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Microscopical Measurement of Orientation of High-Impact Polystyrene Extruded Sheet

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INTRODUCTION

Many commercial high-impact polystyrenes can be classified as heterogeneous or two-phase polymeric systems. Dispersed in the polystyrene matrix are particles of styrene–butadiene copolymer. The composition, structure, and dispersion of these particles has a definite influence on the properties of the material.[1]

The inherent opacity of this material requires that it be reduced to thin sections for microscopical study. A microtome sectioning technique developed by P. A. Traylor[2] makes possible the phase contrast microscopical study of this two-phase system *in situ*.

Microtome sections cut from normal high-impact polystyrene production

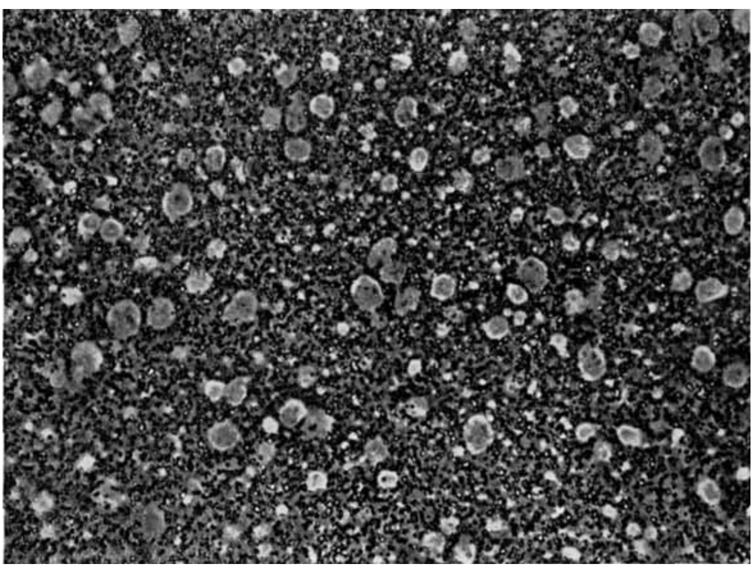

Fig. 1. Microtome section of high-impact polystyrene sheet cut parallel to machine direction. Phase contrast, 800×.

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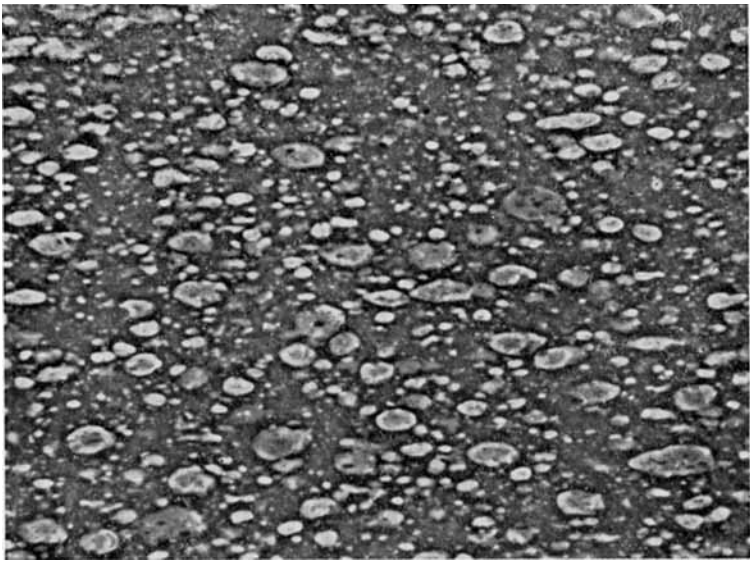

Fig. 2.- Cross section of 90° bend in vacuum-formed natural high-impact polystyrene sheet. Phase contrast, 400×.

sheet, extruded under conditions designed to produce a minimal uniaxial orientation, show that the copolymer particles are essentially spherical in shape. Figure 1 is a photograph taken with a phase contrast microscope of such a section cut parallel to the direction of extrusion. The circular particles are the styrene–butadiene copolymer; the small particles are ti-

