

**A NOVEL COEXTRUSION PROCESS FOR THE MANUFACTURE OF
SHORT FIBER-REINFORCED THERMOPLASTIC PIPE**

by

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ABSTRACT

A novel coextrusion process for the manufacture of three-layer plastic pipe has been developed. In this pipe, the middle layer consists of a short fiber-reinforced thermoplastic (SF RTP) having a controlled fiber orientation to provide improved hoop-direction mechanical properties such as stiffness and strength. The inner and outer surface layers consist of an unreinforced polymer selected to provide a smoother surface finish, as well as certain processing and property advantages.

Conventional pipe dies feature an axially converging geometry. Such a die, if used to extrude SF RTP, leads to a predominant axial orientation of fibers, and thus produces little improvement in the hoop properties. A special die featuring an axially expanding flow channel has been proposed to promote fiber orientation in the hoop direction. A theoretical analysis of the flow through such a die has been developed which shows that while preferential hoop orientation is achieved in the region away from the die wall, axial orientation is unavoidable near the surface due to the high shear rates occurring there. An experimental study of SF RTP extrusion through such an expanding channel die has confirmed these findings.

An extension of the theoretical analysis to the process of coextrusion for producing the three-layer pipe described above suggests that this detrimental effect of high shear rate near the wall can be effectively overcome by subjecting the three layer melt to flow through the expanding channel. The analysis also

provides guidelines for the selection of resins with appropriate rheological characteristics, flow rates of the individual layers, and die geometry to optimize this effect. A coextrusion die constructed on the basis of the above principles has been used to manufacture three-layer coextruded pipe using one SF RTP in combination with two different unreinforced resins. A fairly balanced orientation of fibers in the axial and hoop directions throughout the thickness of the SF RTP layer has been achieved.

The mechanical properties of such coextruded pipe have been studied using single and multi-layered plaques simulating the pipe wall structure. It has been demonstrated that the multi-layer structure of the pipe offers a wide scope for the optimization of properties through resin selection and the control of the layer thickness distribution and the fiber orientation. Since failure by slow crack growth is of critical importance in the performance of plastic pipe, crack initiation and propagation studies have also been carried out. These studies clearly show the strong effect of fiber orientation on the crack growth resistance in SF RTP. Crack growth studies in multi-layer test pieces demonstrate the possibility of achieving an improvement in the resistance to the growth of cracks originating at the surface in the case of coextruded pipe.

RESUMÉ

Un procédé nouveau pour la fabrication de tuyaux de plastique à trois couches a été mis au point. Dans de tels tuyaux la couche centrale est formée de thermoplastique renforcé de fibres courtes (TPRFC) possédant une orientation contrôlée pour l'amélioration de propriétés mécaniques telles que la rigidité et la résistance à la rupture dans la direction circonférentielle. Les couches intérieures et extérieures sont formées de polymère non renforcé pour procurer un meilleur état de surface ainsi que certains avantages liés à la mise en oeuvre et aux propriétés.

Les filières à tuyaux classiques ont une géométrie convergente dans le sens axial. Une telle filière utilisée avec du TPRFC provoque une orientation préférentielle des fibres dans la direction axiale et par conséquent procure peu d'amélioration dans la direction circonférentielle. Une filière spéciale comportant un canal d'écoulement divergent dans le sens axial a été proposée pour favoriser l'orientation des fibres dans la direction circonférentielle. Une analyse théorique de l'écoulement dans une telle filière démontre qu'une orientation circonférentielle prédomine dans les régions éloignées de la surface de la filière mais qu'une orientation axiale est inévitable près de cette surface, à cause des taux de cisaillement locaux élevés. Une étude expérimentale de l'extrusion de TPRFC dans une telle filière a confirmé ces résultats.

Une extension de l'analyse théorique au procédé de

coextrusion liée à la fabrication de tuyaux plastiques à trois couches décrit plus haut, suggère que l'effet négatif des hauts taux de cisaillement près de la surface peut-être supprimé lorsque l'écoulement dans le canal divergent implique les trois couches de matières fondues. L'analyse fournit également des bases pour le choix de résines possédant des propriétés rhéologiques appropriées, des débits des couches individuelles et de la géométrie de filière susceptibles de favoriser l'effet désiré. Une filière de coextrusion construite à partir de ces principes a été utilisée pour fabriquer des tuyaux à trois couches comportant un TPRFC combiné avec deux résines différentes non renforcées. Une orientation des fibres pratiquement équilibrée entre les directions axiales et circonférentielles sur toute l'épaisseur de la couche renforcée a été obtenue.

