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IN POLYMER FILMS

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SUMMARY

Results are presented of measurements of the molecular orientation and of the effect of such orientation on the mechanical properties of some polymer films. A sonic pulse method of detecting orientation is described, and orientation scans are presented of polyethylene, polypropylene, and poly(ethylene terephthalate) film. The mechanical anisotropy is shown by the variation of the tensile properties and the relaxation modulus as a function of direction in the plane of the film. The orientation and anistropy are compared and some practical aspects of the anisotropy are reviewed.

INTRODUCTION

It is a well-established fact that molecular orientation can change the mechanical properties of a polymer, and that these properties will vary depending on the amount or degree of orientation. Moreover, the properties will change with direction relative to the orientation direction, that is, they will be anisotropic (refs. 1 to 3). As a result, a considerable amount of work has been done on orientation effects in moldings (refs. 4 to 6), fibers (refs. 7 and 8), sheets (refs. 9 and 10), and film (refs. 11 to 13).

In recent years there has been an increased use of polymer films in aerospace structures such as solar concentrators and passive communications satellites (refs. 14 to 16). In some of these structures the film may be subjected to sudden or sustained loads in several directions. The mechanical response of the film to such loads can vary depending on the anisotropy which results from molecular orientation. Consequently, it is of value to measure the mechanical anisotropy in films and investigate means of determining the molecular orientation so that the mechanical load response can be estimated.

One method of observing orientation is by measuring the sonic velocity in the polymer. This method has been used for years in the study of the modulus and orientation of fibers (refs. 17 to 21). This paper describes the application of the sonic velocity method to polymer films which have been used or proposed for use in aerospace structures. The molecular orientation and mechanical anisotropy are reported and compared

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for an unoriented polyethylene film, a uniaxially oriented polypropylene film, and a biaxially oriented poly(ethylene terephthalate) film.

DESCRIPTION OF MATERIALS AND TESTS

Materials

Table I lists the polymer films and some important characteristics of each. Although the polyethylene (PE) film had a small amount of transverse orientation which was induced in the normal processing of the film, it is considered to be unoriented for the purpose of this investigation. The polypropylene (PP) film was highly oriented in the transverse direction, and the poly(ethylene terephthalate) (PETP) film was biaxially oriented and should be more nearly isotropic than the other two films. All of the films were crystalline as shown by X-ray diffractograms made at different angles in the plane of the film. The relative peak heights of the diffractograms, based on 10,000 counts full scale, are listed in table II, and the heights are normalized to the longitudinal (0°) direction.

Sonic Velocity Test

The sonic velocity tests were performed with an instrument similar to that used in reference 21. The instrument (fig. 1) includes two transducers, one of which produces a pulse centered at 8,000 Hz, 194 times each second. When a film (or a fiber) is coupled to the transducers, the pulses generated by one are detected by the other transducer. The electronic clock measures the time required for the pulse to travel from one transducer to another. Then the sonic velocity is simply the distance between the transducers divided by the transit time. A 6-inch-square (15.24-cm) piece of film is secured to a frame with the longitudinal and transverse directions in known positions. The film can then be rotated in its plane to obtain sonic velocity values at different angles. Transit times were measured on each film from 0° (longitudinal direction) to 90° (transverse direction) in 10° increments, plus a measurement at 45°, for each of three transducer spacings. The measured times were plotted as a function of the transducer spacing and generally provided the expected straight line. The slope of the line was the velocity for that particular angle in the film.

Once the sonic velocity is obtained the sonic modulus can be calculated from the expression

$$E = \rho(1 - \nu^2)v^2$$

where ρ is the density, ν is Poisson's ratio (taken as 0.4), and v is the sonic velocity. This equation is derived from a consideration of the propagation of longitudinal waves in an infinite plate (see ref. 22). If the sonic velocity is measured in a long, cylindrical shape, such as a fiber, then the Poisson effect can be neglected and the equation reduces to

$$E = \rho v^2$$

which is the form frequently used in measuring the sonic modulus of fibers (ref. 23).

Tensile Stress-Strain and Relaxation Tests

The tensile tests were performed on 0.5-inch-wide (1.27-cm) strips of film utilizing a 3.0-inch (7.62-cm) grip separation and a testing speed of 2.0 inches (5.08 cm) per minute. An integrator associated with the testing machine computed the area under the stress-strain curve so that the energy required to break the film strip could be measured. From the stress-strain curve it was possible to determine the initial tangent modulus, the 0.2-percent offset yield stress, the tensile strength (stress at break), and the elongation at break.

The stress relaxation tests were conducted on 0.5-inch-wide (1.27-cm) strips of film using a 5-inch (12.70-cm) grip separation. The test specimens were extended to approximately 10-percent strain at a testing speed of 2.0 inches (5.08 cm) per minute. With the specimen in the extended position the crosshead was stopped and the stress was allowed to relax for 1,000 seconds (16 minutes and 40 seconds). The stress at any time, divided by the strain (approximately 10 percent in this case), is referred to as the relaxation modulus.

RESULTS AND DISCUSSION

The test results are listed in tables III and IV and illustrated in Figures 2 to 5 and some concepts of molecular orientation are reviewed below.

