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SOLID STATE COEXTRUSION OF HIGH DENSITY POLYETHYLENE. I. EFFECT--ETC(U)

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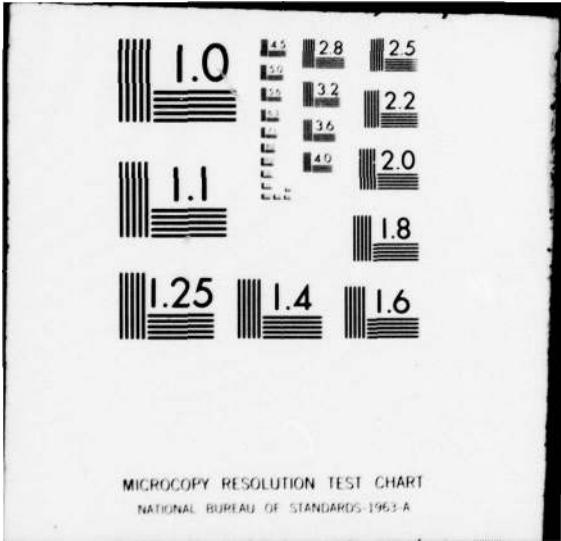
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SOLID STATE COEXTRUSION OF HIGH DENSITY POLYETHYLENE.
I, EFFECTS OF GEOMETRIC FACTORS,

by

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ABSTRACT

The effects of geometry and volume fraction on the solid (crystalline) state coextrusion of two concentric different high density polyethylenes (HDPE) having weight average molecular weights (M_w) 59,000 and 200,000 have been studied. The extrusion rate increased nonlinearly with the volume fraction of the low M_w component. The rate was faster when the low M_w component was the core rather than the sheath in the initial billet. Thus the slow extrusion rate of high M_w HDPE alone was increased up to ten times by coextrusion with a small fraction of the low M_w HDPE component in the middle of the high M_w HDPE billet. Generally, the deformation flow profile changed gradually from a parabolic to a W shape pattern as the volume fraction of the high M_w component increased. However, the geometric arrangement of the two different M_w components in the initial billet had also a pronounced effect on deformation. The deformation patterns showed that upon coextrusion the low and high M_w HDPE's were extruded at the same rate and extrusion draw ratio. Geometry had no substantial effects on the tensile modulus and strength of the extrudates; i.e., they increased linearly with volume fraction of the high M_w HDPE.

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INTRODUCTION

Recently, the improvement of mechanical properties of semicrystalline polymers has been the subject of intensive study by solid state deformation¹⁻⁵. In these studies, high density polyethylene (HDPE) has been the most extensively studied with the highest modulus value measured along the fiber axis at room temperature -70-80 GPa¹⁻³ which corresponds to one third to that of steel wire.

These ultraoriented, high modulus and strength extrudates have been utilized for the preparation of composites by combining them with unoriented materials having poor mechanical properties. Porter with Capiati⁶ and Mead⁷ have studied the preparation of polyethylene one polymer composites by epitaxially bonding ultraoriented high modulus polyethylene strands with unoriented low density polyethylene matrices.

We have earlier⁸ demonstrated that coextrusion of HDPE's of different molecular weight (M_w) is feasible, resulting in cocylindrical two phase extrudates. These extrudates consisting of core and sheath components of different M_w HDPE's had high tensile moduli, strength and showed considerable resistance to core/sheath separation even though nonbonded.

In this work, we discuss the results of coextrusion of different M_w HDPE's in which the two component phases are fused together and solid (crystalline) state extruded. The objective of the present study is multifold; firstly, to prepare coextrudates in a continuous process under conditions which will allow practically the most efficient extrusion draw in terms of enhanced tensile properties. Secondly, to increase the extrusion rate of high M_w polyethylene components via

solid state coextrusion; thirdly, to examine the geometrical arrangement of the different M_w polyethylene components on extrusion rate, the characteristic deformation modes and tensile properties. Finally, to examine the effects of M_w and molecular weight distribution on extrusion rate and on tensile properties of the extrudates. These results are presented in two parts. In the first, we discuss the effects of geometric arrangement of the HDPE's of different M_w on the extrusion rates and deformation flow patterns on solid (crystalline) state coextrusion. In the second part, we discuss the effects of molecular parameters on the extrusion rates and tensile properties of coextrudates.

