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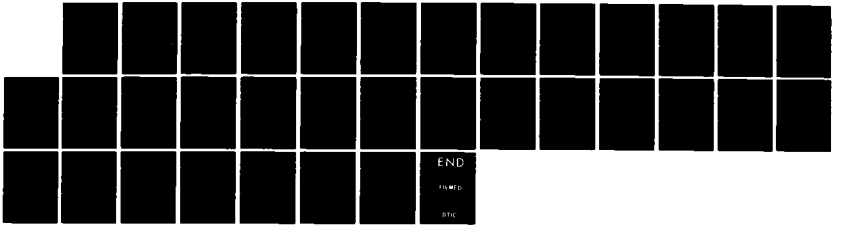

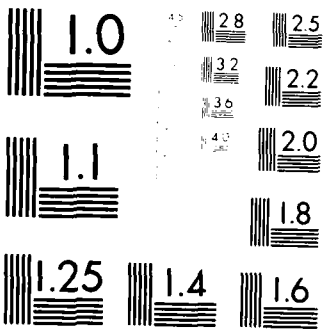

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Technical Report No. 36

PLASTIC DEFORMATION OF CRYSTALLINE POLYMERS

by

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Draw ratios have been measured for samples of polyethylene and trans-polyisoprene, crystallized at various temperatures and at various degrees of orientation. Th values obtained range from unity, i.e., no drawing is observed, up to values of about 15X for materials crystallized in the oriented state and t en drawn in a perpendicular direction. The results are in rough accord ith a simple molecular network model in

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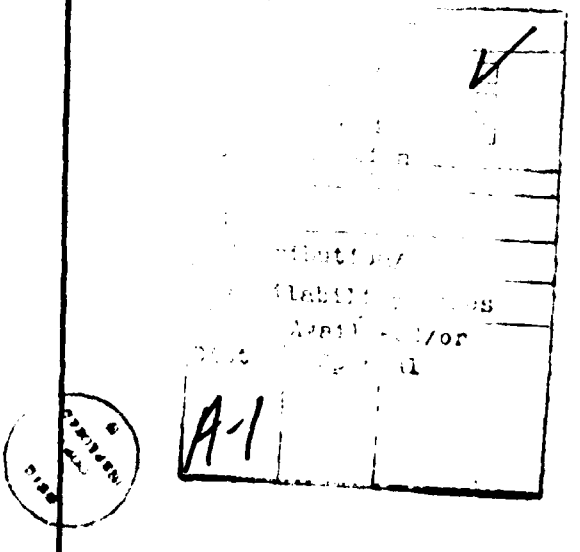
which network strands are incorporated into crystallites with a number of reversals of direction (folds), and the remainder of a strand between network junctions is randomly arranged. The reduction in draw ratio with increasing temperature of crystallization and with increasing orientation at the time of crystallization is then accounted for in terms of a reduction in the number of reversals (folds) per molecular strand. Differences in natural draw ratio for different polymers are attributed to variations in characteristic sequence length within a crystallite and in the number of folds per network strand.



1. Introduction

One of the most remarkable features of partially-crystalline polymers is their ability to undergo large plastic deformations ("drawing") without rupture. This phenomenon is readily distinguished from plastic yielding in ductile metals, which is considerably smaller in extent, or in glassy polymers, where yielding is localized within narrow shear bands. In contrast, plastic yielding in partially-crystalline polymers takes place in a relatively homogeneous way and results in deformations of several hundred percent without rupture(1). The material then strain hardens after drawing so that further deformation is only achieved by imposing higher stresses.

Representative relations between tensile stress and displacement of the clamps securing a tensile specimen are shown in Figures 1 and 2. After the yield stress σ_y is reached the specimen spontaneously thins over part of its length to form a "neck" where the extension is large. The rest of the specimen is still lightly stretched. Further displacement of the clamps is achieved by an increase in the amount of material in the neck at the expense of the lightly-strained material on either side of it. This transformation continues at a constant stress, generally smaller than σ_y, until the whole specimen has become uniformly stretched to the "natural draw ratio", i.e., the stretch ratio set up in the neck, denoted here d.

The natural draw ratio, as discussed in more detail later, is quite different for different polymers. For example, low-density polyethylene (LDPE) can only be drawn to about 5 times its original length whereas high-density polyethylene (HDPE) can be drawn 10X - 12X. Moreover, the draw ratio depends to a considerable degree upon the conditions under which crystallization took place, being much smaller for polymers crystallized in an oriented state.

Although considerable attention has been given to the molecuar structure of semi-crystalline polymers and the rearrangements that accompany plastic yielding and drawing (2,3), relatively little work has been published on quantitative aspects of the drawing process. Some preliminary observations are therefore reported here of the extent of plastic deformation that various crystalline polymers will undergo, and of the effect of the mode of crystallization upon their subsequent deformability. An attempt is then made to account for the marked differences observed in deformability between different polymers, and between different crystallization conditions for the same polymer, in terms of a simple model of the drawing process.