Molecular Orientation

Polymers, such as those described in this paper, consist of molecular chains which are assemblages, or repeat units, of atoms held together by covalent bonds (see table I). These intramolecular bonds are considerably stronger than the intermolecular bonds, or Van der Waal's forces, between the chains. Typical intramolecular bond strengths may range from 80 to 150 kcal per mole, compared to intermolecular bond strengths of less than 10 kcal per mole (ref. 24). Hence, there is a strength anisotropy which is inherent to the molecular chain so that the load response of the chain will depend partially on whether the load direction is parallel or nonparallel to the chain axis.

In addition to the strength anisotropy, the molecular chain has a tendency to assume a kinked and highly contorted configuration which gives it an overall roughly spherical shape (ref. 25). The kinked configuration is the result of the limited number of definite angles which can exist between the atoms in the chain under equilibrium conditions. (The carbon atom, for example, has four such positions at which other atoms may be bonded to it.) Although a straight molecular chain is theoretically possible, a folded, twisted, randomly shaped configuration is more probable. The chain can be extended by mechanical force, however, especially if there is sufficient heat present to increase the thermal vibrations of the atoms and therefore soften the polymer. The chain will result in the bonds being rotated, bent, or stretched relative to one another, analogous to the way a folded carpenter's rule may be extended. (It is bond rotation, rather than bending or stretching, that accounts for most of the chain extension (ref. 26).) When the chain is extended, the movements which require the least force will take place first. Those movements requiring more force will not take place until the chain is extended farther. Therefore, if a molecular chain is oriented by stretching the randomly kinked chain into one which has some preferred axis of orientation, then the result will be a stiffer chain.

In general, then, molecular orientation has at least two effects: it changes the absolute value of the mechanical properties and it changes the distribution of these properties with regard to direction in the polymer.

Sonic Velocity

The results of the sonic velocity measurements are listed in table III and illustrated in Figure 2. Included in table III are values of the sonic modulus.

The PE film, which was essentially unoriented, had a relatively low sonic velocity on the order of 4×10^3 feet per second (1.2×10^3 meters per second). The film had only a small variation of velocity as a function of angle in the plane of the film, the high value of 4.5×10^3 feet per second (1.4×10^3 meters per second) occurring at 90° (transverse direction). Because the sonic velocity and the density of the PE film were low, the sonic modulus (table III) was also low with values ranging from 1.5 to 2×10^5 psi (1.03 to 1.38×10^9 N/m^2). The orientation scan of the PE film (fig. 2) is illustrative of what may be expected from the unoriented film: the sonic velocity (and modulus) is low and there is little variation in the velocity as a function of angle in the plane of the film.

By contrast, the PP film had a large variation in sonic velocity, a variation which reflected the high degree of uniaxial orientation in the 90° direction. It is remarkable also that the velocity shows little change (less than 10 percent) for angles up to 50° after which it rises sharply to the maximum value at 90°. Such a variation suggests that the film would be useful under unbalanced biaxial loads, such as those occurring in internally pressurized cylinders where the circumferential or hoop stress is twice the axial stress. The sonic modulus of the PP film is from three to five times the modulus of the PE film.

The sonic velocity scan for the PETP film was considerably different from what might have been expected inasmuch as the film was biaxially oriented. It is obvious (fig. 2) that biaxial orientation does not necessarily mean the same degree of orientation throughout all directions in the plane of the film. The sonic velocity varies from approximately 8×10^3 feet per second (2.4×10^3 meters per second) at 0° to 11×10^3 feet per second (3.4×10^3 meters per second) at 90°. The sharp decrease in velocity at 45° may be the result of a high degree of biaxial orientation inasmuch as the other less oriented films showed some evidence of velocity minima occurring in the 45° region. The sonic modulus for the PETP film (table III) is the highest of the three films, 1 to 2×10^6 psi (0.69 to 1.38×10^{10} N/m^2). The high modulus is due in part to the high sonic velocity and density. Of considerable importance, however, is the structure of the molecular chain (see table I). The inclusion in the PETP chain of the benzene ring and the $C = O$ dipoles adds a relatively stiff segment and enhances the intermolecular bonding. As a result, the overall polymer structure is denser, stiffer, and has a higher glass transition temperature (ref. 27) than do the less complicated PE and PP structures.

It is apparent, from figure 2, that the sonic velocity measurements give at least a qualitative measurement of molecular orientation. The values of the sonic moduli also are qualitatively correct, that is, the PE film had the lowest and the PETP film had the highest modulus. The values are probably quantitatively correct as well. The sonic velocity for a given film at a given angle was lowest when it was measured on the 6-inch-square (15.24-cm) piece of film. The velocities which were measured on strips of film were generally higher, the highest values being obtained on the most narrow strips. The velocity increase is caused by the boundary conditions which are minimized or eliminated in the 6-inch-square (15.24-cm) piece. Therefore, the velocity method indicates the distribution of molecular orientation in the film, and the velocity is a measure of the sonic modulus if boundary conditions are minimized.

Tensile Properties

The tensile properties of the films are listed in table IV and illustrated in Figure 3. The values in table IV are the average of 25 tests, and one standard deviation follows the ± sign.

