prepared, it became possible to study the crystallization behavior more extensively, particularly with respect to fabricated objects. It will be noted that the quantitative results given are for thin films for reasons stated below; these results are intended to describe the behavior of the polyoxymethylene chains which are distinctive. Application of these results to objects more massive than films is qualitatively possible,³ but requires attention to the specific object in question.

The preliminary studies described in this paper are concerned with three aspects of the crystallization behavior: (1) the extent of crystallization developed in the solid resin after typical thermal treatment; (2) the nucleation and growth of crystallites and spherulitic grain texture during crystallization from the melt; and (3) the variations in mechanical properties of films that can be obtained by factors such as crystallite and spherulite size through variations in the crystallization process.

EXPERIMENTAL PROCEDURES

1. Crystallinity

Estimation of crystallinity in the resins was made primarily by an x-ray diffraction technique. This technique is similar to one employed by Matthews, Peiser, and Richards for polyethylene.⁹

A Geiger-counter x-ray diffractometer was used to obtain a recorded pattern of the relative intensities of the (100) Bragg crystalline reflection and the amorphous diffraction halo. These maxima occurred, in the diffractometer scans, at scattering angles of 22.9 and 20.9°, respectively. The appropriate Lorentz-polarization, atomic scattering, and temperature factors were determined from graphs of these quantities plotted from published tabulations.¹⁰ After measurement of the recorded

amorphous and crystalline peak areas by planimetry, the corrected ratio of integrated amorphous and crystalline intensities was calculated from the equation

$$\frac{P_a}{P_c} = 0.56 \frac{A_a}{A_c} \quad (1)$$

Crystallinity was calculated from the expression

$$X_c = \frac{1}{1 + \frac{P_a}{P_c}} \quad (2)$$

It has been common practice to treat the specific volume of a semicrystalline high polymer as being an average of the specific volumes of crystalline and amorphous phases. Our preliminary attempts to demonstrate this were not successful when samples having widely different crystallization histories were included in the study. It is felt that this may be due to variations in crystalline density, amorphous density, or both.

Since the above samples included those prepared with so great a variety of thermal histories, it appeared still possible to use the specific volume as a measure of variations in degree of crystallinity for limited preparation conditions. The samples used for mechanical properties were such a series. The specific volume was measured and converted to percent crystallinity by the following expression:

$$X_c = \frac{V_a - V}{V_a - V_c}$$

where V_a is estimated to be 0.80 ± 0.02 cc./g. and V_c is estimated to be 0.664 ± 0.003 cc./g.

2. Crystallite Sizes

Estimations of the size of the crystalline regions or crystallites in the polymers were made by means of the Scherrer equation, relating crystallite dimension to x-ray diffraction line width:

$$L = \frac{0.9 \lambda}{(B - B_0) \cos \theta}$$

In this expression L is the crystallite size in Angstroms, λ is the wavelength of radiation in Angstroms, θ is the Bragg angle of the diffraction peak, B is the observed diffraction peak width in radians at half-maximum intensity, and B_0 is the instrumental line width, which depends on sample thickness, x-ray wavelength, and collimation slit size.

Crystallite sizes estimated by this method are of significance only on a relative basis, but are useful

in understanding the changes in physical properties of the polymer brought about by various thermal treatments.

From the two principal crystalline diffraction peaks of the acetal resins, measurements were made of two diameters of the crystallites. The peak at $2\theta = 22.9^\circ$ is the Bragg reflection from the (100) lattice planes parallel to the molecular chain axes. The width of this peak is a measure of the diameter L_1 of the crystallite in a direction perpendicular to the molecular axes, i.e., it is a measure of the square root of the number of chains passing through a crystallite. The peak at $2\theta = 34.4^\circ$ is the Bragg reflection from the (105) planes of the crystal lattice. The perpendicular to this set of planes is inclined at an angle of 41.8° to the molecular chain axes of the crystal. The width of this peak is, therefore, a measure of the length L_2 of the crystallite parallel to the molecular axis.

3. Spherulites

Films of acetal resins appear to crystallize into spherulitic patterns under any static conditions. There is little or no surface nucleation from glass or metal surfaces or heterogeneous nucleation from adventitious impurities. Specific details for obtaining particular spherulite sizes are given in the Discussion.

4. Mechanical Tests and Sample Preparation

Three testing machines were used: (1) the pneumatic impact tester (PIT),¹¹ (2) the microextensometer,¹² and (3) the Instron. The use of the PIT was limited by the relatively large sample required (about 1.5 in. \times 1.5 in.). The microextensometer was very useful for microscopic study of film behavior under stress.