EXPERIMENTAL

1. Preparation of Composite Billets:

Cocylindrical billets of different HDPE's (Alathon 7050, $M_w = 59,000$ and Marlex 6003, $M_w = 200,000$) were prepared in a specially designed apparatus⁹. Briefly, polymer pellets were packed in a press having a cylindrical bore of 0.95 cm. The press was subsequently evacuated and immersed in a silicone oil bath at 165°C for 15 min to completely melt the pellets. The crystallization was allowed to occur under cooling to ambient at a rate of -10 °C/min. A slight pressure was maintained during cooling to prevent formation of voids resulting from volume contraction during the crystallization process. The press was then disassembled and a molded billet 0.95 cm diameter and 7 cm length was removed. Sheath components were prepared from these billets by drilling out the central section, whereas the core was produced by turning down on a lathe a billet of the appropriate polyethylene to a diameter equal the inner diameter of the drilled out billet (Fig. 1). The two phase billets were prepared by inserting the core component into the sheath component and subsequently co-melting the two components in the press and cocrystallizing by the aforementioned method. Thus, molded composite billets were obtained and thereafter split longitudinally in two identical semiperipheral pieces, each piece having equal volume fractions of each component (Fig. 1) The flat surfaces of the semiperipheral pieces were sanded scrupulously and were ink imprinted with a 0.20 cm parallel line pattern in order to measure the extend of draw and observe the deformation mode on extrusion.

2. Solid (Crystalline) State Extrusion:

The semiperipheral pieces were reassembled and were press fitted into the reservoir of an Instron capillary rheometer maintained at the desired extrusion temperature. The billets were extruded through a conical brass die having an included entrance angle of 20° , capillary part of 1 cm and nominal extrusion draw ratio (EDR) 25. The extrusion pressure (P_{ext}) was 0.23 GPa unless is otherwise stated.

3. Mechanical Tests:

The efficiency of draw was evaluated by the mechanical properties of the co-extrudates. Tensile modulus and strength along the fiber axis were measured at room temperature on an Instron testing instrument, Model TTM. An Instron strain gage extensometer (10 mm) was used for the tensile tests. The modulus was determined at a strain rate of $5.0 \times 10^{-5} \text{ sec}^{-1}$ from the tangent to the stress-strain curve at 0.1% strain. The tensile strength was determined at a strain rate $1.5 \times 10^{-3} \text{ sec}^{-1}$.

RESULTS AND DISCUSSION

The structural reorganization that occurs during the crystalline state extrusion depends primarily on the extent of draw which is usually expressed by the EDR, i.e. the ratio of the inlet to outlet cross-sectional area of the extrusion die. No die swelling was observed during the present extrusion and the EDR based on the above definition was in an excellent agreement with that evaluated from changes in deformation patterns.

Another factor which may affect the structure and properties of ultra-oriented semicrystalline polymers is annealing effects which may occur during and after extrusion.

Porter et al.¹⁰ suggested that a partial reorganization might be possible for ultraoriented HDPE extrudates even at a temperature as low as 100°C when they were heated under atmospheric pressure. They also found that laterally imposed constraint remarkably suppressed the reorganization¹¹. In the present study, extrudates upon extrusion were kept under considerable constraint at the capillary part (1 cm) of the conical die while they were hot. Thus, extensive annealing may be prevented for the extrusion at 120°C as will be discussed further in the companion paper¹². The effects of weight average molecular weight (M_w) and molecular weight distribution for a series of HDPE's were examined at the commonly achievable highest EDR since the effects of these parameters became apparent with highly drawn samples and is also discussed in the companion paper¹². From these considerations, we performed the extrusion at $T_{ext} = 120^\circ\text{C}$, pressure $P_{ext} = 0.23$ GPa and through a die having capillary part 1 cm and EDR 25.

The extrusion behavior for the two phase billets is illustrated in Fig. 2. Billets represented by the symbol LH indicate a geometrical arrangement in which the low M_w HDPE ($M_w = 59,000$) and high M_w HDPE ($M_w = 200,000$) were used as sheath and core components respectively. Similarly, HL indicates the inverse arrangement. The number designation corresponds to percent of low M_w HDPE component. The variations in sheath/core composition are shown in Table I. With increasing length of extrudate, the rate decreased as the EDR increased and became constant—steady state extrusion—when the maximum EDR was attained. Rates were calculated and compared for the steady state extrusion in Fig. 3. It is obvious that the low M_w HDPE (L) extrudes at faster rate than the high M_w HDPE (H). However, in the extrusion of combined billets, the two components were extruded simultaneously, at a rate depending on the relative amount of the components, as will be discussed at the deformation mode of the two phase billet. Billets in which the sheath and core components were not uniformly distributed showed inflection and no steady state extrusion. Extrusion was repeated at least twice with each sample to assume reproducibility and uniform distribution of the components even when the extrusion proceeded at steady state.

The steady state extrusion rate as a function of volume fraction of the low M_w HDPE is shown in Fig. 3. Generally the rate increased nonlinearly with the fraction of the low M_w component. Such a nonlinear increase can be explained by a simple calculation based on an empirical relation between extrusion pressure and rate. At a given temperature and EDR, the logarithm of the rate, R , is proportional to the applied pressure¹. Thus

$$\log R = k [P(R) - P_o] \quad (1)$$

where, k = rate constant, $P(R)$ = applied pressure which gives rate R , P_o = pressure constant. The constants k and P_o may vary depending on the molecular characteristics and extrusion conditions.