Les propriétés mécaniques de tels tuyaux coextrudés ont été étudiées à l'aide de plaques à une ou plusieurs couches simulant la structure de la paroi du tuyau. On a pu montrer que la structure multicouche du tuyau procure de vastes possibilités d'optimisation par le choix des résines et le contrôle de l'épaisseur des couches et de l'orientation de fibres. Du fait que la rupture par propagation lente des fissures est d'une importance primordiale dans la performance de tuyaux de plastique, des études d'initiation et de propagation de fissure ont été menées. Ces études démontrent le rôle important de l'orientation des fibres sur la résistance à la propagation de fissures dans le TPRFC. Les études de propagation de fissure dans des éprouvettes multicouches démontrent qu'il est possible

d'améliorer la résistance à la propagation de fissures ayant leur origine à la surface pour des tuyaux coextrudés.

To my parents
for so much love
from so far away

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NOMENCLATURE

a	Crack Length
a_0	Initial Crack Length
(a/D)	Dimensionless Crack Length
$(a/D)_0$	Dimensionless Initial Crack Length
Δa	Crack Extension
A_1	Area of the Section of Layer I in a Two-Layer Test Piece
A_2	Area of the Section of Layer II in a Two-Layer Test Piece
B	Particle Shape Parameter in Jeffrey's Equations, or Test Piece Thickness
C	Test Piece Compliance, or Constant of Integration in Equation 3.8
C'	Constant of Integration in Equation 3.11
C_0	Compliance of a Test Piece corresponding to the Initial Crack Length
D	Test Piece width, or Pipe Diameter
$\underline{D}$	Stretching Rate Tensor
$\underline{D}_{ij}$	ij th Component of the Stretching Rate Tensor
e	Bagley End Correction
E	Young's Modulus
E_f	Flexural Modulus
E_1	Modulus of the Layer I Material of a Two-Layer Test Piece
E_2	Modulus of the Layer II Material of a Two-Layer Test Piece
E_u	Modulus of the Unreinforced Resin
f	Fiber Orientation Distribution Function
f_0	Fiber Orientation Distribution at the Entrance to the Expanding Channel
f_1	Fiber Orientation Distribution at the Exit from the Expanding Channel
$\bar{F}$	Overall Fiber Orientation Distribution Function with respect to Angle ψ
F	Force
G_I	Strain Energy Release Rate due to Crack Propagation in Mode I
G_{IC}	Critical Strain Energy Release Rate in Mode I
H	Channel Half Thickness
I_1	Moment of Inertia of Layer I about its Neutral Axis
I_2	Moment of Inertia of Layer II about its Neutral Axis
I_1'	Moment of Inertia of Layer I about the Neutral Axis of the Two-Layer Section
I_2'	Moment of Inertia of Layer II about the Neutral Axis of the Two-Layer Section
$\bar{I}$	Moment of Inertia of the Homogeneous Section of Equivalent Flexural Rigidity as the Two-Layer Section about its Neutral Axis
J	J-Integral Value
K	Consistency, or Stress Intensity Factor
K_1	Consistency of the SF RTP Melt
K_2	Consistency of the Unreinforced Melt

K_I	Mode I Stress Intensity Factor
K_{R}	Stress Intensity Factor during Slow Stable Crack Extension
K_{IC}	Critical Stress Intensity factor
l	Length of a Capillary used for Rheological Characterization of a Polymer Melt
l_0	Initial Gauge Length of a Tensile Test Piece
Δl	Change in the Gauge Length of a Tensile Test Piece
L	Direction of Preferential Fiber Orientation of a Compression Molded Plaque
n	Power-Law Index, or the Normal Direction of a Cylindrical Coordinate System
n_1	Power-Law Index of the SF RTP Melt
n_2	Power-Law Index of the Unreinforced Melt
P	Pressure
Q	Flow Rate in Simple Extrusion
Q_I	SF RTP Melt Flow Rate in Coextrusion
Q_{II}	Unreinforced Melt Flow Rate in Coextrusion
Q_I^*	Dimensionless SF RTP Melt Flow Rate in Coextrusion
Q_{II}^*	Dimensionless Unreinforced Melt Flow Rate in Coextrusion
Q_i	Flow Rate of the i th Layer in Coextrusion
r	Radial Direction of a Cylindrical Coordinate System
r_p	Particle Aspect Ratio
R_0	Mean Radius at the Entrance to the Expanding Channel
R_1	Mean Radius at the Exit from the Expanding Channel
S	Span in the Three-Point Bending Test
t	Pipe Wall Thickness, or Time
t_i	Thickness of the i th Layer of Coextruded Pipe
T	Direction Normal to that of Preferential Fiber Orientation for a Compression Molded Plaque
$\vec{u}$	Velocity Vector
u_s	Magnitude of the Velocity Vector
u_{zy1}	Velocity Component in the z Direction at the Interface in Coextrusion
u_{zm}	Velocity Component in the z Direction at the Center Line
U	Area Under the Force versus Displacement Curve
V	Linear Velocity of Pipe Extrusion
x	Hoop Direction of a Cartesian Coordinate System
y	Normal Direction of a Cartesian Coordinate System
y_1	Interface Location in Coextrusion
y_{1c}	y Coordinate of the Centroid of the Section of Layer I of a Two-Layer Test Piece
y_2	y Coordinate of the Centroid of the Section of Layer II of a Two-Layer test Piece
$\bar{y}$	y Coordinate of the Centroid of the Homogeneous Section of Equivalent Flexural Rigidity as the Two-Layer Test Piece
y_1^*	Interface Location Corresponding to the Interfacial Shear Rate $\dot{\gamma}^*$
z	Flow Direction of a Cartesian Coordinate System
z_i	Component of the Vorticity Tensor in Jeffrey's Equations