The tests were all carried out on thin films using non-standard tests. Therefore, the results are not numerically equal to standard ASTM test data. Test specimens for the microextensometer were cut out of the films with the use of a razor blade and a metal template. The width (0.06–0.10 in.) was measured with a microscope filiar eyepiece calibrated against a stage micrometer. Thickness was measured with a micrometer. The length was determined by the jaw position of the microextensometer at negligible load. Samples varied in length from $1/2$ to $1\frac{1}{2}$ in. Specimens used in the Instron were stamped out with a special die that gave a sample 0.02 in. wide and 0.8 in. long with tab ends tapering off at 30° . The thickness was measured with a micrometer.

Films used for these tests were prepared by molding between aluminum sheets in a Preco press. These were generally held at 210°C . for 3 minutes and quenched and annealed for various cycles as given below. Test specimens were selected from portions of the films that revealed no bubbles or dirt under careful microscopic examination.

RESULTS AND DISCUSSIONS

1. Extent of Crystallization in Films

Some representative values of x-ray crystallinity of stable acetal resins are given in Table I. These data show the effect of compression molding upon crystallinity and show how number average molecular weight affects crystallinity in molding.

TABLE I
X-Ray Crystallinity of Polyoxymethylenes

Sample	$\bar{M}_n = 40,000$	$\bar{M}_n = 70,000$	$\bar{M}_n = 100,000$
Unmelted, dried resin	90%	87%	85%
Compression-molded film	81%	74%	70%

The crystallinity of molded and quenched film increases regularly with increasing quench temperature as shown in Figure 2. The level of crystallinity produced in films by quenching in the range 0 to 150°C . appears to be stable indefinitely at room temperature. Lower levels of crystallinity, produced by quenching at very low temperatures, are not stable and increase rapidly to higher levels on standing at room temperature. This behavior is associated with the low amorphous transition temperature found in linear acetal resins.^{3,13}

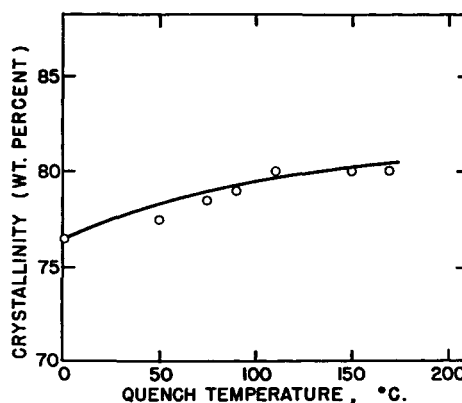


Fig. 2. Effect of quench temperature on room temperature crystallinity of acetal resin films compression-molded at 210°C .

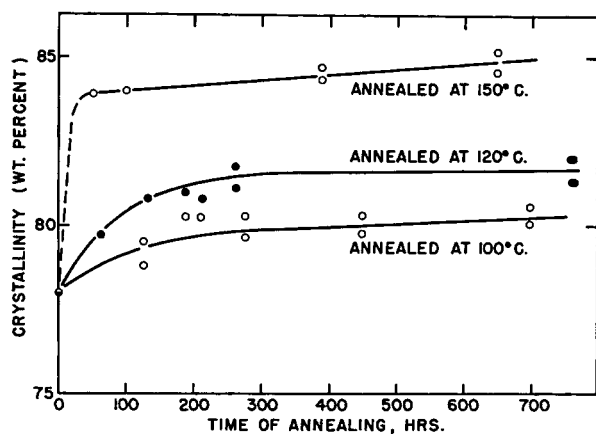


Fig. 3. Effect of annealing on room temperature crystallinity of polyoxymethylene (films molded 210°C., quenched to 50°C.).

The crystallinity of molded samples is increased moderately by annealing at elevated temperatures. For example, in Figure 3 is shown the increase of crystallinity with time of annealing at 150°C. of compression molded film samples quenched to 50°C.

The x-ray crystallinity, measured as a function of temperature, provides a means of studying the melting and crystallization properties. In Figure 4 is shown the variation of x-ray crystallinity with temperature for a compression molded film of polyoxymethylene of \bar{M}_n near 70,000. The crystallinity remained essentially constant up to 150°C. A gradual decrease of crystallinity occurred between 150 and 175°, followed by an abrupt decrease in the interval 175–180°C. The crystalline melting point, estimated from the melting curve, is between 179 and 181°C.