For the convenience of further treatments Eq. (1) is expressed by the applied force, $F(R)$, and cross-sectional area of the billet, S_o ,

$$\log R = \frac{k}{S_o} [F(R) - F_o] \quad (2)$$

Assuming the same extrusion flow rate for the two polyethylene components which having parallel arrangement in the initial billet, the balance of the forces during extrusion may be expressed as follows:

$$F(R) = XF_L(R) + (1-X)F_H(R) \quad (3)$$

where X is the volume fraction of the low M_w component, and $F_L(R)$ and $F_H(R)$ are the forces required to extrude the low and high M_w polyethylenes at a rate R , respectively.

From Eq's. (2) and (3)

$$\frac{F(R)}{S_o} = P(R) = \left(\frac{X}{k_L} + \frac{1-X}{k_H} \right) \log R + (P_o^L - P_o^H) X + P_o^H$$

or

$$\log R = \frac{(P_o^H - P_o^L) X + P(R) - P_o^H}{\frac{X}{k_L} + \frac{1-X}{k_H}} \quad (4)$$

From the pressure dependence of the extrusion rate, the constants characteristic for each component polyethylene were determined as $k_L = 22$, $P_O^L = 0.13$ (GPa), $k_H = 7.6$, and, $P_O^H = 0.39$ (GPa) at $T_{ext} = 120^\circ\text{C}$ and $\text{EDR} = 25$. Substituting these values for the constants in Eq. (4), the rate for billets was calculated and shown by the dotted line in Fig. 3.

In agreement with observations, the calculation showed a nonlinear increase in the rate with fraction of the low M_w component. However, deviation from the experimental data became larger at higher fraction (>0.75) of the low M_w component. Although the rate generally increased according to Eq. (1) with P_{ext} (or rate) depending on the molecular characteristics of HDPE and extrusion conditions, above a critical P_{ext} stick-slip extrusion occurred. For example, HL-72 and L exhibited stick-slip at $P_{ext} = 0.23$ GPa and so the rates shown in Fig. 3 were obtained at $P_{ext} = 0.20$ and 0.16 GPa, respectively. The phenomena imposed a limitation on the maximum attainable steady-state extrusion rate and also on the applicability of Eq's. (1) and (2).

The geometric arrangement of low and high M_w components in the initial preformed billet had also a remarkable effect on the extrusion rate. The rate is faster when the low M_w HDPE component was used as a core (HL series) rather than as a sheath (LH series). The result was unexpected since it was anticipated that the more deformable low M_w component when used as a sheath, might act as a kind of lubricant and therefore promote the extrusion process. This observation suggests the operation of a deformation mode which is affected by the geometric arrangement of the two components in the billet. Thus the very slow extrusion rate of

high M_w HDPE component alone (~ 0.06 cm/min) could be increased up to 10 times by coextrusion with 25% of low M_w PE component in the middle of the high M_w HDPE billet.

The flow profiles which must be related to the flow rates of combined extrudates and their individual HDPE components (low M_w Alathon 7050 and high M_w Marlex 6003) are shown in Fig. 4. As reported¹³, the individual low M_w HDPE exhibited a typical shear parabolic pattern and the high M_w HDPE a complex "W" shape flow pattern. For the composite extrudates, the flow pattern changed gradually from a parabolic through an approximately flat line and then to a W shape flow pattern or vice versa with the volume fraction of low M_w PE component. The trend was also affected by the geometrical arrangement of the low and high M_w polyethylene components as is depicted in Fig. 4. For example, LH-75 and HL-73 have almost the same composition, nevertheless, the former exhibited a shear parabolic pattern whereas the latter an approximately flat line pattern and have different extrusion rate (viscosities). The flow pattern difference is responsible for the extrusion rate difference, see Fig. 3. This phenomenon is particularly important in the preparation of two phase coextrudates by means of solid state extrusion. The idea was also applied to preparation of coextrudates of different polymers.

The flow patterns of coextrudates was usually less symmetrical than those for single polymer extrudates due probably to some asymmetric disposition of the two components in the initial billet. Nevertheless, these patterns were superimposable along the extrusion direction. As no die swelling was exhibited, the EDR of both inner and outer sections of the coextrudates, which was calcu-

lated as the ratio of final to original distance of the line marks, was in a good agreement with that defined as the ratio of the entrance to exit cross-sectional area of the extrusion die. This means that sheath and core (low and high M_w) polyethylenes were extruded at the same rate and at the same EDR.

The deformation efficiency on solid state extrusion was examined by tensile studies of the coextrudates. The tensile modulus of the extrudates of EDR 25 is plotted as function of the low M_w HDPE fraction in Fig. 5. The high M_w HDPE homopolymer showed a markedly higher modulus than the low M_w HDPE. Although the geometric arrangement in the initial billets affected the extrusion rate and deformation profiles, all the modulus data can be reasonably represented by a straight line.