2. Experimental Details

(i) Materials

Samples were prepared in the form of molded sheets about 2 mm thick from several materials: trans-polyisoprene (TPI), supplied by Polysar Limited, denoted TP-301; high-density polyethylene (HDPE-1), supplied by Asahi-Kasei Industries, denoted Microsuntec R340P; a second sample of molding-grade high-density polyethylene (HDPE-2) supplied by Union Carbide Corporation, denoted 8908; a sample of pipe-grade medium-density polyethylene (MDPE) supplied by Union Carbide Corporation, denoted E608; and a sample of film-grade low-density polyethylene (LDPE), also supplied by Union Carbide Corporation and denoted DFDY 0774. Trans-polyisoprene (TPI) was also examined in the lightly-crosslinked state, brought about by adding 1 percent by weight of dicumyl peroxide and 1 percent of an antioxidant (Antioxidant 2246, American Cyanamid Company) to the material before hot-pressing for 1 h at 150°C. The crosslinked material is denoted TPI-X.

Samples of HDPE-1 and TPI-X were crystallized in the oriented state by melting the molded sheets at 160°C and 95°C, respectively, for 15 minutes and then rapidly stretching them to a predetermined length. They were then cooled rapidly in the stretched state so that crystallization

took place completely before release. The exact state of orientation was determined from the dimensions of an inked grid applied to the molded sheet initially.

Measured values of the density $\underline{d}$ of the crystallized sheets and values of the degree of crystallinity $\underline{C}$ calculated from them are given in Table 1. No significant changes in $\underline{d}$ and hence $\underline{C}$ were observed with the extent of orientation imposed during crystallization.

(ii) Determination of draw ratio

When a specimen deforms by cold drawing, the extension becomes highly non-uniform until the whole sample has been transformed into the drawn state. Further deformation then occurs homogeneously under increasing stress up to the point of rupture. The natural draw ratio is that set up in the necked region before the onset of strain hardening and subsequent uniform deformation. It was measured by the separation of grid lines placed on the sample initially. The measurements were made while the samples were under tension and in the process of being drawn.

Typical tensile stress-strain relations are shown in Figures 1 and 2 for oriented test pieces of HDPE-1 and TPI-X, stretched at a nominal strain rate of $0.05\ s^{-1}$ at $25^{o}C$. When the stretching direction was at right angles to the direction of prior orientation, represented by an extension

ratio λ, then the corresponding value of extension ratio in the direction of extension, given by $\lambda^{-\frac{1}{2}}$, was less then unity. Values of the extension ratio in the direction of subsequent stretching have been employed in Figures 1 and 2 to characterize the degree of prior orientation imposed during crystallization.

3. Experimental Results

(i) Stress-strain relations

Necking and drawing took place when the prior orientation ratio λ was less than about 8 for HDPE-1 and less than about 2 for TPI-X, Figures 1 and 2. For greater amounts of prior orientation a distinctly different type of deformation occurred. No signs of necking were observed and, instead of a pronounced drop in tensile load at the yield point, only a change in slope of the load-displacement relation was noted, Figures 1 and 2.

Measured values of the yield stress σ_Y are plotted in Figure 3 against the pre-orientation ratio λ. For HDPE-1 they did not change significantly over the entire range of orientation, being about 24 MPa, and for TPI-X only a slight increase was found, from about 9 to 14 MPa, as the degree of prior orientation increased. The difference in σ_Y between the two

polymers may be due largely to the different levels of crystallinity; 65 percent and 36 percent, respectively. Changes in crystallite orientation or microstructure brought about by prior orientation appear to have little effect on the yield stress.

(ii) Draw ratio: Unoriented samples

Values of the natural draw ratio λ_d for unoriented samples of all of the materials examined are given in Table 1. They range from 2.6 for TPI-X to 12 for HDPE-2. Surprisingly, they are consistently larger for the more highly-crystalline materials, whereas one might well have expected the reverse: a greater ductility for less-crystalline materials. Moreover, the largest values are somewhat larger than one would expect for the maximum extensibility of polymer chains in a loose molecular network: rubbery materials will undergo extensions of only up to about 10X at the most, before rupture. As the present materials appear to recover completely on melting, there is no indication of molecular flow during drawing. The molecules thus appear to be retained within a network of entanglements as if they were lightly crosslinked. The high extensibility of HDPE-2 during drawing is therefore quite

surprising, particularly in view of its high degree of crystallinity. A possible model of the drawing process that accounts for these features is advanced in a later section.