If the temperature of the melt was not taken above 181°C., crystal nuclei were not completely destroyed, and reversible melting and crystalliza-

tion was observed along the curve PM on increasing or decreasing the temperature. If the nuclei were destroyed by heating for several minutes above 181°C., then a characteristic and reproducible supercooling effect was observed. Crystallization took place along the curve FP', which intersects the temperature axis near 160°C. If crystallization was not allowed to go to completion it was found that reversible crystallization and melting could be observed along the curve FP'.

It may be concluded that these observations are in accord with observations on polymers generally, and with the notion that crystallization will proceed more rapidly once the chains have been "anchored" to a forming crystal lattice.

2. Crystallites and Spherulitic Grain Texture in Films

The crystallite sizes can be varied either by varying the quench temperature or by annealing. Time in the melt and melt temperature did not have appreciable effects over the range 190°C. to 220°C. The data plotted in Figure 5 show the effect of quench temperature on crystallite size. A general increase in size with temperature, especially noticeable at 150°C., is in general agreement with the predictions of the induced nucleation theory of spherulite growth.¹³

Annealing at temperatures above 50°C. increases crystallite size. The increase in average crystallite size was such as to define a two- to threefold increase in average crystallite volume. The corresponding increase in over all crystallinity in the samples, however, was only about 4%. From these facts it was concluded that crystallite growth involves the growth of larger crystals at the ex-

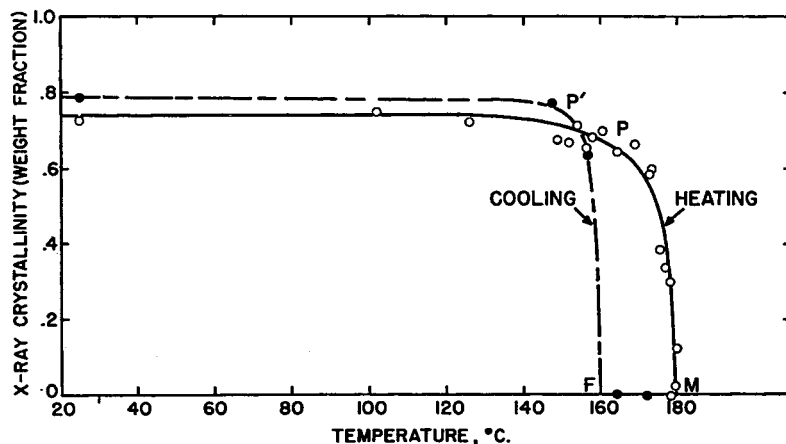


Fig. 4. Effect of temperature on crystallinity of acetal resin.

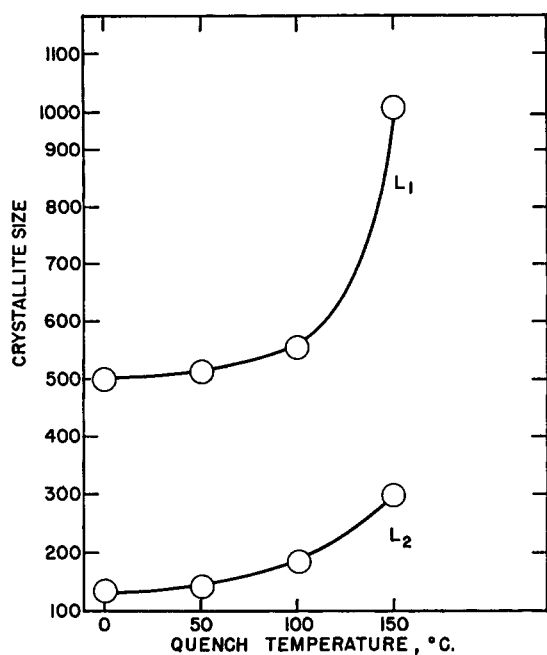


Fig. 5. Effect of quench temperature on crystallite size, at room temperature, of acetal resin.

pense of smaller crystals of lower stability. While the accuracy and precision of the measurements were not sufficient to justify the calculation of rates and activation energies, data from a large number of measurements enabled us to see some details of the transformation. The crystallite diameter parallel to the polymer molecule, L_2 , undergoes a fairly