The tensile strength of the coextrudates is shown in Fig. 6. The high M_w HDPE alone showed a higher strength than the low M_w HDPE. Within wide experimental error the tensile strength data can be represented again by a straight line. The achievement of higher tensile strength with higher M_w HDPE is consistent with the results reported by Perkins et al.¹⁴. On the other hand, it has been reported that M_w has no substantial effect on tensile modulus of highly drawn fibers prepared by solid state extrusion¹⁴ and cold drawing¹⁵. However, Capacio and Ward² found some M_w effects on the modulus and suggested that the presence of high M_w chains was necessary for an achievement of high modulus.

Many authors¹⁴⁻²⁰ emphasize the important role of fully extended chain components in either crystalline or noncrystalline regions on the stiffness of ultraoriented semicrystalline polymers. Keith and Padden²¹ found that the number of tie molecules formed during crystallization process increases with M_w . Thus

it is anticipated that if ultradrawing is carried out under conditions where semicrystalline polymers deform plastically²² and relaxation of extended chains in noncrystalline regions (oriented tie molecules) is suppressed to minimal, the concentration of oriented tie molecules may be higher with higher M_w HDPE. This is further supported by data on ultradrawn HDPE¹ which showed that the total birefringence at around EDR \approx 25 is higher for higher M_w HDPE.

CONCLUSION

The geometric arrangement and volume fraction of the low (or high) M_w HDPE component in preformed billets composed of different M_w HDPE's had significant effects on the solid (crystalline) state coextrusion behavior and on tensile properties of the resultant coextrudates. The extrusion rate increased nonlinearly with the volume fraction of the low M_w HDPE. The rate was also faster when the more deformable low M_w HDPE component was the core rather than the sheath. This effect was associated with an observed change in deformation flow profiles. The geometry had no substantial effects on the tensile modulus and strength of the coextrudates. Both increased linearly with the volume fraction of high M_w HDPE component.

ACKNOWLEDGEMENT

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TABLE I

Composition of the Initial Billets

Symbol	Volume Fraction (%)	
	Sheath	Core
LH-86	86 (L)	14 (H)
LH-75	75 (L)	25 (H)
LH-61	61 (L)	37 (H)
LH-27	27 (L)	73 (H)
HL-14	86 (H)	14 (L)
HL-25	75 (H)	25 (L)
HL-39	61 (H)	39 (L)
HL-73	27 (H)	73 (L)

Where L = low M_w HDPE, $M_w = 59,000$

H = high M_w HDPE, $M_w = 200,000$

= % of low M_w HDPE component

FIGURE CAPTIONS

Figure 1: Schematic for the preparation of a split billet composed of two different M_w HDPE's. Billets 1 and 2 are made of low ($M_w = 59,000$) and high ($M_w = 200,000$) M_w HDPE's. After combining the sheath and core, the two phase billet was melted to improve bondings at the interface, and then split into two halves.

Figure 2: Extrudate length v.s. time. After the critical extrudate length, $L_c = 22$ cm (dotted line) the extrusion reaches steady state (or constant EDR 25). Coextrusion was performed at 120°C , 0.23 GPa (0.16 GPa for L) through a conical die having EDR 25 and entrance angle 20° . No lubrication was employed.

Figure 3: Effects of volume fraction of low M_w HDPE component on the steady state extrusion rate. Calculated rate based on a simple parallel model is shown by the dotted line.

●, sheath: low M_w HDPE ($M_w = 59,000$) and core: high M_w HDPE ($M_w = 200,000$).

■, sheath: high M_w HDPE and core: low M_w HDPE.

▲, individual HDPE components.

The P_{ext} was 0.23 GPa except for □ (0.20 GPa) and ▲ (0.16 GPa).

Figure 4: Schematic drawing of the deformation flow profiles of the two individual HDPE components and their composite extrudates.

Figure 5: Tensile modulus of the coextrudates measured along the fiber axis at room temperature as a function of the low M_w HDPE component.

●, sheath: low M_w HDPE ($M_w = 59,000$) and core: high M_w HDPE (200,000).

■, sheath: high M_w HDPE and core: low M_w HDPE.