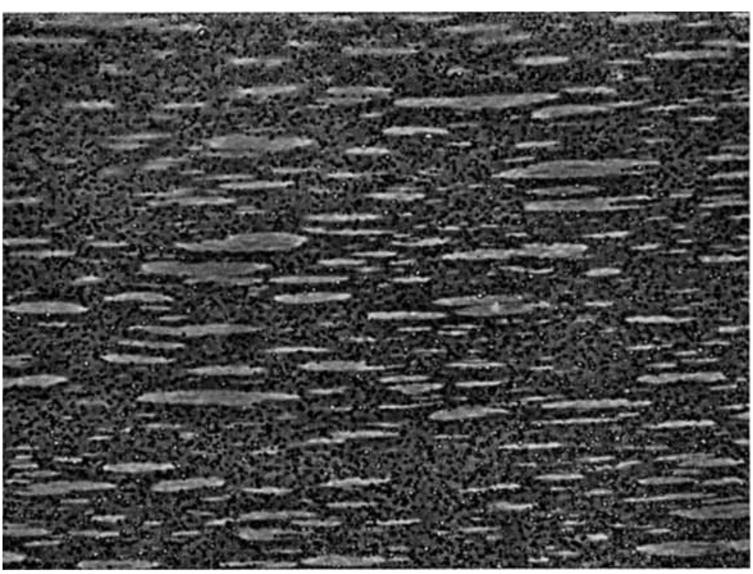

Fig. 3. Microtome section of deep draw. Vacuum-formed high-impact polystyrene sheet. Phase contrast, 700×.

tanium dioxide filler. Sections cut in the cross direction also present circular cross sections of the copolymer particles.

In the course of the microscopic investigations of high-impact polystyrene sheet extruded at this laboratory it was observed that in the vacuum forming of the sheet the styrene–butadiene particles were distorted in a way which reflected the drawing of the sheet. In cross sections of a 90° bend of a formed part, the particles were elongated in the direction of draw (Fig. 2). In cross sections of an area in the bottom of a deep draw the copolymer particles were extremely elongated (Fig. 3).

Fig. 4. Microtome section cut parallel to extrusion direction of high-impact polystyrene sheet. High degree of orientation. Phase contrast, 700×.

EXPERIMENTAL

Since the copolymer particles responded to forces in forming, it seemed reasonable to assume that they would respond in a like manner to the stretching normally found in sheet extrusion. A series of samples was produced on a 1-in. 20/1 (L/D) Modern Plastics Machinery Laboratory extruder on which different degrees of orientation were obtained by varying the speed of the take-off roles while maintaining constant sheet thickness. These samples had orientation shrinkage values of 10, 20, 34, and 43% as measured by the orientation shrinkage test method described by Davis et al.[3]

Microtome sections cut in both machine direction and cross machine direction of the most highly oriented sheet indicated that the copolymer particles were ellipsoidal, their major axes oriented parallel to the direction of orientation of the sheet. Figure 4, of a section cut parallel to machine direction, presents elliptical cross sections of the copolymer particles.

Sections cut in the cross machine direction showed copolymer particles of almost circular cross sections.

A sample of this highly oriented sheet, after relaxation in the orientation shrinkage test, was sectioned in both machine and cross direction. These microtome sections showed that the copolymer particles had returned to their spherical shape (Fig. 5).

If the copolymer particles respond directly to machine elongation, then the ellipticity of these particles, as measured in cross sections parallel to the direction of orientation, should give a measure of the degree of orientation of the sheet. According to this assumption, the per cent orientation shrinkage, O, could be defined by the following empirical formula:

$$O = [1 - (b/a)]100$$

where a is the diameter of the major axis, b of the minor.