The tensile properties of the PE film at 0°, 45°, and 90° are plotted in Figure 3(a). (Recall that the orientation increases slightly between 0° and 90° even though the film is nominally unoriented.) It can be seen from Figure 3(a) that, as the orientation increases, the tangent modulus and elongation also increase, the tensile strength and energy to break decrease, and the yield strength remains unchanged. The changes are fairly modest, however, being on the order of 10 percent except for the tangent modulus which increased nearly 20 percent. As would be expected, the tangent modulus had a much lower value than did the sonic modulus. Because they are measured at short times and small strains, dynamic moduli, such as the sonic modulus, generally are higher than the relatively static moduli of which the tangent modulus is one. Consequently, under high-velocity or impact loads the PE film may be twice as stiff as it would be under static loads.

The tensile properties of the PP film are shown in Figure 3(b). The changes are larger in this film than in the PE film, ranging from 50 to over 100 percent of the 0° values. In particular, the yield strength, tensile strength, and tangent modulus increase and the elongation and energy to break decrease with increasing orientation. Of importance in impact applications of the film is the sharp drop in energy to break. This property, which is the area under the stress-strain curve and primarily a function of tensile strength and elongation, is also a qualitative measure of the impact energy absorbing ability of the film. Consequently, although the tangent modulus and tensile strength increase with orientation, the impact strength may drop significantly.

The variation of tensile properties with angle in the plane of the PETP film is shown in Figure 3(c). Although the film was biaxially oriented, the sonic velocity generally increased between 0° and 90°, with a low value at 45° (Fig. 2). The tangent modulus shows a similar variation, the elongation varies in essentially the opposite sense, and the yield strength is virtually unaffected. The tensile strength also is unchanged except by the highest degree of orientation. The energy to break reaches a maximum at 45° where the sonic velocity and tangent modulus are a minimum. Although little variation of properties might be expected for this film, only the yield strength and energy to break varied less than 10 percent relative to the 0° value. The tensile strength, tangent modulus, and elongation varied by 25 to 35 percent. It is obvious that even the biaxially oriented PETP film is mechanically anisotropic.

In general, then, molecular orientation affects the tensile properties of the films in the following ways. The yield strength is nearly independent of orientation except for very high degrees of orientation, such as that found in the 90° direction of the PP film. By contrast, the tangent modulus is a direct function of the orientation and its value is one-half to one-third that of the sonic modulus. The tensile strength and elongation may increase or decrease with increasing orientation. For a given film, if one property increases the other will decrease. The energy to break varies inversely as the orientation. The energy absorbing ability of the films is highest where the degree of orientation is lowest. It is possible, by means of orientation, to obtain a film with a large variation in tensile properties such as that shown by the uniaxially oriented PP film. Although it was biaxially oriented, the PETP film was mechanically anisotropic. The most nearly isotropic film was the unoriented PE film.

Stress Relaxation Modulus

The relaxation modulus for times up to 1,000 seconds is plotted in Figure 4 where a logarithmic time axis is used. The values for the PE film are low, as would be expected for an unoriented film, and the 45° and 90° curves coincide. For the PP film, there is an increase in the relaxation modulus from 0° to 45° to a much higher value at 90°. The curves for the PETP film show the beneficial effect of biaxial orientation (only the data points and not the curves are shown for the 22.5° and 67.5° tests). The relaxation modulus is approximately the same, at a given time, at all angles except 90° where it is 25 to 30 percent higher.

Stress relaxation may be thought of as the change of recoverable elastic strain into irrecoverable plastic strain. In a polymer film the stress relaxation characteristics are influenced by, among other factors, the number and shape of the molecules which sustain the stress. Segments of the molecular chain, for example, can move or jump from one equilibrium position to another and thereby contribute to the irrecoverable plastic strain. If the curves in Figure 4 are normalized, then the PE film curves and most of the PETP film curves coincide. Such coincidence indicates that the difference in these curves is due primarily to the number and distribution of molecular chains rather than any shape or configuration which might have been given to the chains during orientation. By contrast, the PP film curves and the 90° PETP film curve not only have different absolute values but also have different shapes when they are normalized. The different shapes indicate that the chains have a slightly different configuration and therefore the relaxation mechanism is different. More chain segments are available to move and reduce the imposed stress in a chain which has a large number of kinks than in one which is more extended. Consequently, the relaxation modulus can be affected by both the molecular chain distribution and chain extension.

CONCLUDING REMARKS

The molecular orientation and the resulting mechanical anisotropy have been measured in three polymer films - unoriented polyethylene, uniaxially oriented polypropylene, and biaxially oriented poly(ethylene terephthalate). A sonic pulse method has been used to detect orientation and to measure the sonic modulus which varies as the orientation. It has been shown that, as the molecular orientation increases, the tangent modulus also increases, the energy to break (an estimate of the impact strength) decreases, and the yield strength is virtually unaffected. The stress relaxation modulus increases with increasing orientation, and the relaxation mechanism is altered by high degrees of orientation.