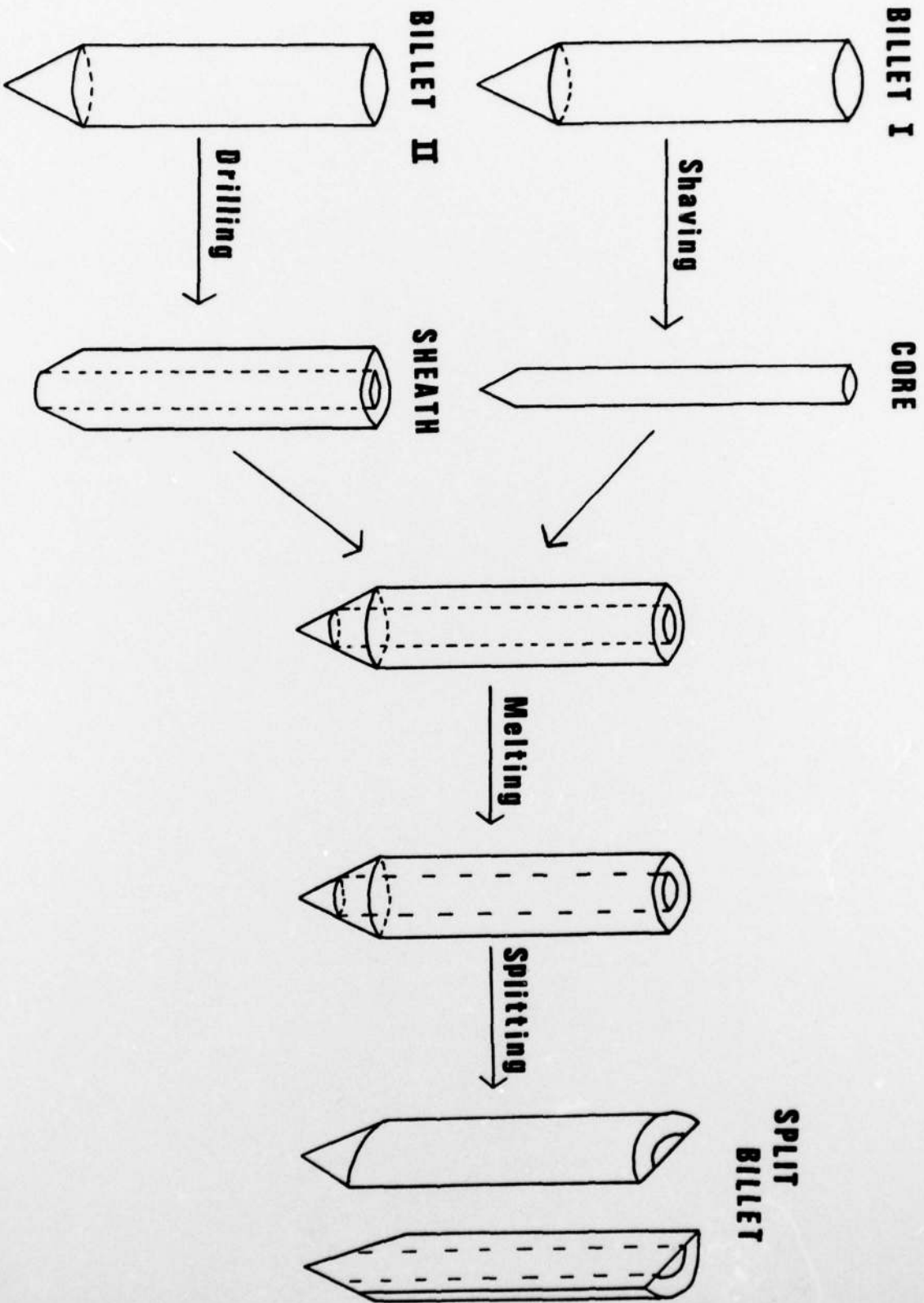
▲, individual HDPE components.