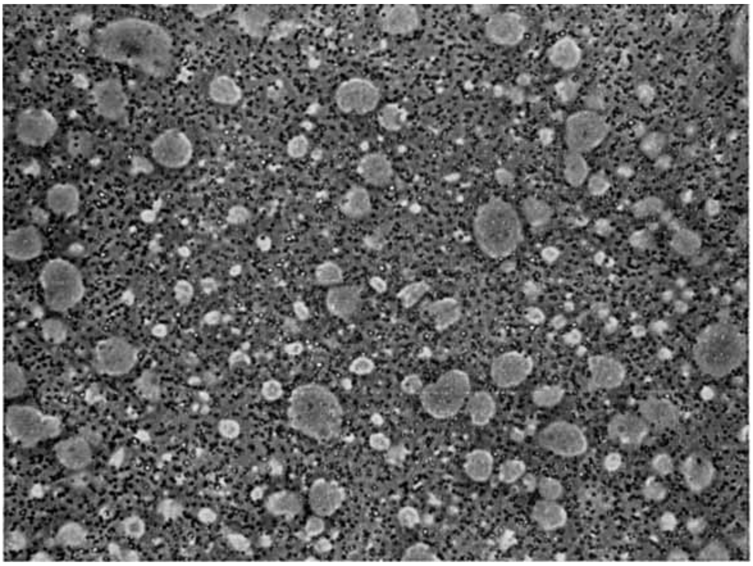

Fig. 5. Microtome section cut parallel to extrusion direction of high-impact polystyrene sheet. High orientation. After oven shrinkage test. Phase contrast, 700×.

It is apparent that for a nonoriented sheet, in which particles would be essentially spherical, this function approaches zero. At the extreme of high orientation, the function approaches unity, or 100%.

To determine some correlation between the shape of the copolymer particles in microtome sections of oriented sheet and the orientation shrinkage values obtained by the oven test, a series of measurements was made. Microtome sections were cut parallel to the machine direction of each of the oriented samples. A number of phase contrast photomicrographs was made, of different areas of several sections. These photomicrographs were enlarged photographically to a total magnification of 2500× for convenience in measuring. The major and minor axes of 100 particles of each orientation were measured. The data resulting from measurement of the four samples are summarized in Table I.

TABLE I

Sample no.	Ratio b/a	S. D.	Orientation shrinkage, %	Orientation shrinkage, oven method, %
1	0.861	0.087	13.9	10
2	0.810	0.115	19.0	20
3	0.635	0.085	36.5	34
4	0.526	0.103	47.4	43

The orientation shrinkage, O values, taken from these samples by the oven method were based on averages of four measurements of three samples from each lot of oriented sheet. A statistical analysis of data previously acquired in establishing the oven method produced a standard deviation of the means of 1%.

While the spread of the data obtained from measurement of the photomicrographs is much greater than that of the oven shrinkage data, the data indicate that an assessment of degree of orientation may be made in this way. No difficulty would be encountered in ranking the samples whose sections appear in Figure 6. By making a few measurements of the ellipticity of the copolymer particles, an approximate value for degree of orientation may be obtained.

DISCUSSION

A good assessment of the degree of orientation of high-impact polystyrene sheet may be made by measuring the ellipticity of the copolymer particles in microtome sections of the sheet. In cases of very small samples, this technique can be extremely useful. Its real value is its potential application to the study of vacuum forming and injection molding of high-impact polystyrene. Many studies of flow patterns and frozen-in stresses in polystyrene have been made with the use of birefringence measurements.[4,5] The application of birefringence techniques to an opaque material is, of course, impossible. The orientation of the copolymer particles in injection moldings has been pointed out,[6] but no attempt has been made to relate quantitatively the particle shape to any property of the material.

The fact that the styrene–butadiene copolymer particles respond in a measurable way to deformations imposed on the styrene matrix can be used as a built-in indicator of strains in the material. This should open new avenues of investigation in sheet forming as well as injection molding.

The author wishes to express appreciation to D. A. Davis and other members of the Chemistry Laboratory for samples as well as advice and encouragement.