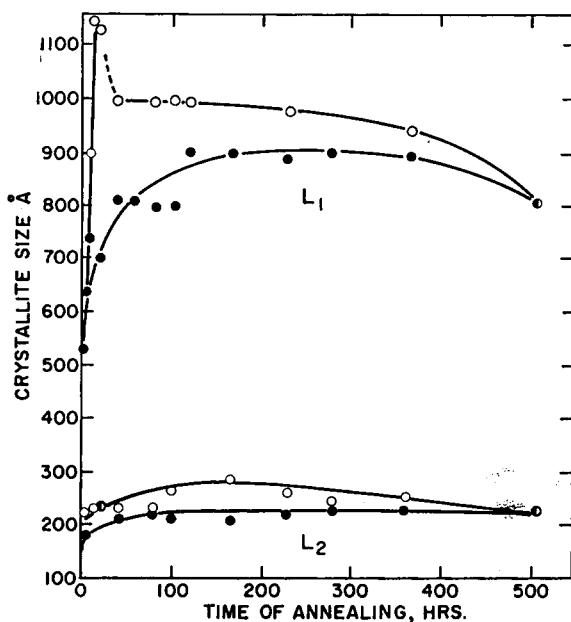
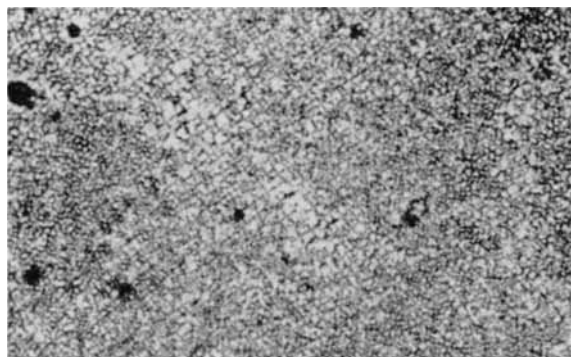
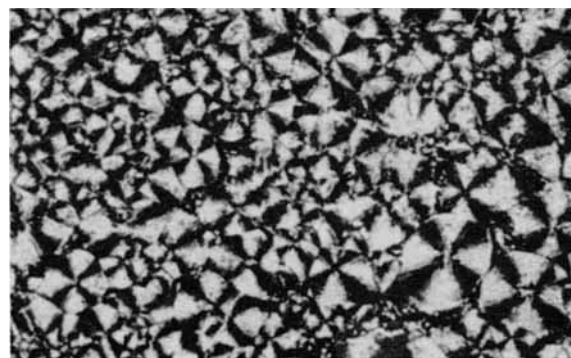


Fig. 6. Effect of annealing on crystallite size: (O) annealed at 120°C.; (●) annealed at 100°C.

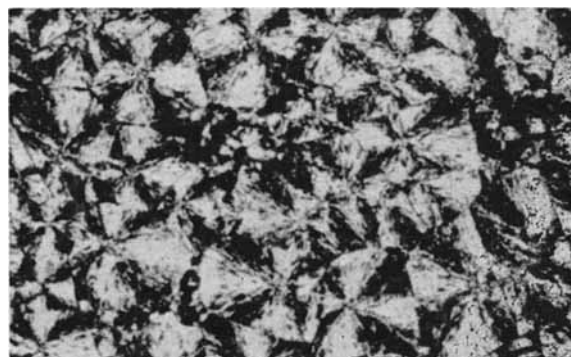
regular increase, generally approaching a value of 250 Å. in samples quenched to 100°C. or lower, and 300 Å. for samples quenched to 120°C. (Fig. 6). The rate at which the maximum size is approached varies directly with the temperature of annealing. The crystallite diameter perpendicular to the polymer molecule, L_1 , passes through a maximum value in the range of 1000 to 1500 Å. An observed decrease in this diameter upon prolonged annealing is most probably explained as due to the development of flaws in the crystallites, which causes fragmentation parallel to the polymer chains.



(a)



(b)



(c)

Fig. 7. Temperature ranges of nucleation (melt 2 min. at 190°C.): (a) 0°C. quench; (b) 25°C. quench; (c) 140°C. quench.