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TABLE I.- PROPERTIES AND CHARACTERISTICS OF POLYMER FILMS

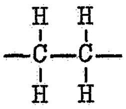
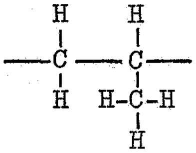
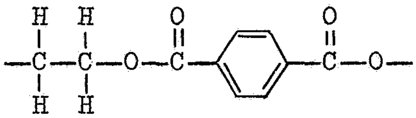
Name and abbreviation	Thickness, in.	Specific gravity	Molecular repeat unit
Polyethylene, PE	0.0013	0.950	 $-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{H}{C}}-$
Polypropylene, PP	0.0006	0.907	 $-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{H-\underset{H}{C}-H}{C}}-$
Poly(ethylene terephthalate), PETP	0.001	1.395	

TABLE II.- RELATIVE CRYSTALLINITY OF POLYMER FILMS AS DETERMINED BY X-RAY DIFFRACTOGRAMS AT THREE ANGLES IN THE PLANE OF THE FILM

Angle, deg	PE film		PP film		PETP film	
	Peak height	Ratio	Peak height	Ratio	Peak height	Ratio
0	1,620	1.00	2,020	1.00	7,630	1.00
45	1,800	1.11	1,940	.96	8,620	1.13
90	1,930	1.19	2,230	1.10	8,750	1.15

TABLE III.- SONIC VELOCITY AND SONIC MODULUS OF POLYMER FILMS AT DIFFERENT ANGLES IN THE PLANE OF THE FILM

Angle, deg	PE film		PP film		PETP film	
	Velocity, ft/sec	Sonic modulus, psi	Velocity, ft/sec	Sonic modulus, psi	Velocity, ft/sec	Sonic modulus, psi
0	3.79×10^3	1.55×10^5	6.94×10^3	4.96×10^5	8.22×10^3	1.07×10^6
10	3.88	1.63	6.85	4.82	8.46	1.13
20	3.79	1.55	6.80	4.70	8.98	1.27
30	3.70	1.48	6.62	4.49	9.25	1.36
40	3.70	1.48	6.94	4.94	9.57	1.45
45	3.79	1.55	7.20	5.31	4.93	3.80×10^5
50	3.70	1.46	7.58	5.88	1.02×10^4	1.65×10^6
60	3.85	1.58	8.34	7.08	1.14	2.07
70	4.08	1.78	9.25	8.79	1.10	1.91
80	4.43	2.09	1.01×10^4	1.04×10^6	1.08	1.84
90	4.49	2.17	1.08	1.19	1.10	1.91

TABLE IV.- TENSILE PROPERTIES[a] OF POLYMER FILMS AT DIFFERENT ANGLES IN THE PLANE OF THE FILM

Film and load direction	Yield strength, psi	Tensile strength, psi	Tangent modulus, psi	Elongation, percent	Energy to break, in/lb/in^3
PE-0°	710 ± 70	3,430 ± 400	$(0.78 \pm 0.25) \times 10^5$	633 ± 51	$(50.0 \pm 7.0) \times 10^3$
PE-45°	740 ± 90	3,140 ± 260	(0.76 ± 0.13)	653 ± 74	(46.3 ± 6.4)
PE-90°	800 ± 90	[b]2,660 ± 110	(0.89 ± 0.15)	[b]720 ± 32	[b](43.7 ± 4.4)
PP-0°	3,720 ± 160	8,680 ± 1,000	$(2.40 \pm 0.20) \times 10^5$	520 ± 63	$(104.8 \pm 1.6) \times 10^3$
PP-45°	3,680 ± 160	12,000 ± 720	(2.60 ± 0.25)	327 ± 43	(92.6 ± 1.5)
PP-90°	8,800 ± 320	23,600 ± 1,880	(4.09 ± 0.30)	110 ± 27	(55.4 ± 2.0)
PETP-0°	10,600 ± 800	24,600 ± 3,000	$(5.45 \pm 0.38) \times 10^5$	138 ± 29	$(79.7 \pm 21.5) \times 10^3$
PETP-22.5°	11,000 ± 600	24,600 ± 2,800	(5.46 ± 0.32)	150 ± 27	(86.0 ± 19.1)
PETP-45°	10,800 ± 1,000	24,600 ± 2,200	(5.15 ± 0.45)	157 ± 64	(88.0 ± 15.0)
PETP-67.5°	11,000 ± 600	32,600 ± 2,600	(6.32 ± 0.24)	112 ± 17	(82.9 ± 17.1)
PETP-90°	11,600 ± 800	34,000 ± 2,400	(6.89 ± 0.61)	92 ± 13	(71.7 ± 12.4)

[a] Each value is the average of 25 tests unless otherwise specified; one standard deviation follows the ± sign.

[b] Average of 15 tests.