(iii) Draw ratio: effect of prior orientation

Values of the draw ratio λ_d for oriented samples of TPI-X and HDPE-1 are plotted in Figure 4 against the prior orientation ratio $\frac{1}{\lambda}$, using logarithmic scales for both axes. As the degree of prior orientation was increased, so the draw ratio decreased, becoming unity; i.e., no drawing took place; when the prior orientation ratio was about 2.4 for TPI-X and about 10 for HDPE-1. On the other hand, when extension was imposed in a direction perpendicular to the prior orientation, i.e., $\frac{1}{\lambda} < 1$, then the draw ratio increased, reaching values of about 4 for TPI-X and about 14 for HDPE-1.

The variation of λ_d with prior orientation ratio $\frac{1}{\lambda}$ was found to be approximately an inverse proportionality: $\lambda_d = \text{constant}/\frac{1}{\lambda}$. The broken lines in Figure 4 are drawn with a slope of -1, consistent with this relationship. They are seen to describe the experimental results reasonably well, except in the low-orientation region when $\frac{1}{\lambda}$ is close to unity. A dependence of this form would arise from the limited extensibility of polymer molecules, because prior orientation by a factor $\frac{1}{\lambda}$ would reduce the additional extension ratio λ_d that molecular chains could undergo

before reaching the fully-stretched state, by the factor λ^{-1}. A treatment of the drawing process in terms of the maximum possible extensibility of polymer molecules is given in the following section.

4. Theoretical Considerations

A simple molecular model of the process of cold drawing in partially-crystalline polymers is now put forward. A representative molecular chain between attachment points to the network, i.e., between molecular entanglements or crosslinks, is shown in Figure 5. It consists of n freely-jointed units, each of length a, so that its fully-stretched length L_m is given by na and its mean end-to-end length L_o in the molten state, Figure 5a, is given by $n^{\frac{1}{2}}a$. On crystallization the center part of the molecule is assumed to enter a crystallite of length L_c and fold back on itself to enter the same crystallite a number of times, denoted f, Figure 5b. In order for the ends of the molecule, where it is joined to other molecules in the network, to be located on either side of the crystallite, only odd values of f are considered here. In this simple model the degree of crystallization C is given by

$$C = fL_c/L_m \tag{1}$$

It is now assumed that drawing takes place when the molecular chains entering a crystallite apply a sufficiently high stress to it so that it becomes disrupted and transforms into a new extended form, Figure 5c. A further assumption is made that the requisite stress is relatively high so that the molecules are almost fully extended at the onset of crystal rearrangement, and subsequently. Thus, the natural draw ratio λ_d is given by

$$\lambda_d = L_\pi / L, \qquad (2)$$

where L is the end-to-end distance for a representative chain in the unoriented partially-crystalline state, Figure 5b. This is not necessarily the same as the corresponding distance L_o in the molten state, Figure 5a. Indeed, it is hypothesized that the process of crystallization will alter the distribution of chain end-to-end distances, $L_o \rightarrow L$, by excluding junction points from the crystal lattice. In particular, those chains which become fully-extended first on stretching and thus initiate the disruption of crystallites will be those with junction points disposed in the direction of stretching and on either side of a crystallite, as shown schematically in Figure 5b.

An estimate of the initial junction separation distance L can be made as follows. The distance L is composed of a

crystalline sequence length L_c and an amorphous distance L_a, given approximately by the random-coil value $L_a = (1-C)^{\frac{1}{2}} n^{\frac{1}{2}} a$.

Hence,

$$L = L_c + L_a = (CL_m/f) + (1-C)^{\frac{1}{2}} L_m/n^{\frac{1}{2}}, \qquad (3)$$

on substituting for L_c in terms of L_m from equation (1). Thus, the natural draw ratio λ_d is given by

$$1/\lambda_d = (C/f) + (1-C)^{\frac{1}{2}}/n^{\frac{1}{2}}, \qquad (4)$$

from equations 2 and 3.

This simple treatment suggests that the natural draw ratio will be large for materials in which the molecules re-enter the crystallites many times and f is large, even when the degree of crystallization is high. In other circumstances, the predicted draw ratio is small. Two extreme cases are now considered. In each case the number n of random links per molecular strand is given the representative value, 200.

For TPI-X, the degree of crystallinity C is 36 percent and the natural draw ratio is about 2.6, Table 1. From equation 4, the average number of times f that a chain passes through a crystallite is obtained as 1.1, indicating that for this material there is relatively little re-entry or chain-folding. For HDPE-2, on the other hand, the degree of crystallinity is 72 percent and the natural draw ratio is about 12. From equation 4 the average number of times f

that a molecular chain passes through a single crystallite is obtained as 15.6, indicating that there is a great deal of re-entry or folded-chain crystallization in HDPE-2, as prepared here.