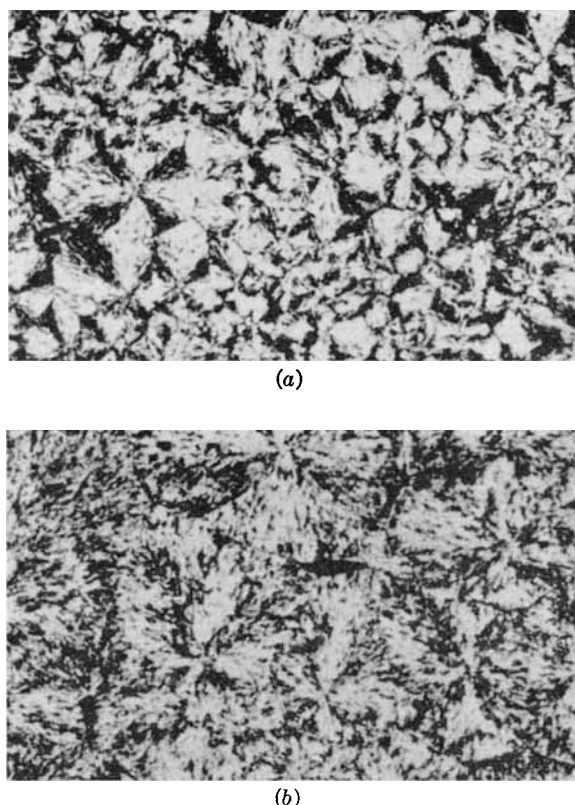


Fig. 8. Temperature ranges of nucleation (melt 2 min. at 190°C.): (a) 150°C. quench; (b) 156°C. quench.

The acetal resins show a highly developed spherulitic grain structure over a wide range of conditions of solidification from the melt.

Spherulite patterns can be divided into three classes by the temperature ranges in which they were grown, below 150°C., from 150 to 155°C., and above 155°C. Spherulites produced from melts quenched to temperatures below 150°C. show the normal type of spherulite pattern with a sharply defined Maltese cross in polarized light (Fig. 7). Spherulites grown at a temperature in the neighborhood of 150–155°C. have a less distinct pattern which becomes progressively more diffuse as the temperature of spherulite growth is increased. As shown in Figure 8, there is a gradual transition from the sharp, Maltese-cross spherulites to the dendritic-type spherulites. In preparing spherulites by slow growth at the higher temperatures, 160–164°C., a ring formation is sometimes observed. This type of band or ring formation has been observed to occur in other polymers, such as polyglycol adipate¹⁴ and linear polyethylene. We have found that by repeated meltings and slow cooling this effect could be amplified to give

enormous spherulites, 3–5 mm. in diameter, as seen in Figure 9, showing only a heavy intense banding rather than a characteristic cross pattern.

3. Effects of Fine Structure on Mechanical Properties of Thin Films

The aforementioned work on characterization of the fine structure of these acetal resins may be examined to elucidate the role played by these features on the mechanical properties. Inspection of the crystallization results showed that thickness of the sample could be an important factor; i.e., thick samples would have uneven cooling rates and therefore be nonuniform in fine structure. The effects reported below, if present in molded bars, are obscured in virtually all standard tests for mechanical properties. Consequently, the effect of fine structure (spherulite size, crystallite size and crystallinity) on mechanical properties was studied for compression-molded films. Surfaces of thicker objects would be expected to show some of the effects discussed. Use of films also facilitated determination of spherulite size, crystallite size, and crystallinity.

The measurements in the following section were all conducted at 25°C. It does not appear possible to quench a sample rapidly enough to reduce the crystalline level much below 65%. That is to say, samples were quenched to much lower temperature, say –80°C., but after they were raised to 25°C. for observation, they had already obtained these higher levels of crystallinity. Thus, if purely amorphous, i.e., glassy, polyoxymethylene is to be studied, the measurements must be carried out at very low temperatures, say below –100°C. As a result we are limited to a range of crystallinity between 65 and 85%.

From the stress-strain curve the tensile modulus, the yield point, and the total elongation to break were measured. The effect of varying the crystallinity on these three properties is shown in Figure 10. The modulus increases about 75% and yield point about 30% over this range. The elongation dropped to about 30% of its highest value for this range of specific volume. In order to obtain samples having the lower values of crystallinity, it was necessary to quench them to low temperatures and then to make the measurements immediately. If the samples were held at room temperature for longer than this time, they increased their crystallinity to a value above 75%.

The major effect of variations of spherulite morphology was the effect of toughness. This



Fig. 9. Spherulite structure produced in polyoxymethylene resin on repeated melting. Scale: $1\frac{1}{4}$ in. = 1 mm.

was produced through the ductile behavior of individual spherulites. Both brittle failure and ductile behavior originated at boundaries between spherulites. Brittle failure usually consisted of propagation of a crack along a spherulite surface (Fig. 11c), but the fracture could also be found to propagate through a spherulite. Ductility seemed to consist of a cold drawing from the surface of a spherulite. Two examples of this are shown. Figures 11a and 11b show progressive stages of this. In Figure 12a is shown a spherulite after drawing. Prior to this, the boundaries within this specimen

were circular. Thus the surface appeared to progress across the spherulite. This is in contrast to the behavior of spherulites in polyethylene, which appear to deform as whole units.¹¹

A single large spherulite appeared to have a slightly higher yield point than a specimen containing a family of spherulites. This suggested that rate of loading should have an important effect on the ductility, and it was found that the samples with larger spherulites lost ductility as the testing rate was increased.