Greek Symbols

α	Angle of Expansion of an Expanding Channel Die
β	Angle of the Velocity Vector with respect to z axis
$\dot{\gamma}$	Shear Rate
$\dot{\gamma}^*$	Shear Rate Corresponding to the Point of Cross-Over of the Flow Curves of the SF RTP and Unreinforced Melts
$\dot{\gamma}_A$	Apparent Wall Shear Rate in Capillary Rheometry
$\dot{\gamma}_W$	Actual Wall Shear Rate in Capillary Rheometry
γ_0	Surface Energy in Griffith's Equation (Equation 5.2)
$\dot{\Delta}$	Rate of Deformation Tensor
$\dot{\epsilon}$	Extensional Rate
η	Viscosity
θ	Circumferential Direction of a Cylindrical Coordinate System
II_{Δ}	Second Invariant of the Rate of Deformation Tensor
ρ	Density
σ_C	Fracture Strength
σ_H	Tensile Strength in the Hoop Direction
σ_A	Tensile Strength in the Axial Direction
σ_Y	Tensile Yield Strength
τ	Stress Tensor
ϕ	Angle Describing Fiber Orientation with respect to the Shear Plane
ϕ_0	Value of ϕ at the Entrance to the expanding Channel
ϕ_1	Value of ϕ at the Exit from the Expanding Channel
ψ	Angle Describing Fiber Orientation with respect to the Hoop Direction
ψ_0	Value of ψ at the Entrance to the Expanding Channel
ψ_1	Value of ψ at the Exit from the Expanding Channel
$\bar{\psi}$	Average Angle of Orientation with respect to the Hoop Direction

ABBREVIATIONS

ABS	Acrylonitrile-Butadiene-Styrene
EPFM	Elastic-Plastic Fracture Mechanics
FRP	Fiber-Reinforced Plastic
GRP	Glass-Reinforced Plastic
LEFM	Linear Elastic Fracture Mechanics
PE	Polyethylene
PP	Polypropylene
PVC	Polyvinyl Chloride
SEM	Scanning Electron Microscopy
SF RTP	Short Fiber-Reinforced Thermoplastic
TLM	Transmission Light Microscopy

Chapter 1

INTRODUCTION

Plastic pipe is now in common use for the transport of fluids and slurries, and for electrical insulation and mechanical protection of underground cables (1-3). Its major applications include:

- 1) Natural gas distribution systems
- 2) Potable water supply systems
- 3) Underground wire and cable systems
- 4) Irrigation and sewage systems
- 5) Industrial applications (in mines, oil fields, chemical processing plants etc.)

The use of plastic in these applications offers several advantages over other materials of construction such as metal or ceramic. These include better performance at a lower cost, easier handling because of lighter weight and greater flexibility, ease of joining and resistance to chemical attack by the environment or by the contained material. However, the performance of plastic pipe is often limited by the relatively low mechanical properties of plastics compared to metals (4-8). Field experience has demonstrated that improvements in the mechanical and other characteristics, discussed below, are desirable for optimal service life and economics.

1.1 Desirable Characteristics of Plastic Pipe

- 1) Strength:- The basic requirement of a piping system is that it sustain the stresses imposed as a result of internal or external pressures and other loads. When a pipe is subjected to an internal pressure, P , the stress in the hoop direction, σ_H , is approximately

$$\sigma_H = \frac{P.D}{2 t} \quad (1.1)$$

while that in the axial direction, σ_L , is approximately

$$\sigma_L = \frac{P.D}{4 t} \quad (1.2)$$

where D and t are the pipe diameter and wall thickness, respectively. The stress in the hoop direction is, thus, about twice that in the axial direction. Consequently, it is highly desirable to improve the hoop strength of pipe.

- 2) Stiffness :- Pipe stiffness or rigidity is a measure of its dimensional stability under mechanical loads. A high transverse stiffness is desirable, particularly, in underground applications such as in cable conduits and gas distribution lines. On the other hand, some degree of axial flexibility is desirable for ease of conforming and layout, and protection against seismic movements. Moreover, axial flexibility is important in small diameter pipes to allow coiling and uncoiling for ease of transportation and handling.