From the number of random links involved in crystalline sequences, 72 for TPI-X and 144 for HDPE-2, and the inferred number of times that each chain enters the same crystallite, we may deduce the mean length $\underline{L_c}$ of a crystallite to be 65 random links for TPI-X and 9.2 random links for HDPE-2. The latter number is consistent with the known crystal microstructure of polyethylene, corresponding to about 5-10 nm, but the value for TPI-X seems unacceptably high. It should be noted, however, that the present analysis does not distinguish between a chain passing through a single crystalline sequence in the direction of stretching or a chain passing through two or more crystalline sequences, without reversing direction, before a junction point is reached. Thus, the large number of random links deduced for the crystalline sequence length in TPI-X may in reality be the sum of several crystallite lengths traversed by the same molecular strand. Nevertheless, a clear implication of the present analysis is that chain-folding is much less prominent in TPI-X and that the crystallites are considerably longer (thicker) than in HDPE. It would be interesting and worthwhile to examine other partially-

crystalline polymers and to make direct comparison with their crystallite thicknesses, determined, for example, by SAXS.

The large effect of prior orientation upon the draw ratio λ_d, discussed previously and shown in Figure 4, can be interpreted in terms of the molecular model advanced here. It is in accord with a continuous change in the chain end -to-end length L with prior orientation, as might well be expected, with a corresponding reduction in the number of times that a molecular strand traverses a single crystallite.

5. Effect of Annealing or Quenching

A direct implication of the analysis given here is that an increase in crystallite thickness, brought about by annealing, for example, should result in fewer re-entries and a decrease in the natural draw ratio. Conversely, a decrease in crystallite thickness and corresponding increase in number of molecular re-entries should cause an increase in natural draw ratio. Unoriented samples of HDPE-1 and TPI-X were therefore prepared by melting sheets and allowing them to crystallize at different temperatures so that the crystallite layer thicknesses would be somewhat different. Values of the natural draw ratio are given in Table 2. As can be seen, the higher the temperature T_c of crystallization (and, hence, the thicker the crystallite), the lower is the natural draw ratio λ_d, in general. This trend is in accord with the mechanism of cold drawing put forward here.

6. Conclusions

Measurements have been made of the tensile properties of several representative partially-crystalline polymers, crystallized both in the oriented and unoriented state. The yield stress was found to be virtually unchanged by prior orientation but the natural draw ratio decreased in inverse proportion to the amount of preorientation.

Striking differences were found between the natural draw ratio for various polymers. A simple molecular model for the drawing process was developed in terms of a loose network of molecules, held together by entanglements or crosslinks which are excluded from the crystal lattice. As a result, the end-to-end distance for molecular strands in the partially-crystalline material is different from that in the melt. Under tension, certain strands become fully-stretched and initiate disruption of the crystallites, followed by their rearrangement into the fully-drawn state. The principal term in the analysis appears to be the number of times that a molecule reverses direction and re-enters the same crystallite. When this is large, the natural draw ratio is large also.

This simple concept accounts successfully for the major features of cold drawing: large differences between different materials, a continuous decrease in the natural draw ratio with the extent of prior extension in the melt, and a decrease in the natural draw ratio on annealing.

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Table 1. Physical properties of the materials examined

Material	Density ρ (kg/m^3)	Crystallinity C (%)	Yield Stress σ_y (MPa)	Draw Ratio λ_d
TPI-X		36*	9.0	2.6
TPI		37*	9.0	2.7
LDPE		50*	10.5	5
MDPE	940	62**	21.5	7
HDPE-1		64*	24	8.6
HDPE-2	955	72**	29	12

* Measured by DSC with a heating rate of 0.17 C/s. Reported values for 100% crystallinity are: 44.5 cal/g for TPI (4) and 69 cal/g for polyethylene (5).

** C was calculated from the measured density using a relation given by Chiang and Flory (6).

Table 2. Natural draw ratio λ_d for quenched and annealed samples.

Crystallization temperature, T_c (^oC)	λ_d
TPI-X	
0	2.6
40	2.4
45	2.1
LDPE	
Quenched (T_c = 20-90 C)	4.8
100	5.3
MDPE	
Quenched (T_c = 20-100 C)	6.4
118	7.7
HDPE-1	
Quenched (T_c = 20-100 C)	8.6
115	7.0
121	3.5
HDPE-2	
Quenched (T_c = 20-100 C)	10.5
115	11.0
121	3.0

Figure Legends

Figure 1. Relations between tensile stress σ and apparent extension e for oriented samples of HDPE-1.

Figure 2. Relations between tensile stress σ and apparent extension e for oriented samples of TPI-X.

Figure 3. Dependence of yield stress σ_y upon the prior orientation extension ratio λ in the stretching direction, for HDPE-1, ●, and TPI-X, 0.

Figure 4. Dependence of natural draw ratio λ_d upon the prior orientation extension ratio λ in the stretching direction, for HDPE-1, ●, and TPI-X, 0. The broken lines are drawn with a slope of -1.

Figure 5. (a) Sketch of a molecular strand between tie points in the melt.

(b) In the crystalline state.

(c) In the drawn state.