Both spherulite and crystallite size decrease with

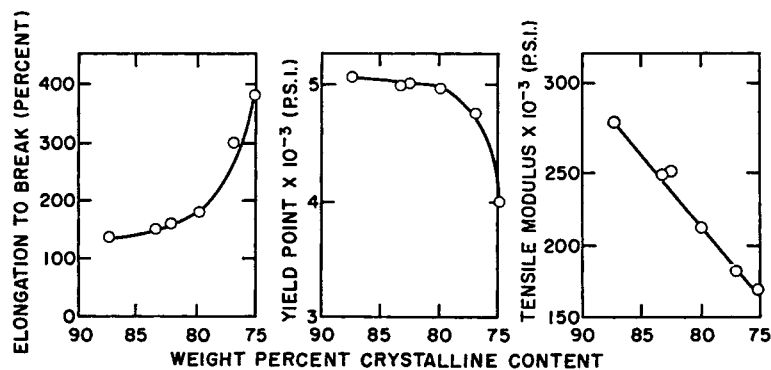


Fig. 10. Relationship of mechanical properties and crystalline content of acetal resin (compression-molded film, strain rate of 250%/min.).

TABLE II
Effect of Spherulite Size on Elongation to Break

Quench temperature, °C.		Elongation to break, % ^a		
		At 25%/min.	At 250%/min.	At 2500%/min.
0	Increasing	150 ^b	37 ^b	29
50	spherulite	63 ^b	42 ^b	10
100	size	18	14	2
150	↓	20	2	2

^a All values average of three determinations.

^b Obvious necking of samples.

decreasing quench temperature, but the major effect on properties appears to be that due to variations in spherulite size. Results from measurements on the Instron are shown in Table II.

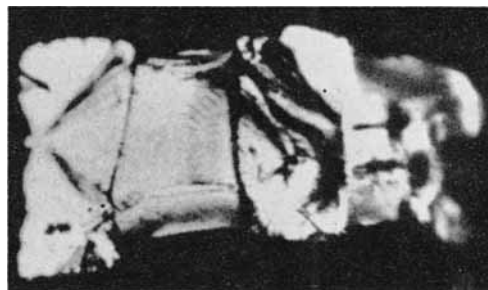
For acetal resins, the impact toughness of a sample depends primarily on the ductility of the sample. To demonstrate this point, samples were tested in the Pneumatic Impact Tester to attain sufficiently high rates of loading to differentiate between samples.

In order to reduce the effects of crystallite size and degree of crystallinity, samples were quenched to attain differing spherulite sizes and then annealed. This enabled us to obtain samples with similar crystallite dimensions and very nearly identical levels of specific volume but with markedly different spherulite sizes. The results were consistent with the view that the larger spherulites provide less ductility and are illustrated in Table III.

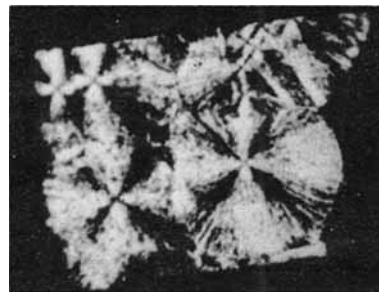
The effect of crystallite size on mechanical properties is exceedingly difficult to separate from other variables and to determine precisely. There appears to be little effect on the modulus, with one exception: very large crystallites (1600 Å. × 500 Å.) were obtained in a lower molecular weight sample ($M_n = 28,000$). The stiffness was about four times the value for other samples of this degree of crystallinity but which had smaller crystallites and higher molecular weights. It is difficult to ascribe this result to the low molecular



(a)



(b)



(c)

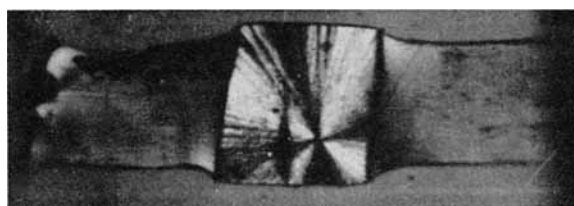
Fig. 11. (a) Photomicrograph of a strip of film being drawn in the microextensometer; (b) photomicrograph showing (a) a short time later; (c) photomicrograph of a strip of film after brittle tensile fracture showing that the crack had followed cleanly along the boundary of a spherulite.

weight since we should expect high polymeric behavior to occur much before chain lengths of 1000 atoms.