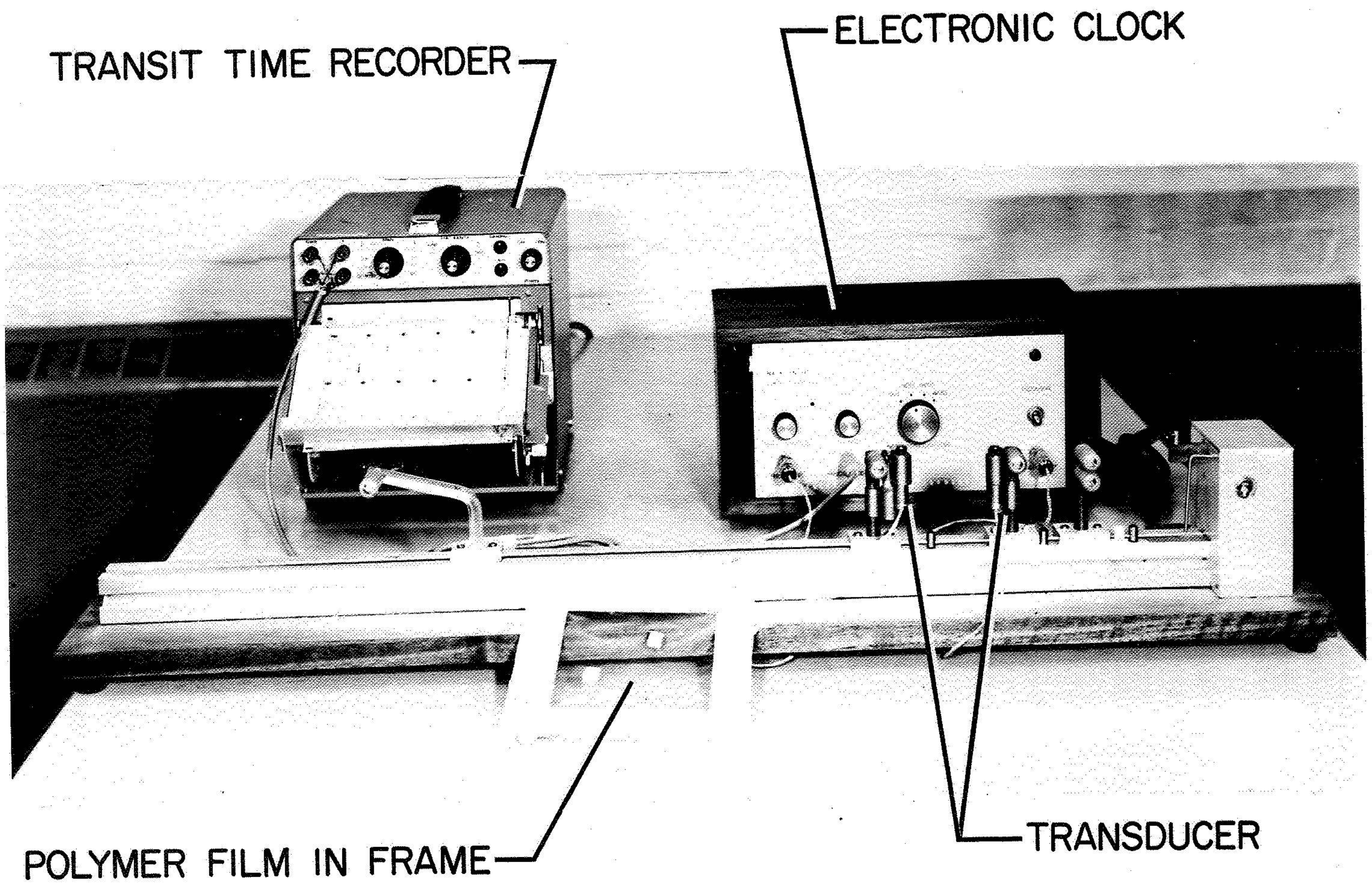


Figure 1.- Sonic pulse propagation meter.