- 3) Toughness :- A piping system should be strong as well as tough

to be able to endure rough handling during shipping, storage and installation, and unforeseen impacts while in service. It should also have a superior resistance to slow crack growth for a long service life. As explained in Chapter 5, failure by slow crack growth has been the most prominent mode of service failure, and is the major current limitation on the reliability of plastic pipe and the extension of its application.

- 4) **Ease of Installation:-** This is important from the standpoints of convenience and economy. An important factor to be noted here is the ease of joining pipe ends and fittings. Conventional methods of joining thermoplastic pipe are described in Appendix 6.1.

The current project concerns the development of a new process for manufacturing plastic pipe that leads to improvements in several of the above-mentioned characteristics. Before describing the specific goals of this research, a brief account of the polymeric materials most often used for making plastic pipe and the traditional methods of manufacturing pipe are presented.

1.2 Resins and Methods Used for Manufacturing Plastic Pipe

For reasons of economy and performance, several general purpose thermoplastic resins have been used to make plastic pipe. The leading materials are polyethylene (PE) and polyvinyl

chloride (PVC). Underground natural gas distribution lines use almost exclusively PE pipe, while underground conduits are usually made of PVC. Both PE and PVC are used for water distribution, sewage and irrigation systems. Several pipe grade PE resins are commercially available which offer a spectrum of properties from superior toughness and ductility to high stiffness and strength. Resins formulated specially for pipe applications by control of properties through variation in molecular structure and molecular weight distribution are also available. Such resins usually incorporate comonomers and long chain branching. Several variations of PVC resins are also available. Rigid PVC incorporating a high loading of filler is generally used to make conduits for cables. Impact grade or plasticized PVC is used to manufacture flexible hose for domestic and automotive applications. Chlorinated PVC is used to make pipe for hot water distribution systems (9). Other thermoplastic materials used to make pipe or hose include crosslinked polyethylene, polypropylene, acrylonitrile-butadiene-styrene (ABS), polybutadiene and nylons (7-10).

Traditional method of manufacturing plastic pipe involves extrusion through an annular die (11) such as the one shown in Figure 1.1. It has a central mandrel held in position by a spider-type support. The molten polymer flows around the mandrel and emerges as a tubular extrudate. Separation of the flow stream around a spider leg and its subsequent recombination may lead to formation of a weld line along the length of pipe. The converging