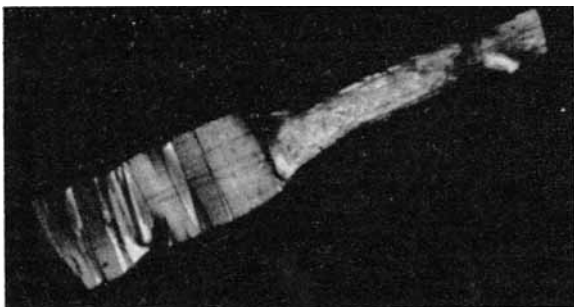
In order to show that crystallite size and degree of crystallinity played a minor role in toughness, samples were quenched to 0°C., and then some were annealed to increase the crystallite size and degree of crystallinity. The results were consistent in this respect and are illustrated in Table IV.

TABLE III

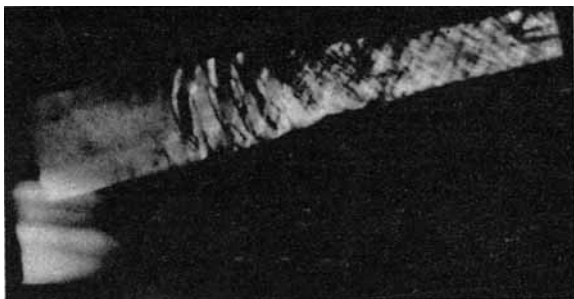
Sample preparation	Crystallite size, Å.		Spherulite size	PJT (av. of 3 detns.), ft.-lb./in.	
	L_1	L_2			
Preparation A	Press 200°C., quench 150°C.	820	300	very large	83
Preparation B	Press 200°C., quench 0°C., plus annealing 50 hr. at 150°C.	900	330	small	378



(a)



(b)



(c)

Fig. 12. (a) Photomicrograph of a film strip which was spanned by a single spherulite. Note that a portion of the spherulite retained its original identity. (b) Photomicrograph of a strip of film which was cut perpendicular to the direction of orientation. Note the crossed, Lender-type of cold draw. (c) Photomicrograph of strip of film which was cut and drawn in the direction parallel to the orientation.

TABLE IV

Sample preparation	Crystallinity, %	Crystallite size, A.		PIT (av. of 3 detns.), ft.-lb./in.
		L_1	L_2	
Press 200°C., quench 0°C.	80	470	140	383
Annealed 50 hr. at 150°C.	85	820	260	385

When films are extruded, the melt can be partially oriented. Under such conditions, only very small spherulites are produced. We would expect but could detect no directional dependence on tensile modulus or yield point on these samples.

The cold drawing behavior, however, was markedly different in the two directions (Fig. 12).

References

1. C. E. Schweitzer, R. N. MacDonald, and J. O. Punderson, *J. Appl. Polymer Sci.*, **1**, 158 (1959).
2. T. A. Koch and P. E. Lindvig, *J. Appl. Polymer Sci.*, **1**, 164 (1959).
3. W. H. Linton and H. H. Goodman, *J. Appl. Polymer Sci.*, **1**, 179 (1959).
4. H. Staudinger, *Die Hochmolekularen Organischen Verbindungen Kautschuk und Zellulose*, Springer, Berlin, 1932.
5. H. Staudinger, H. Johner, R. Signer, G. Mie, and J. Hengstenberg, *Z. physik. Chem.*, **A126**, 425 (1927).
6. H. Staudinger, R. Signer et al.; *Ann. Chem. Liebigs*, **141**, 474 (1929); *Z. Krist.*, **70**, 193 (1929).
7. J. Hengstenberg, *Z. physik. Chem.*, **A126**, 435 (1927); *Ann. Physik*, **84**, 245 (1927).
8. E. Sauter, *Z. physik. Chem.*, **21B**, 186 (1933).
9. J. L. Matthews, H. S. Peiser, and R. B. Richards, *Acta Cryst.*, **2**, 85 (1949).
10. H. P. Klug and L. E. Alexander, *X-Ray Diffraction Procedures*, Wiley, New York, 1954.
11. R. D. Spangler and E. B. Cooper, *J. Appl. Phys.*, **28**, 329 (1957).
12. W. M. D. Bryant, *J. Polymer Sci.*, **2**, 447 (1947).
13. C. F. Hammer and P. H. Geil, Jr., paper presented at the San Francisco Meeting of the American Chemical Society, April 15, 1958.
14. J. J. Point, *Bull. classe sci.*, Acad. roy. Belg., **39**, 435 (1953).
15. B. B. Burnett and W. F. McDevit, *J. Appl. Phys.*, **28**, 1101 (1957).