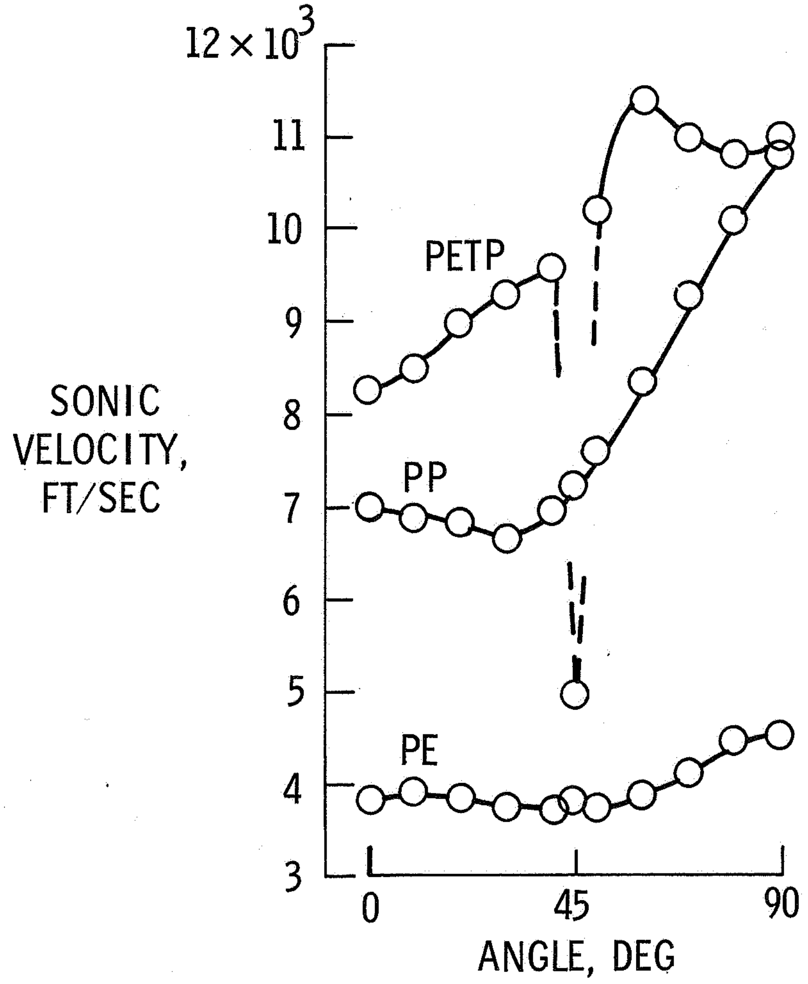
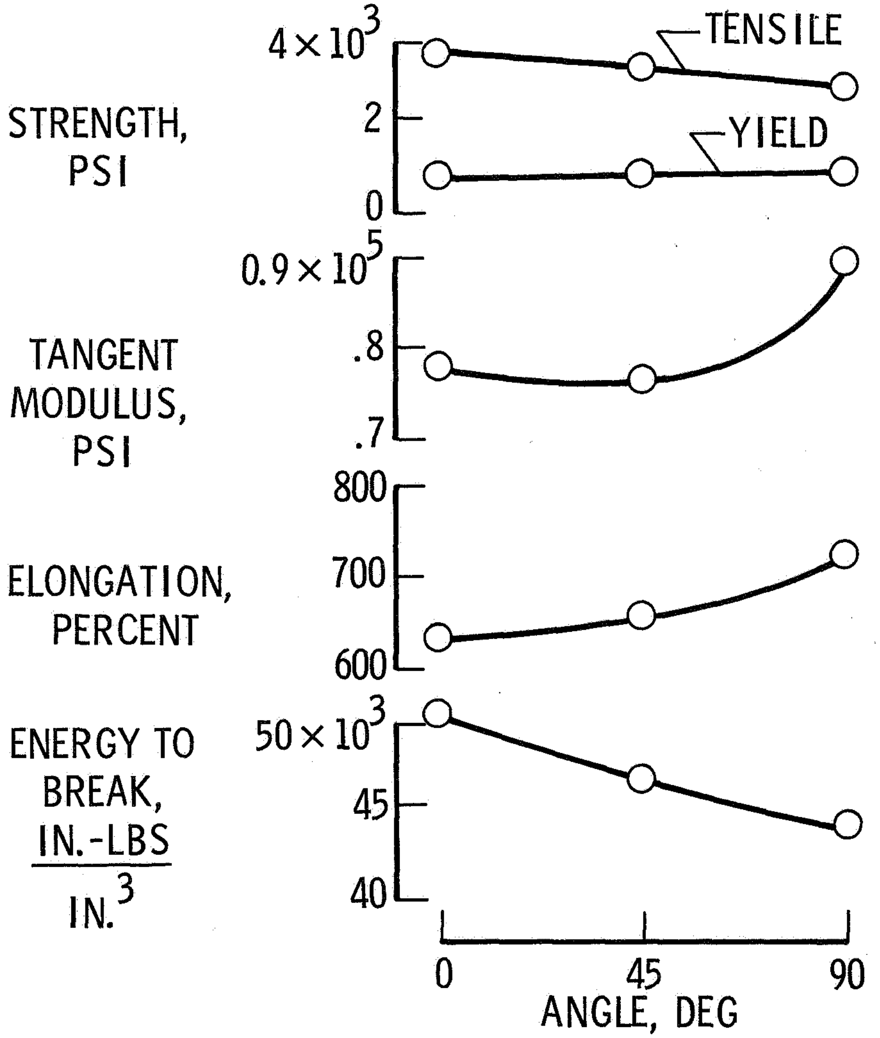
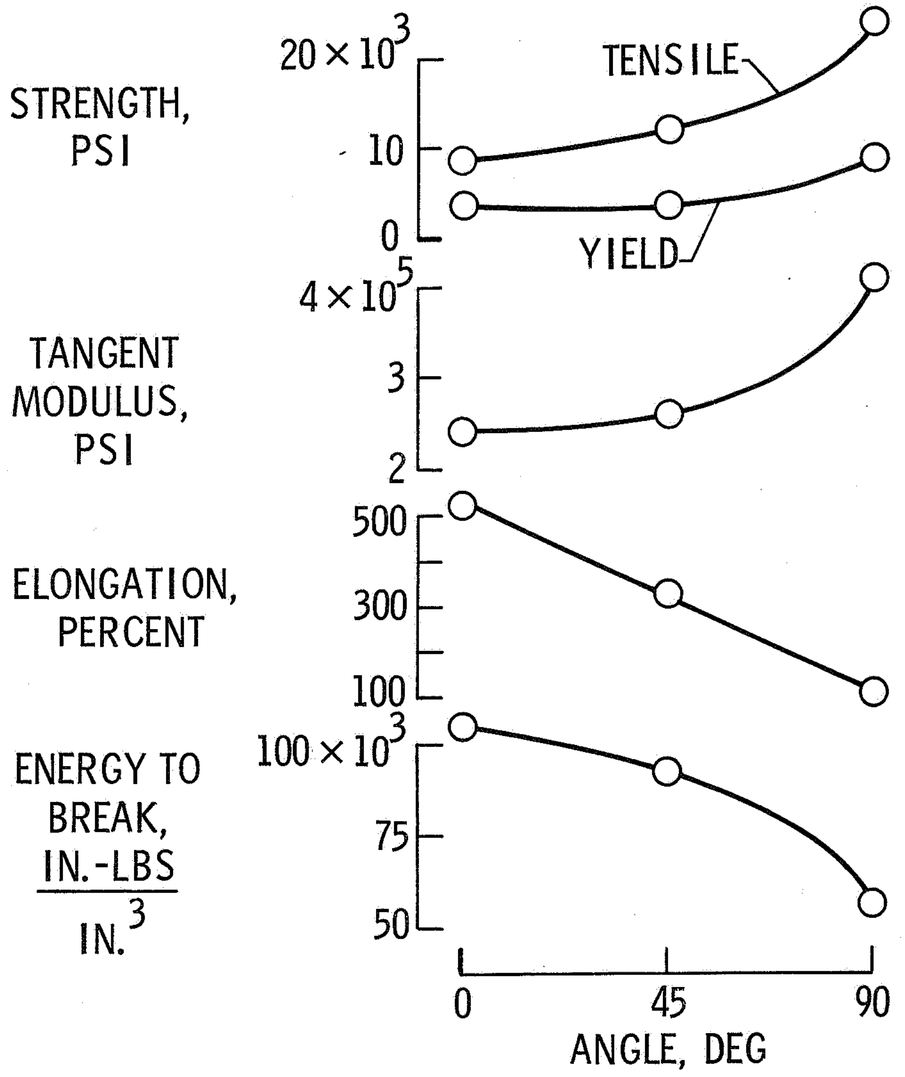