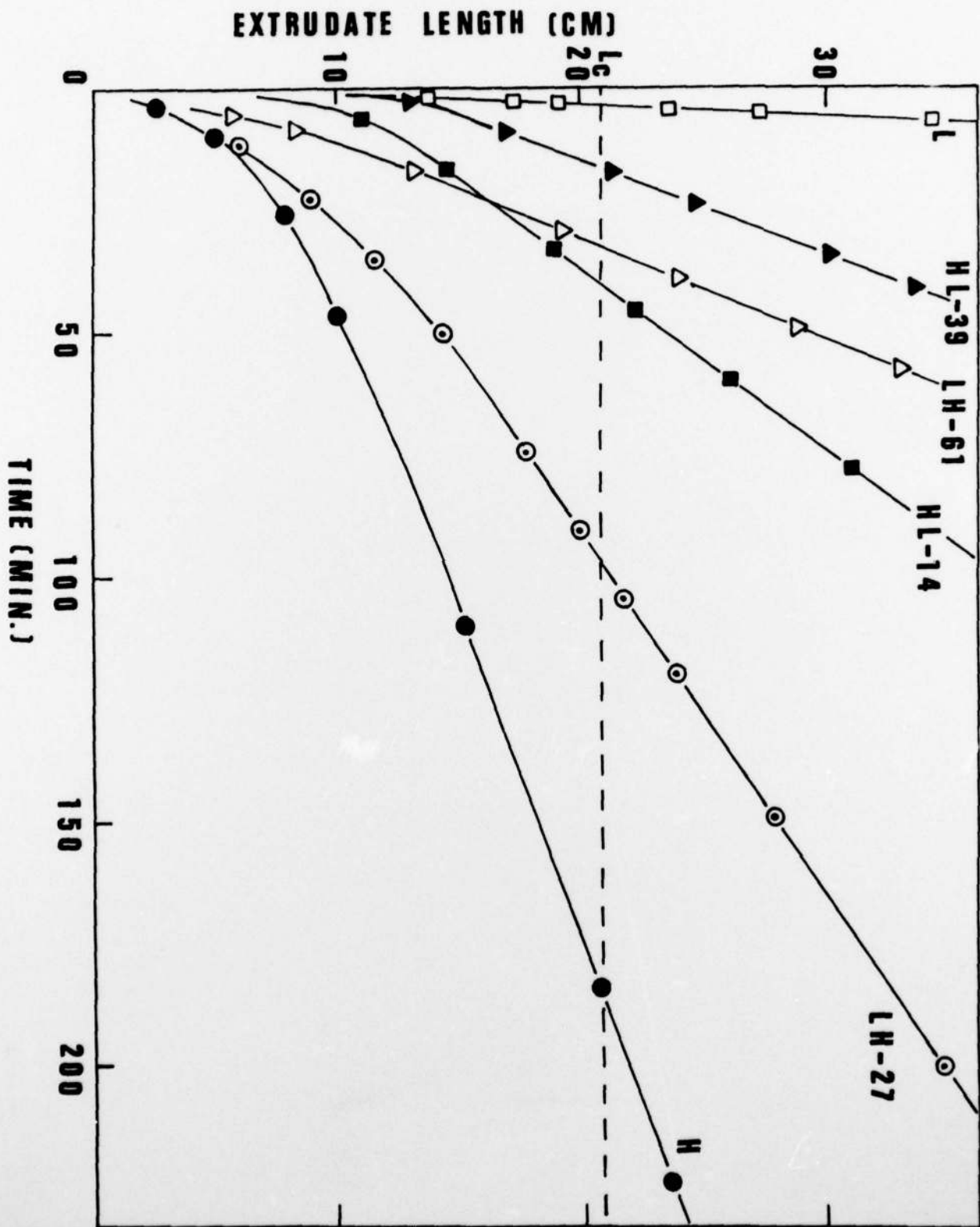
Figure 6: Tensile strength of the coextrudates measured along the fiber axis at room temperature as a function of volume fraction of the low M_w HDPE component.

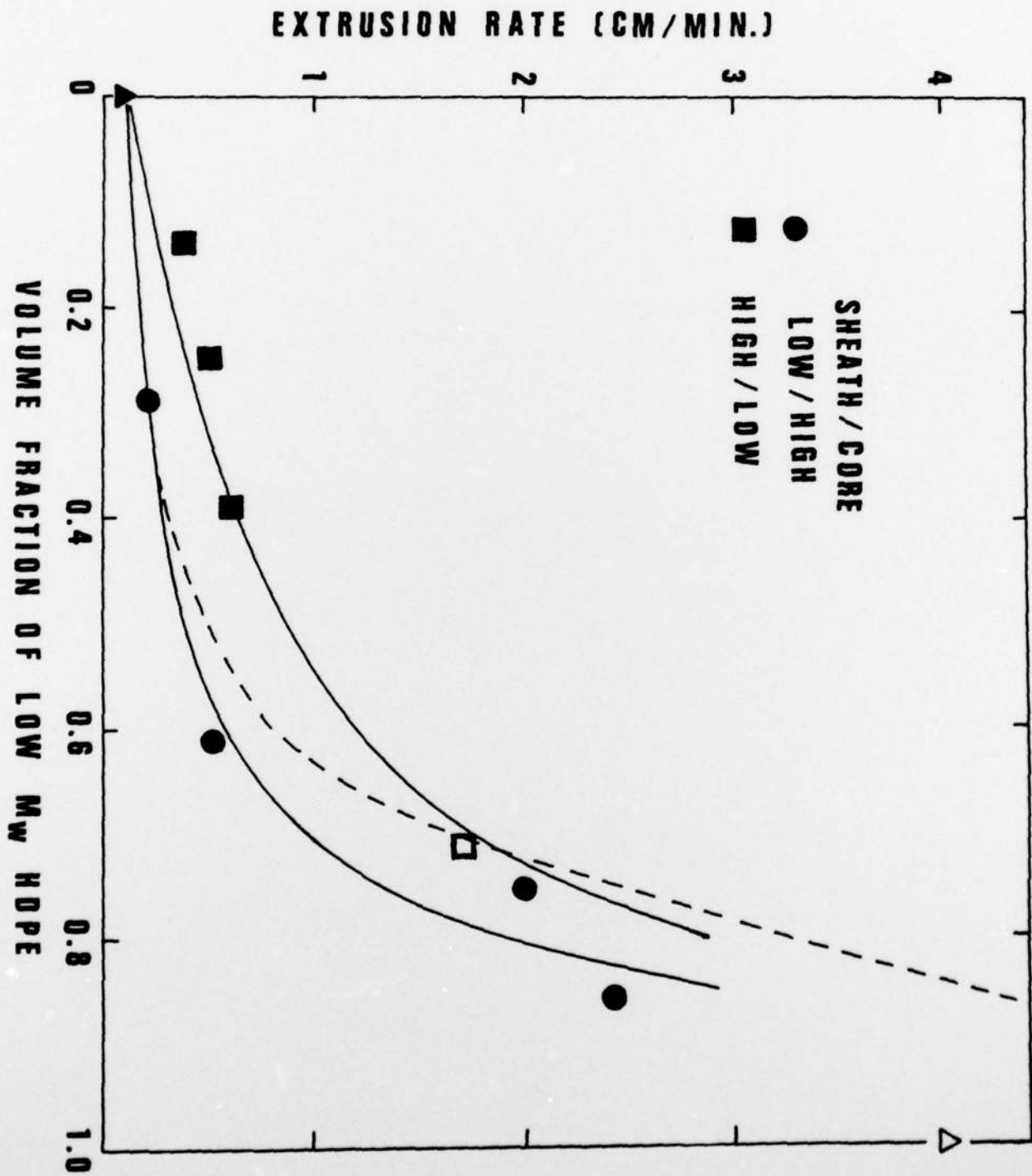
●, sheath: low M_w HDPE and core: high M_w HDPE.