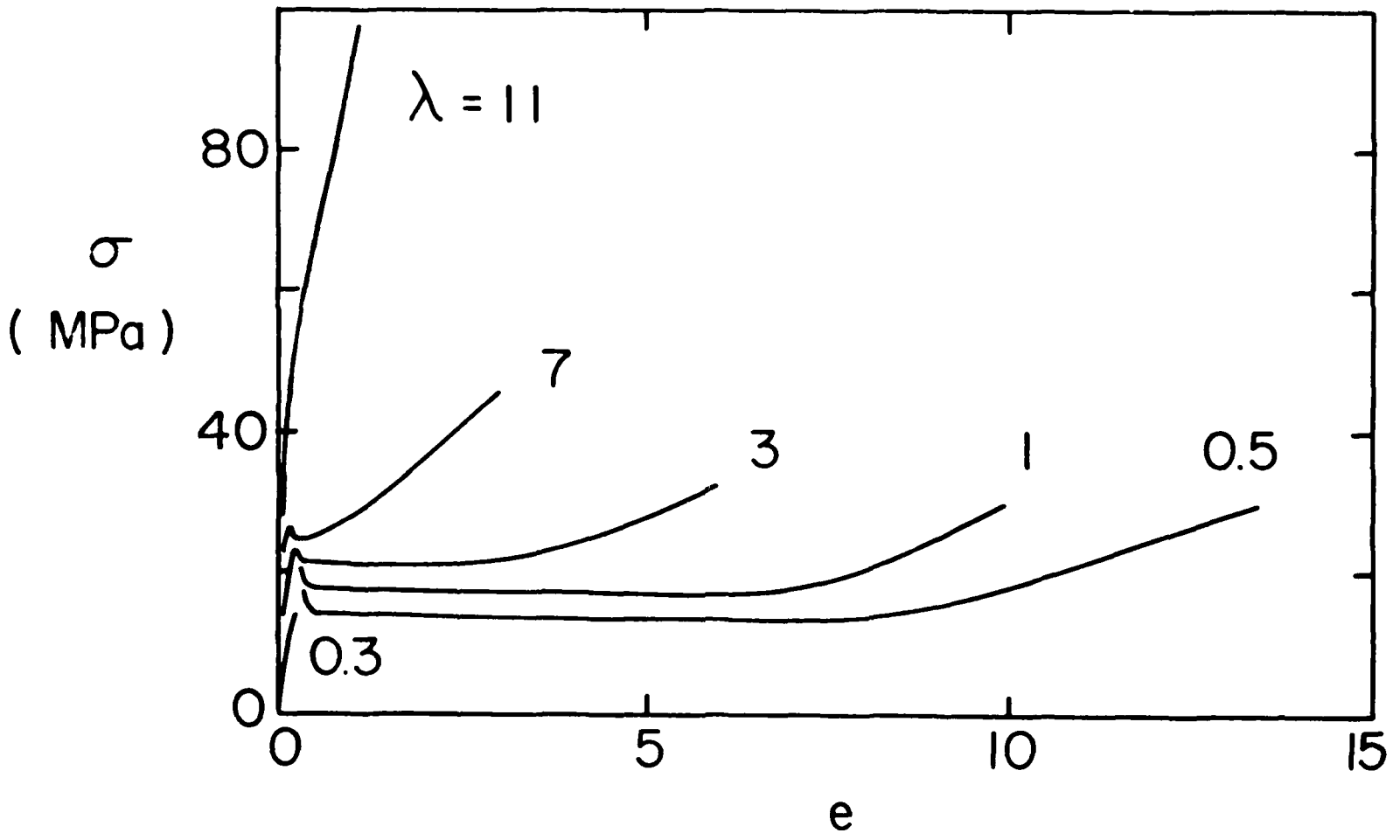

Figure 1

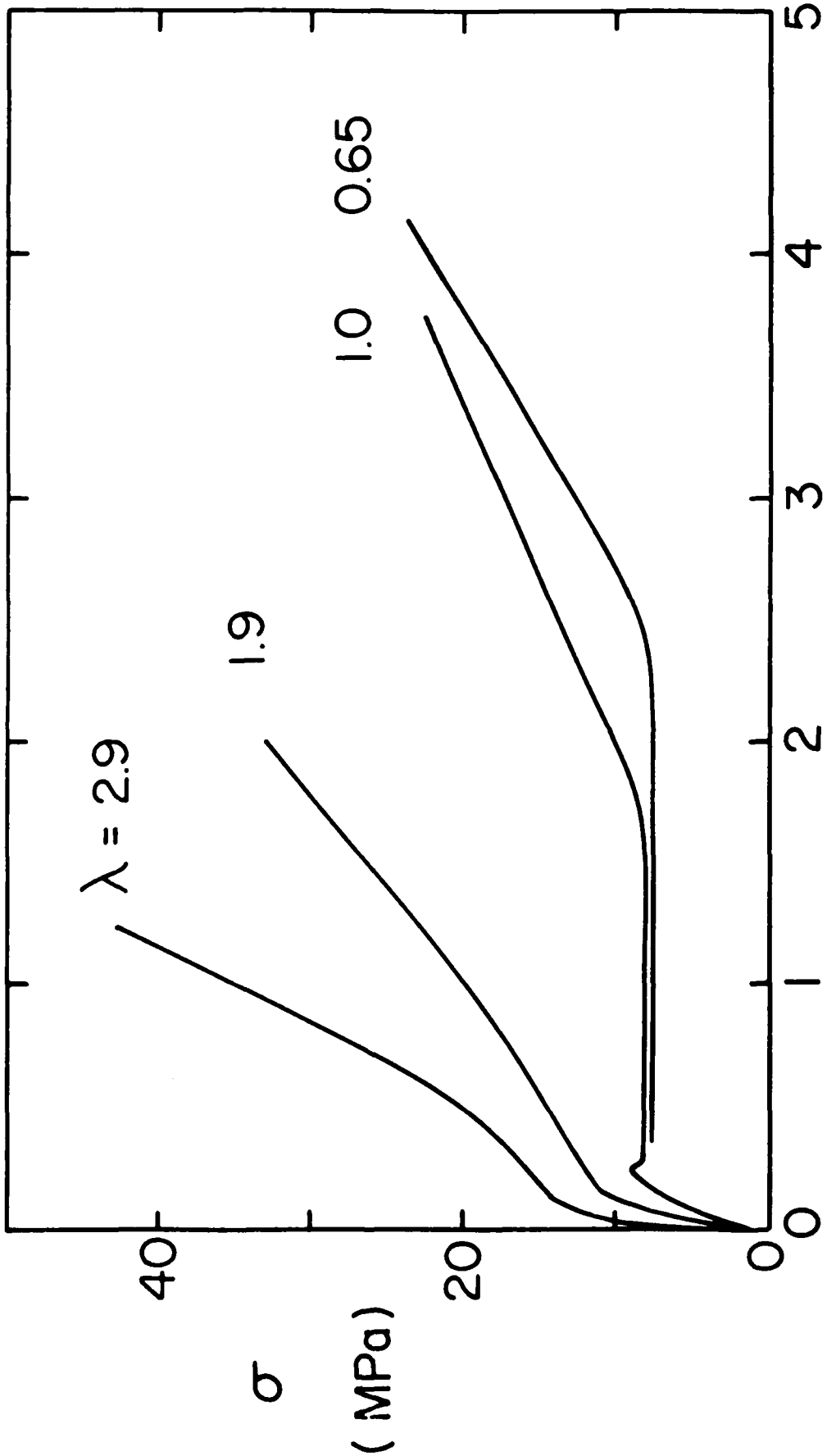