Figure 2.- Sonic velocity in polymer films as a function of direction in the plane of the film. The abbreviations are PE-polyethylene, PP-polypropylene, and PETP-poly(ethylene terephthalate).



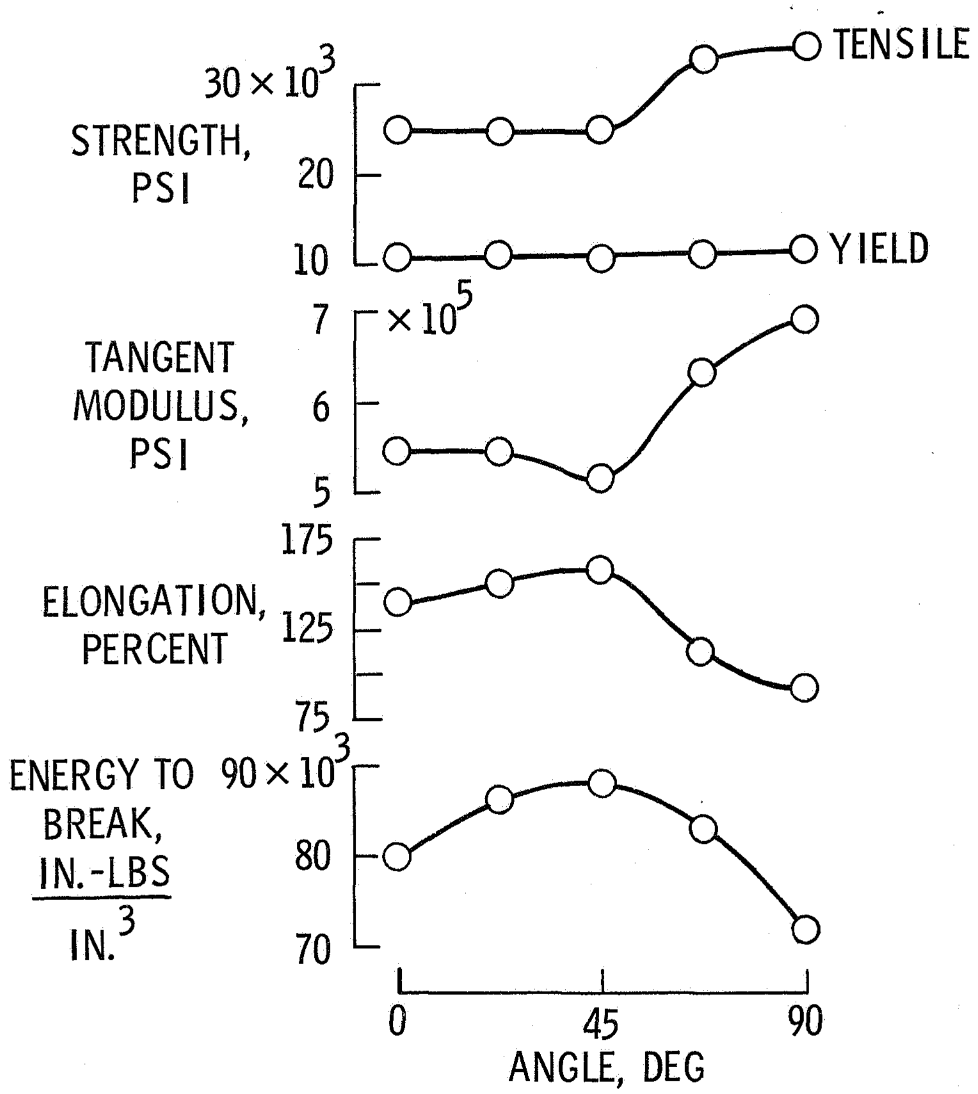
(a) Polyethylene film.