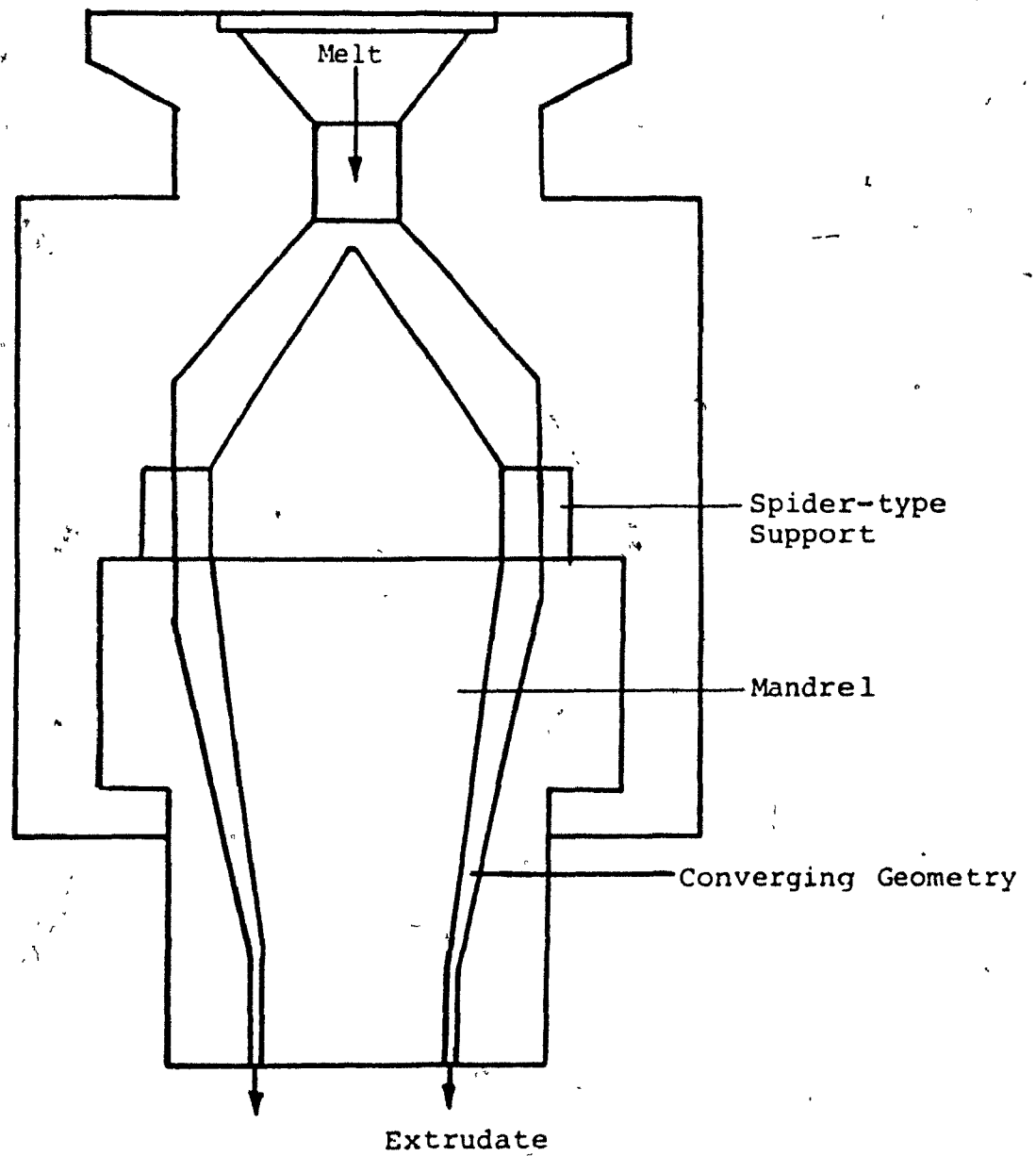


Figure 1.1 Typical Pipe Extrusion Die

section helps improve the flow uniformity around the mandrel, and thereby assists in the healing of weld lines. However, it also promotes less desirable axial molecular orientation in the pipe and, thus, does not contribute to improving hoop properties. The use of a rotating mandrel die has been proposed not only to promote the healing of weld lines and thickness uniformity around the circumference but also to promote more desirable hoop orientation of molecules (14,15). Spiderless die designs have also been proposed to avoid weld line formation (16).

Once the pipe comes out of the die, it enters a calibrating/cooling section. The calibrating section is usually under vacuum and air is blown inside the pipe. The pipe achieves the shape and dimensions of the inner wall of the calibrator due to the resulting pressure difference along with the freezing of a thin layer of the molten plastic at the surface. Further cooling occurs in flooded cooling tanks operated under vacuum, and the pipe is subsequently taken off by caterpillar haul-offs (12-13).

Continuous fiber-reinforced pipe or duct of thermosetting materials (commonly referred to as glass reinforced plastic-GRP or fiber-reinforced plastic-FRP) represents another class of plastic-based pipe. Such a pipe is usually extremely stiff and strong and is used as drainage duct, conduit for high voltage cables and as a structural member. Various thermosetting resins such as epoxy, phenolic and polyester are used for its manufacture. A common method of manufacturing involves winding of

the resin-impregnated continuous reinforcing fibers over a rotating mandrel, followed by curing (17). Several variations of this method have been proposed to incorporate different types of reinforcing elements or to achieve certain processing advantages (18-21). Reinforced plastic mortar (RPM) pipe is manufactured by centrifugal casting a mixture of resin, fibers and filler in a cylindrical mold and can incorporate short fiber reinforcement (10,22).

Representative mechanical and other properties of different types of pipe are summarized in Table 1.1 (23). Properties of some metallic and ceramic piping materials are also included for comparison. As mentioned above, while GRP pipe possesses outstanding stiffness and strength, it lacks flexibility. Thermoplastic materials (particularly PE) offer excellent flexibility and chemical inertness but lack the superior stiffness and strength of GRP or metallic materials.

A plastic pipe that combines the superior properties of the GRP with the flexibility and chemical resistance of the thermoplastics thus seems highly desirable. In addition, it is highly desirable to develop a relatively simple continuous process for its manufacture that is similar to the traditional method of extrusion.