Figure 2

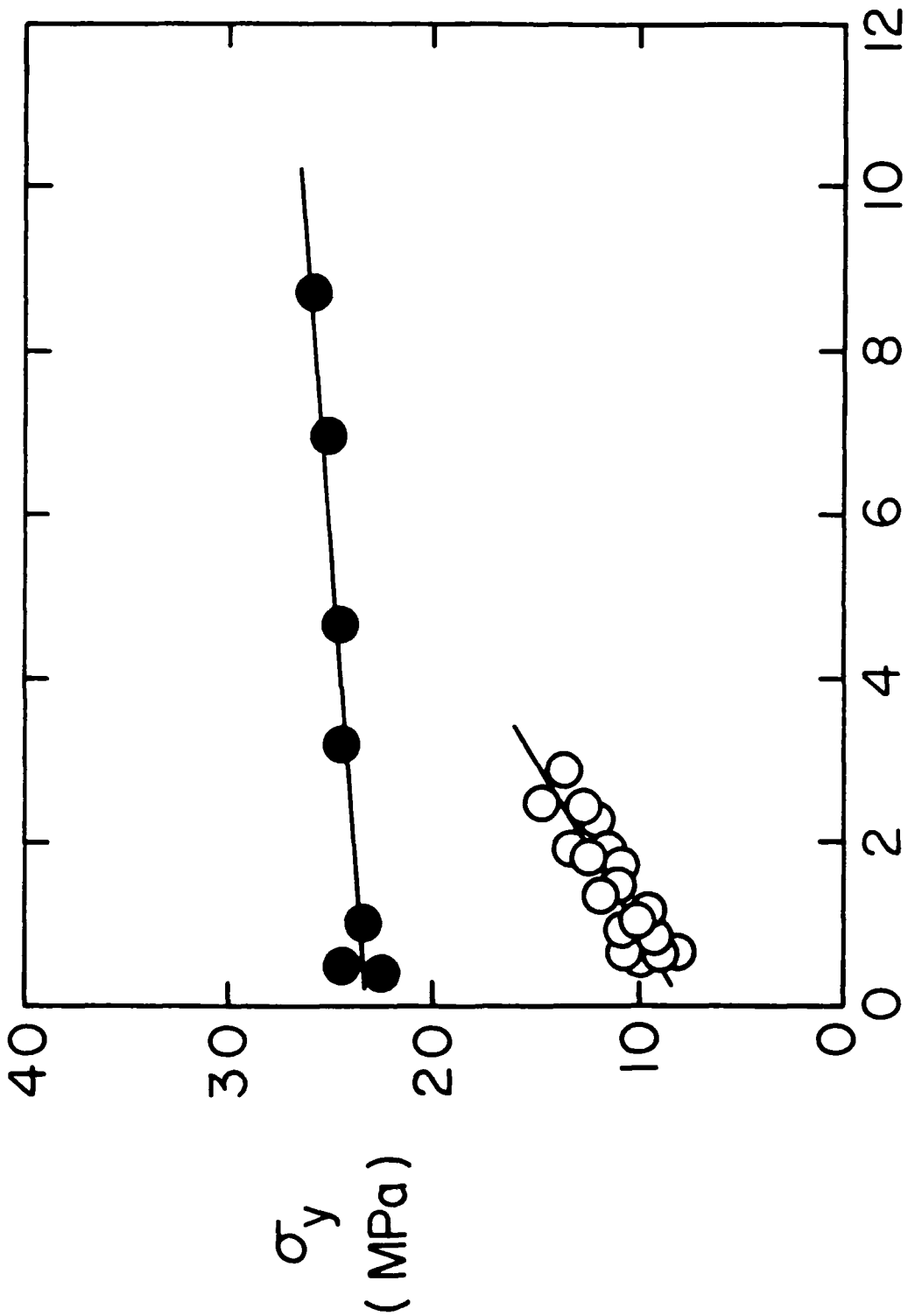

Figure 3