Figure 3.- Tensile properties of polymer films as a function of direction in the plane of the film.



(b) Polypropylene film.

Figure 3.- Continued.



(c) Poly(ethylene terephthalate) film.

Figure 3.- Concluded.

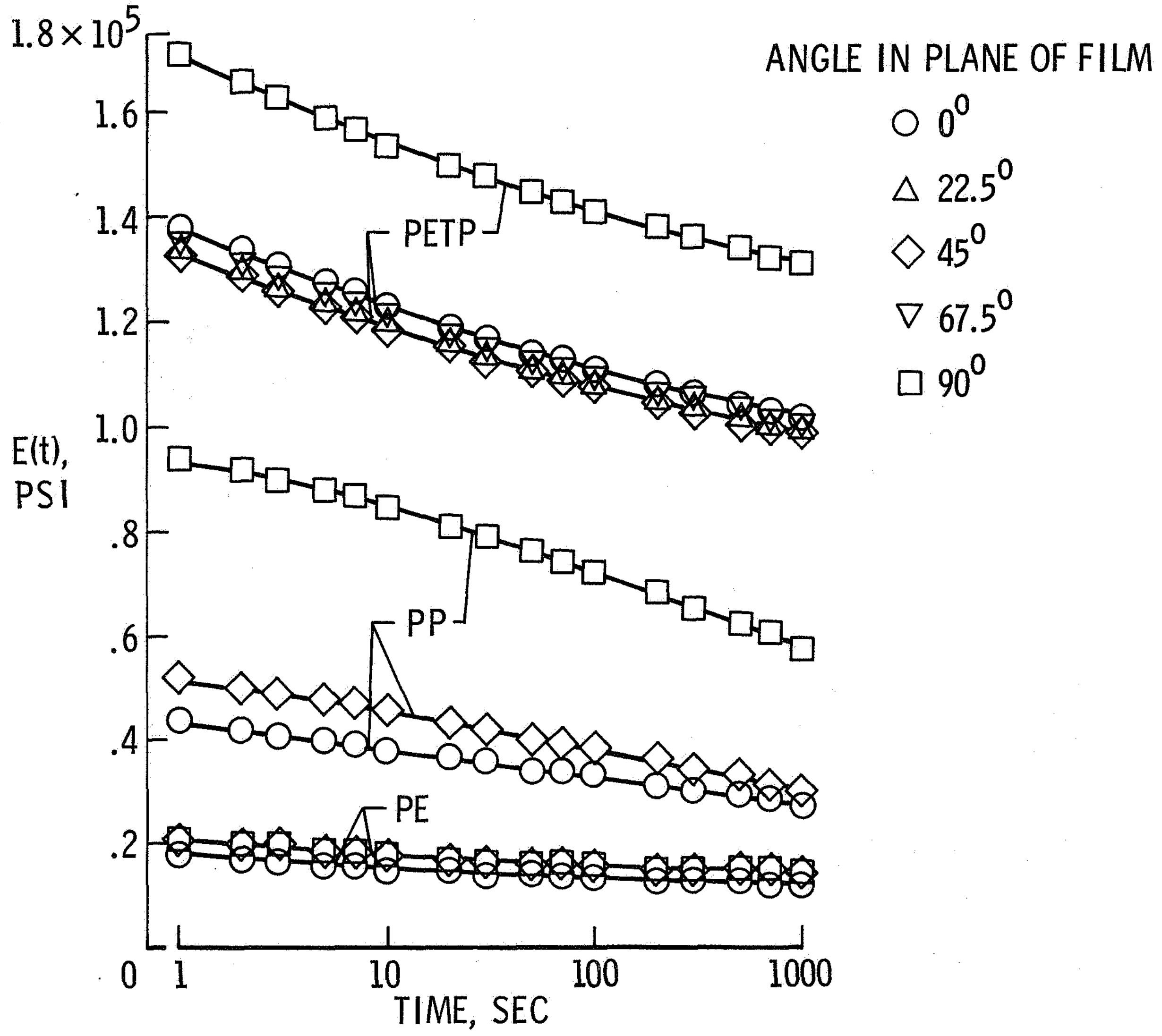


Figure 4.- Tensile stress relaxation modulus of polymer films as a function of time and of direction in the plane of the film. The abbreviations are PE-polyethylene, PP-polypropylene, and PETP-poly(ethylene terephthalate). Data points only are shown for the 22.5° and 67.5° relaxation modulus of the PETP film.

NASA-Langley, 1967