Table 1.1

Comparative Properties of Various Materials Used to Manufacture Pipe (23)

Material	Mechanical properties					Thermal properties		Chemical resistance in temperature range 20-100°C		
	Tensile				Compressive strength Nmm ⁻²	Thermal expansion coefficient °C ⁻¹ × 10 ⁻⁶	Resistant to heat up to °C	Acids	Alkalies	Solvents
	U.T.S. Nmm ⁻²	Long term/ design stress Nmm ⁻²	Elongation at break %	Young's Modulus KNmm ⁻²						
GRP Unidirection.										
to fibres	400-1200	130-400	1.8	21-41	200-500	5-14				
⊥ to fibres	20-50	10	0.2-0.6	8	90-250	-	180	Fair	Fair	Good
C.S.M.	60-140	20-45	1.2	6-12	130-170	22-36	150	Good	Good	Good
U.P.V.C.	45-60	5-12	1.6-2.2	2.8	55-90	50-60	65-80	Very good	Excellent	Good
L.D.P.E.	12-13	3.2	350-550	0.1-0.25	16	170-280	80-100	Good	Excellent	Poor
H.D.P.E.	20-30	5.0	150-600	0.6-1.0	17	120-160	110-130	Very good	Excellent	Fair
Polypropylene	32-39	5.0	40-600	1.0-1.8	60-70	100-200	130-150	Very good	Excellent	Very good
A.B.S.	35-55	7.5	10-45	2-3	47-86	70-110	95-105	Poor	Fair	Fair
Mild steel	400-500	10-125	35-40	206	400-500	12	820	Poor	Fair	Excellent
Stainless steel	500-1600	150-400	40-60	193	500-1600	10.5	1150	Good	Good	Excellent
Asbestos cement	22-45	6-10	0.1	20-30	50-150	8.5	Limited by jointing method	Very poor	-	-
Clayware	5-15	5-15	0.02-0.06	30	10-20	5		Good	Fair	Excellent

1.3 Overall Goal of the Project

The main objective of this research was to develop a novel process of coextrusion in order to produce a short fiber-reinforced thermoplastic (SF RTP) pipe with a three-layer wall structure in order to achieve improvements in several of the above-mentioned characteristics. In such a pipe, the inner and outer surface layers are of unreinforced polymer and the middle layer is of SF RTP composite material, as shown schematically in Figure 1.2.

The use of orientable discontinuous particles in polymers is a proven way of improving mechanical properties such as stiffness and strength. Short fibers of glass, carbon or cellulose, ceramic whiskers, mica flakes etc. are becoming increasingly popular reinforcing elements in products made by injection molding. The short-fiber-reinforcement allows the polymer to be processed employing the same methods as those used for unreinforced polymers. However, the improvement in properties due to fiber reinforcement is strongly dependent on the structure of the produced article, particularly the fiber orientation pattern present in it (24). While significant improvement of mechanical properties usually occurs in the direction of preferential fiber orientation, very limited improvement or even deterioration of properties can occur in the direction normal to it.

The orientation is mainly governed by the flow pattern in

the processing equipment. As was mentioned earlier, conventional pipe dies tend to promote axial orientation primarily because of their converging geometry and, therefore, if used to extrude SF RTP composites, would lead to property enhancement in the axial direction. In the case of pipe, however, improvement in the hoop properties is desired. Nonconventional die geometries were, therefore, considered in this work, and an expanding channel geometry was deemed suitable in order to promote hoop orientation of fibers in the proposed SF RTP pipe.

The inclusion of fibers is also known to cause a deterioration of the surface quality of an extruded product such as pipe due to the protrusion of fibers out of the surface. Such a rough surface is undesirable for reasons of aesthetics, difficulties in handling, possibility of fiber contamination and increased frictional drag in the case of fluid transportation. A coextrusion process was considered to circumvent this problem. Coextrusion is a process of producing a multi-layered product by simultaneous extrusion of various layers of different materials through a single die. The proposed process involves the formation of a three-layer pipe with the inner and outer surface layers of an unreinforced polymer, as shown in Figure 1.2, in order to provide a smooth surface, and the middle layer of SF RTP with fibers oriented preferentially in the hoop direction in order to improve hoop properties.