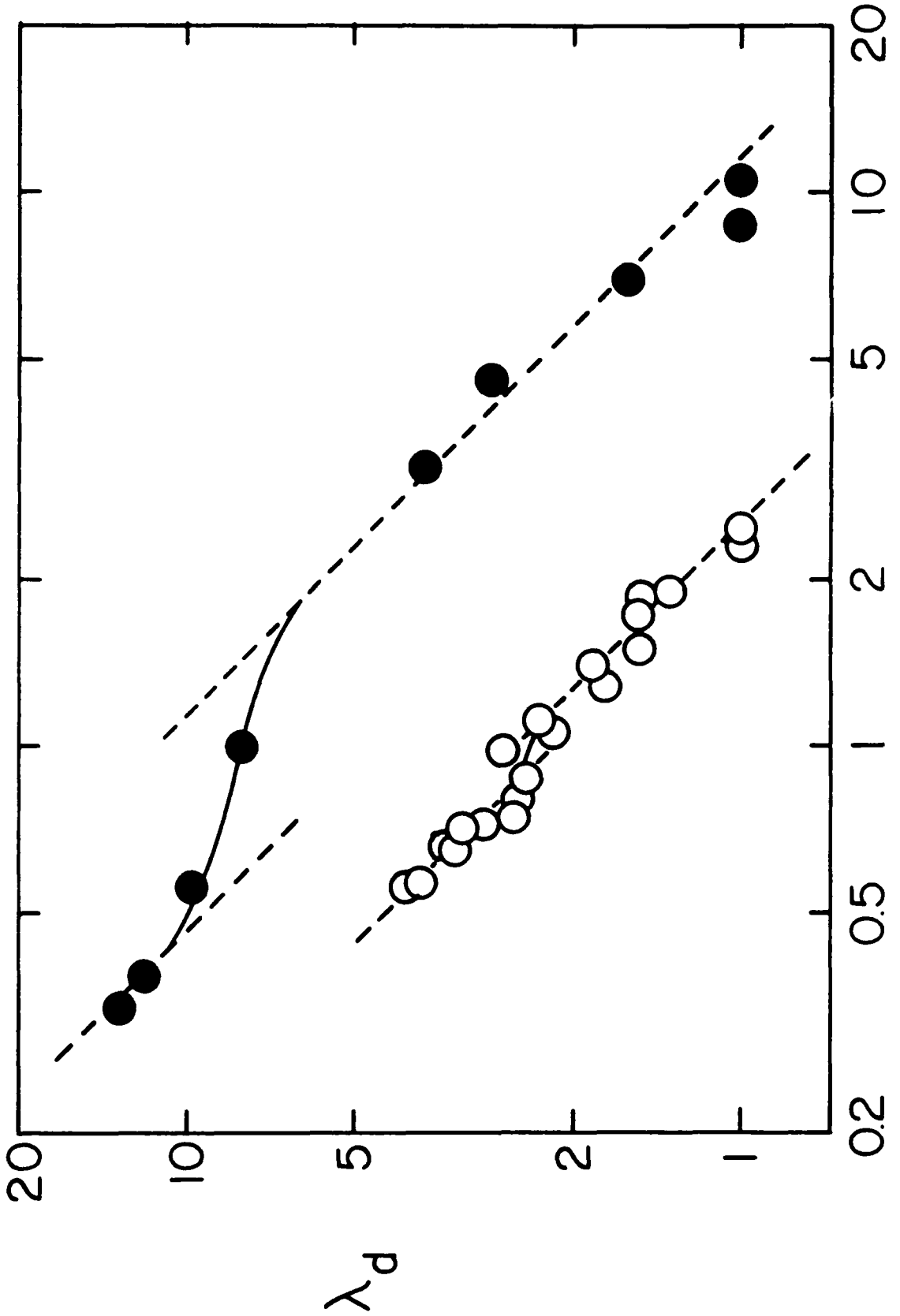

Figure 4

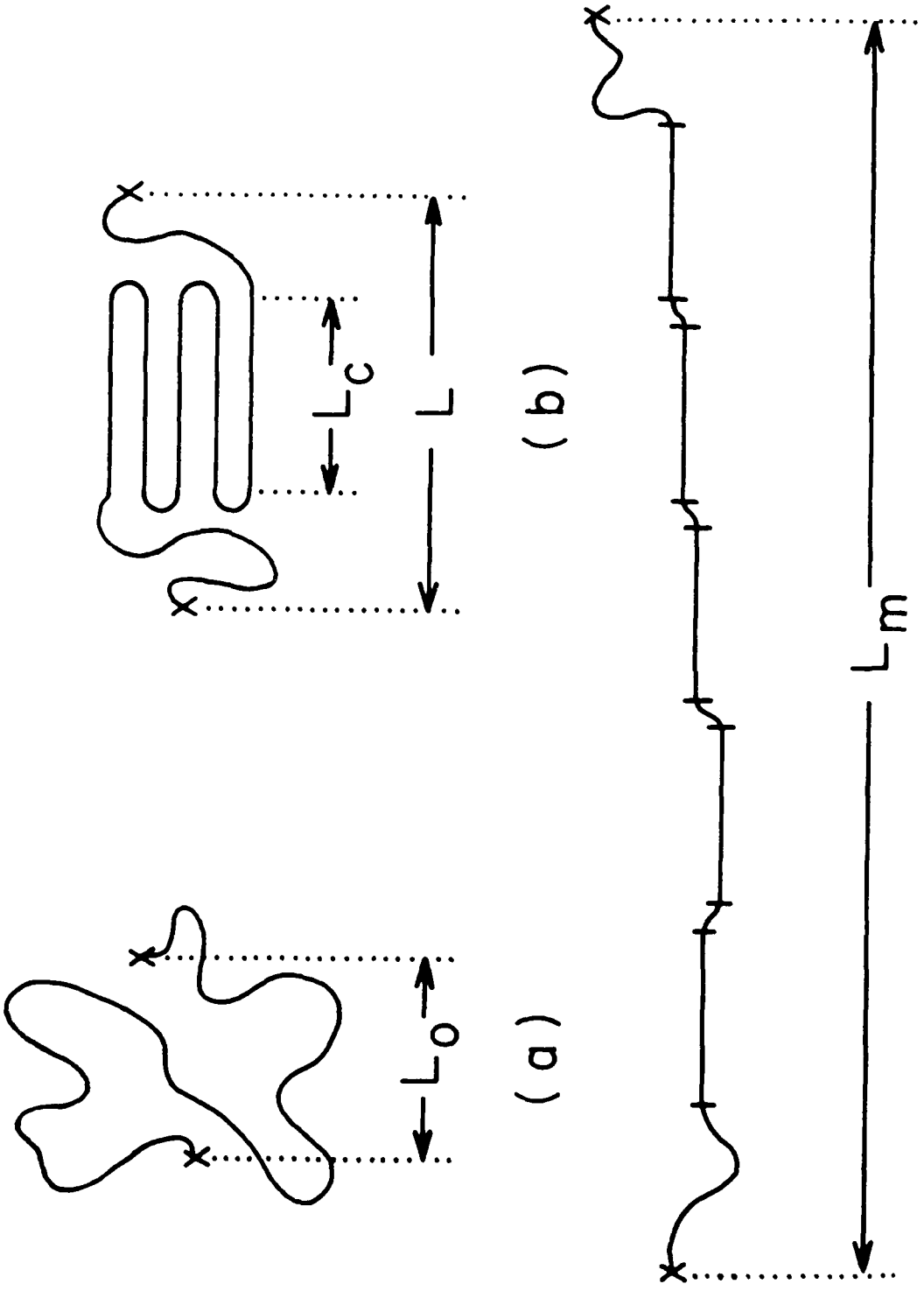

Figure 5

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