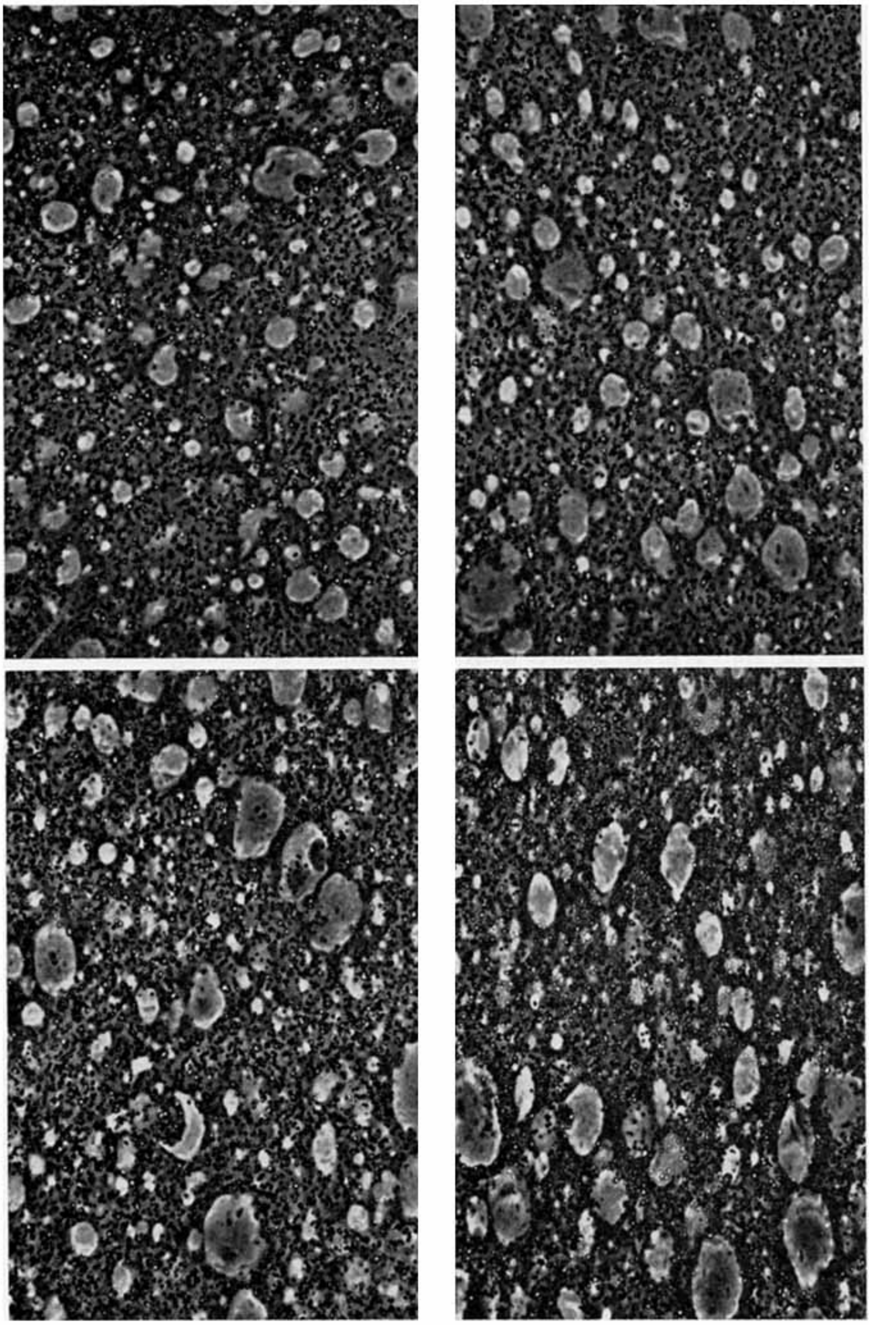

Fig. 6. Microtome sections cut parallel to extrusion direction. Orientation series: (*a*) sample 1, 13%; (*b*) sample 2, 19%; (*c*) sample 3, 37%; (*d*) sample 4, 47%. Phase contrast, 700×.

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Synopsis

The polystyrene–butadiene copolymer particles in high-impact polystyrene sheet respond to forces imposed on the sheet. Measurement of the ellipticity of these particles in thin microtome sections provides a measure of orientation of extruded sheet.

Résumé

Les particules de copolymère styrène–butadiène, dans une feuille de polystyrène à haute pression, répondent aux forces imposées à la feuille. La mesure de l'ellipticité de ces particules, dans les sections minces coupées au microtome, permet de déterminer l'orientation de la feuille extrudée.

Zusammenfassung

Die Polystyrol–Butadienecopolymerparitkel in Filmen aus schlagfestem Polystyrol reagieren auf Kräfte, die auf den Film einwirken. Die Messung der Elliptizität dieser Teilchen in dünnen Mikrotomschnitten liefert ein Mass für die Orientierung extrudierter Filme.

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