An understanding of the processing-structure-properties

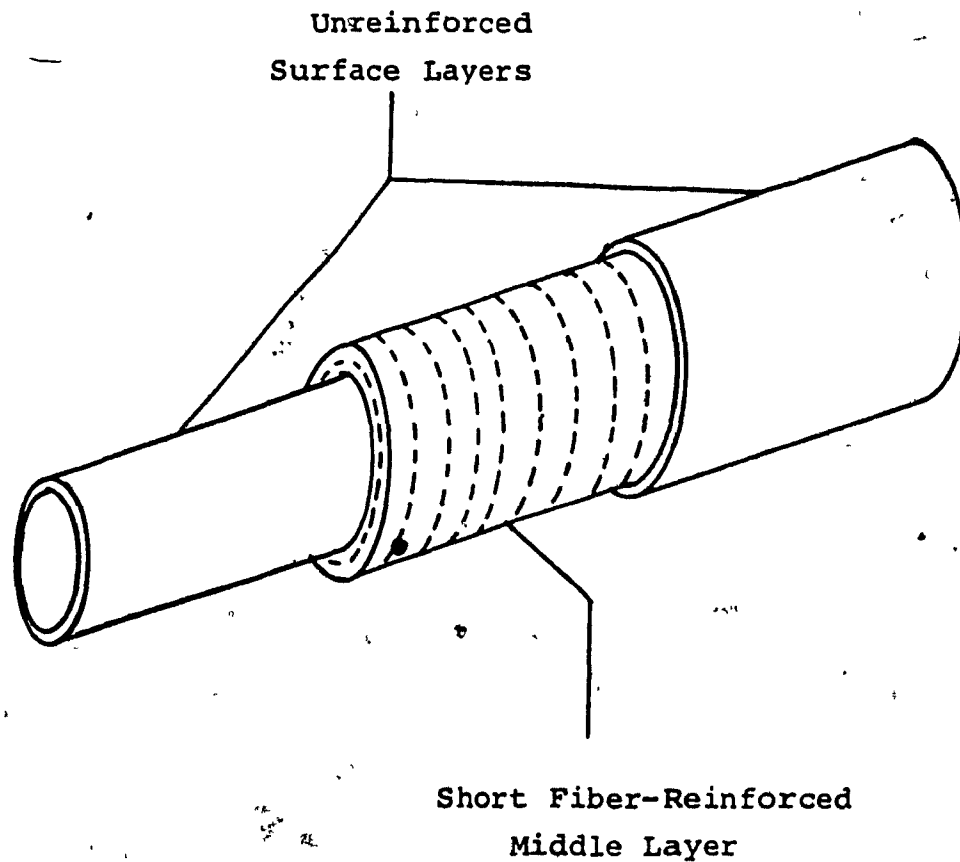


Figure 1.2 Coextruded Pipe with Three-Layer Wall Structure.

relationships of the SF RTP composite materials is of primary importance in the development of the proposed process. The specific objectives of the project hence involved a theoretical and experimental study of these aspects in relation to the coextrusion process, and a study of the mechanical properties of coextruded pipe. The sequential objectives of the project and the thesis organization are described in the following section.

1.4 Specific Project Objectives and Thesis Organization

First of all, it was necessary to select SF RTP and unreinforced resins to be used in the experimental study. In Chapter 2 (Materials and Their Characterization), the selection, and rheological and mechanical characterization of the materials are discussed.

Chapters 3 to 5 are individually devoted to the three major sequential objectives as discussed below:

- 1) The first objective was to consider a non-conventional pipe extrusion die incorporating a special geometry in order to study the feasibility of promoting hoop orientation of fibers by simple extrusion of SF RTP through it.

This objective is explored both theoretically and experimentally in Chapter 3 (Simple Extrusion Studies). This chapter begins with a short review of the previous studies

related to the flow-induced fiber orientation in SF RTP composites and their processing-structure-properties relationships. The review leads to the selection of an expanding channel annular geometry for promoting hoop orientation of fibers. The case of simple extrusion through a die incorporating such a geometry is analyzed theoretically in order to estimate the fiber orientation distribution in the extrudate. An experimental study of simple extrusion of SF RTP carried out using an appropriately designed die is then described, and the results are discussed in terms of a qualitative comparison with the theoretical predictions. These results lead to the development of a strategy for the design of a coextrusion die to produce the desired pipe.

- (2) The second objective was to study the proposed process of coextrusion both theoretically and experimentally in order to assess the feasibility of producing the coextruded pipe with the desired wall structure and desired fiber orientation pattern in the SF RTP layer.

This objective is dealt with in Chapter 4 (Coextrusion Studies). The theoretical analysis developed in Chapter 3 is extended to predict the fiber orientation distribution developed in the coextruded pipe. This analysis provides further insight into the effects of various material, processing and geometrical parameters on the process of fiber orientation during flow. The design of a coextrusion die based on the general conclusions of the theoretical analysis is then presented. Coextrusion

experiments carried out using a die constructed according to this design are described and the results of the experimental characterization of the wall structure and fiber orientation pattern in the SFRTF layer of coextruded pipe are discussed.

- (3) The final objective was to study mechanical properties of coextruded pipe and special characteristics of its multi-layered structure.

Chapter 5 (Mechanical Properties of Coextruded Pipe) concerns this objective. In this chapter, a review of the modes of reported field failures of plastic pipe is presented to identify the properties of importance in pipe performance. Several tests carried out to characterize the mechanical behavior of the coextruded pipe in terms of the above properties are described and the results are discussed.

Finally, the overall conclusions of this study, along with the recommendations for further work, are presented in Chapter 6 (Conclusions and Recommendations).