■, sheath: high M_w HDPE and core: low M_w HDPE.

▲, individual HDPE components.







HIGH M_w HDPE



LOW M_w HDPE



EXTRUSION DIRECTION



HL-COMPOSITES



HL-14

HL-25



HL-39

HL-73

LH-COMPOSITES



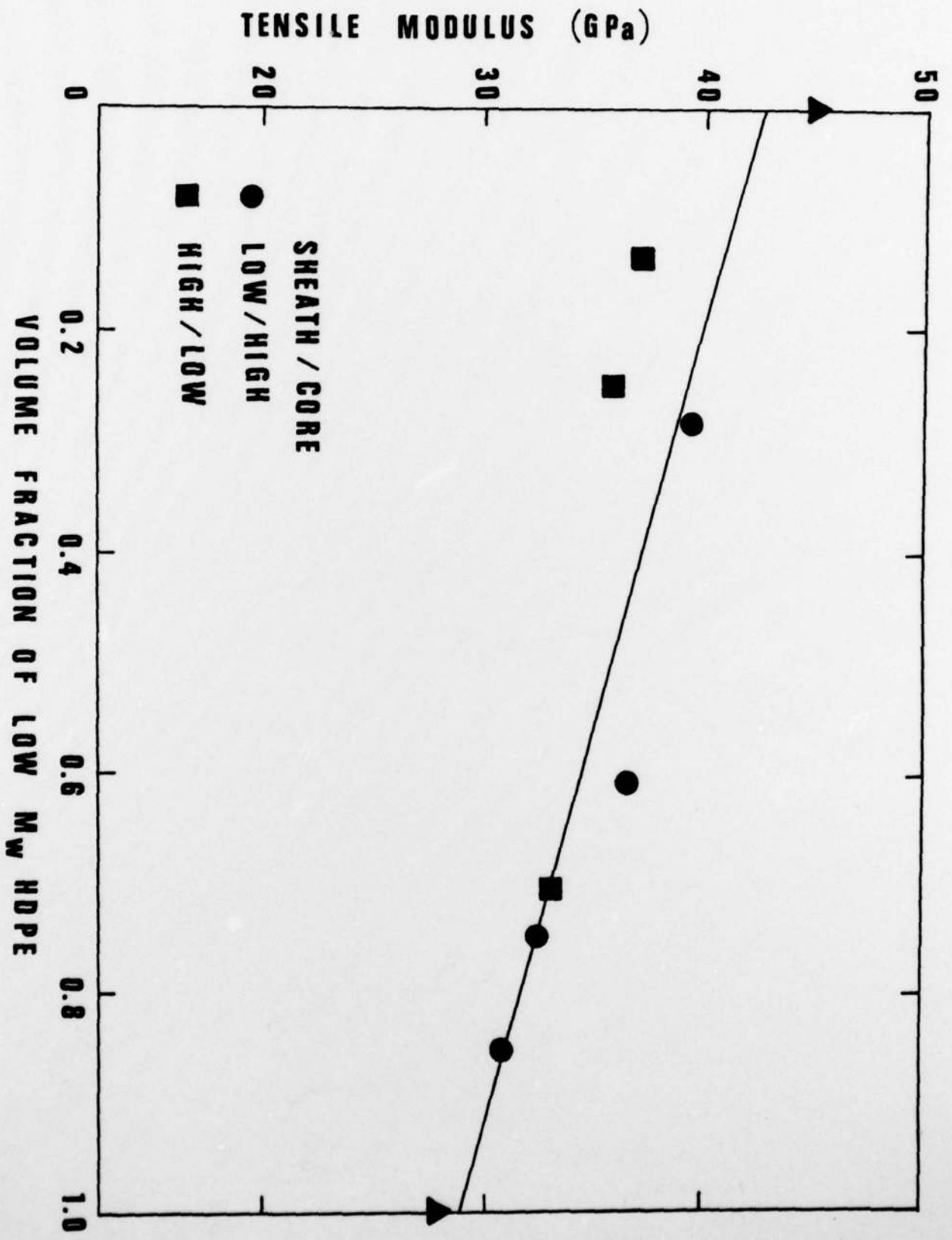
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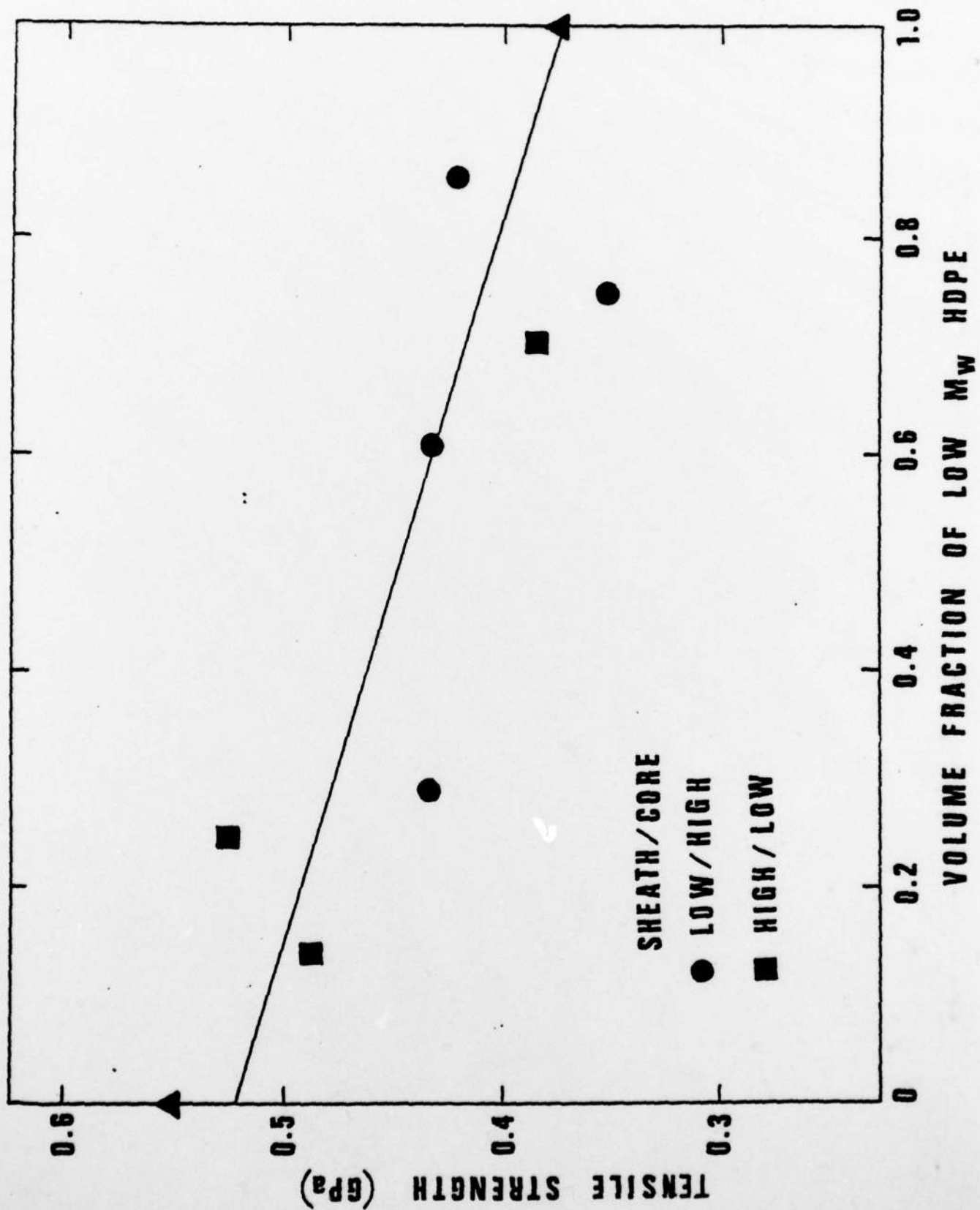
LH-75



LH-61

LH-27





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