Synopsis

Stable, high molecular-weight polyoxymethylene resins show the room temperature x-ray crystalline patterns expected from early work on unstable or low molecular weight polyoxymethylene resins. High-temperature studies show the specific features of this resin to be: (1) disappearance of crystalline peaks at 180°C., and (2) a super-cooling of 15°C. (i.e., nucleation periods much greater than 15 minutes for temperatures above 165°C.). Studies utilizing optical birefringence show similar melting and freezing behavior. Birefringence studies show effect of quench temperature and melt time and temperature on the average spherulite size and appearance. Under suitable conditions, very large (1 mm. diameter) spherulites can be grown. The effect of variations in the fine structure (spherulite size, crystallite size, and degree of crystallinity) on mechanical properties of thin sections shows that: (1) an increase in crystallinity (by specific volume) gives higher moduli, higher yield strengths, and lower values of elongation to break; (2) smaller spherulites increase the ductility, or ability to retain ductility as the rate of strain of the test is increased; (3) changes in crystallite size over a twofold range have lesser effects on these properties.

Résumé

Des résines polyoxyméthyléniques stables de haut poids moléculaires présentent les diagrammes cristallins aux

rayons-X auxquels on pouvait s'attendre de par les travaux antérieurs sur les résines polyoxyméthyléniques instables ou de bas poids moléculaires. Des études à haute températures montrent que les aspects spécifiques sont: (1) la disparition des pics cristallins à 180°C et (2) une surfusion à 15°C (c'est à dire: des périodes de nucléation de beaucoup supérieures à 15 minutes pour des températures supérieures à 165°C). Des études utilisant la biréfringence optique montrent un comportement similaire pour la fusion et la congélation. Des études de biréfringence montrent l'effet de la température d'extinction et du temps de fusion sur l'aspect et la taille moyenne des sphérulites. Dans des conditions optima on peut obtenir de très larges sphérulites (1 mm. de diamètre). L'influence des variations de la structure fine (taille des sphérulites, taille des cristallites et degré de cristallinité) sur les propriétés mécaniques de sections minces a montré (1) qu'une augmentation de la cristallinité (par volume spécifique) donne de plus hauts modules, des ténacités plus élevées et de plus faibles valeurs de l'élongation à la rupture; (2) que des sphérulites plus petits accroissent la ductilité ou l'aptitude à garder la ductilité lorsque la vitesse de tension du test est accrue et (3) que les changements de la taille des cristallites du simple ou double ont moins d'influence sur ces propriétés.

Zusammenfassung

Stabile hochmolekulare Polyoxymethylenharze zeigen bie

Raumtemperatur kristalline Röntgenbeugungsdiagramme, wie sie nach den früheren Untersuchungen an instabilen oder niedrigmolekularen Polyoxymethylenharzen zu erwarten sind. Untersuchungen bei hohen Temperaturen zeigen das folgende spezifische Verhalten dieses Harzes: (1) Verschwinden der kristallinen Maxima bei 180°C und (2) eine Unterkühlung von 15°C (d.h. Keimbildungsdauern weit grösser als 15 Minuten bei Temperaturen oberhalb 165°C). Untersuchung der optischen Doppelbrechung zeigt ein ähnliches Schmelz- und Erstarrungsverhalten. Doppelbrechungsmessungen zeigen den Einfluss der Abschrecktemperatur und der Schmelzdauer und -temperatur auf die mittlere Sphärolithgrösse und -ausbildung. Unter geeigneten Bedingungen können sehr grosse (1 mm Durchmesser) Sphärolithe gezüchtet werden. Der Einfluss von Änderungen der Feinstruktur (Sphärolithgrösse, Kristallitgrösse, und Kristallinitätsgrad) auf die mechanischen Eigenschaften dünner Schnitte besteht in folgendem: (1) eine Zunahme der Kristallinität (aus dem spezifischen Volumen) ergibt höhere Moduli, höhere Fliessgrenzen und niedrigere Werte der Bruchdehnung; (2) kleinere Sphärolithe erhöhen die Duktilität oder die Fähigkeit bei Erhöhung der Verformungsgeschwindigkeit bei der Prüfung Duktilität beizubehalten und (3) Änderungen der Kristallitgrösse über einen zweifachen Bereich haben auf diese Eigenschaften geringeren Einfluss.

Received November 17, 1958