Chapter 2

Materials and their Characterization

The experimental work was carried out using short fiber-reinforced and unreinforced resins based on polyethylene. Commercial polyethylene pipe is usually manufactured from resins specially formulated according to proprietary procedures for specific mechanical properties and extrusion characteristics. A low melt flow index (MFI) and a high melt strength are normally desirable for the ease of processing. For the purpose of this work, however, it was recognized that the high viscosity of such resins would hinder the uniform dispersion of the reinforcing glass fibers in the melt during compounding. Since short fiber-reinforced composite systems based on such resins are not commercially available, their selection would have required developmental compounding. For the present project, however, it was desirable not only to acquire large quantities of resins, but also to ensure the availability of additional supply, if required. Under these circumstances and after consultation with a commercial supplier (25), a commercially available SF RTP composite system based on an injection molding grade of polyethylene resin was selected. In addition, two unreinforced polyethylene resins were selected for reasons discussed below.

2.1 Materials Used

The SF RTP system was based on a polyethylene resin, SCLAIR PE 2906 M6, manufactured by Du Pont Canada, Inc. It had a glass fiber loading of 30% by weight. The glass fibers were in the form of chopped strands of E-glass and were coated with a proprietary silane coupling agent (25). The system was compounded by Wilson Fiberfil (Canada) and was supplied in pellet form.

Of the two unreinforced polyethylene resins used, one was the same as the matrix resin in the SF RTP system, i.e., SCLAIR PE 2906 M6. The other resin was SCLAIR PE 74A, also manufactured by Du Pont Canada, Inc. Properties of these two resins as supplied by the manufacturer are given in Table 2.1. PE 2906 M6 is a high density, low MFI injection molding grade resin. It is referred to as the "high density unreinforced resin" in this thesis. Its choice was guided by the fact that its use in the manufacture of the proposed coextruded pipe would minimize problems arising from melt incompatibility and layer adhesion. PE 74A is a low density film grade resin, and has about the same MFI as PE 2906 M6. It will be referred to as the "low density unreinforced resin". Its selection was guided by its excellent extrusion characteristics demonstrated in some preliminary pipe extrusion runs, and its distinct mechanical properties such as low stiffness and yield strength, and high toughness. Because of these characteristics, it could be used not only for the start-up of extrusion as described in Chapter 4, but also for demonstrating certain

Table 2.1

PROPERTIES OF UNREINFORCED RESINS*

Property	ASTM Test	Value	
	Method	PE 2906 M6	PE 74A
Solid Density	D 1505	0.96 g/cm ³	0.935 g/cm ³
Tensile Yield Strength (500 mm/min)	D 1248	31 MPa	1.7 MPa
Elongation at Yield (500 mm/min)	D 1248	40 %	700 %
Tensile Impact Strength	D 1822	18 J/cm ²	35 J/cm ²
Flexural Stiffness (Secant Modulus at 1% Strain)	D 747	965 MPa	425 MPa
Shore Hardness	D 1706	D 65	D 63
Vicat Softening Point	D 1525	130° C	115° C
Brittleness Point	D 746	< - 70° C	< - 70° C
Melt Index	D 1238	0.75 dg/min	0.75 dg/min

* Information Supplied by the resin manufacturer.

Table 2.3

Mechanical Properties of Materials Used

Material	Flexural Stiffness	Tensile Yield
	(MPa)	Strength (MPa)
High Density Unreinforced Resin (PE 2906 M6)*	1250	22
Low Density Unreinforced Resin (PE 74 A)*	820	13
SF RTP**	4280	53

* Compression Molded Test Pieces

** Injection Molded Test Pieces

interesting mechanical characteristics of the multi-layer structure of coextruded pipe, as will be discussed in Chapter 5.

2.2 Characterization of the Materials

The flow curves (shear rate versus viscosity) of the above materials were obtained using an Instron capillary rheometer. A series of capillaries with a diameter of 0.76 mm (0.03 in) and l/d ratios ranging from 10 to 40 were used. All measurements were carried out at a temperature of 200 °C and in the apparent shear rate range of about 10 to 1300 s⁻¹. The Bagley end correction and the Rabinovitsch correction were made using the following equations in order to obtain shear stress (τ_w) and true shear rate ($\dot{\gamma}_w$) at the wall (26).

$$\tau_w = \frac{\Delta P}{4(l/D + e)} \quad (2.1)$$

$$\dot{\gamma}_w = \dot{\gamma}_A \left[\frac{3}{4} + \frac{1}{4} \left(\frac{d \ln \dot{\gamma}_A}{d \ln \tau_w} \right) \right] \quad (2.2)$$

where ΔP is the axial pressure drop across the capillary, e is the Bagley end correction and $\dot{\gamma}_A$ is the apparent shear rate at the wall. The viscosity, η , as a function of shear rate was then calculated as follows:

$$\eta(\dot{\gamma}) = \frac{\tau_w}{\dot{\gamma}_w} \quad (2.3)$$

The viscosity-shear rate relationships for the SF RTP and the two unreinforced resins are shown in Figure 2.1. The numerical

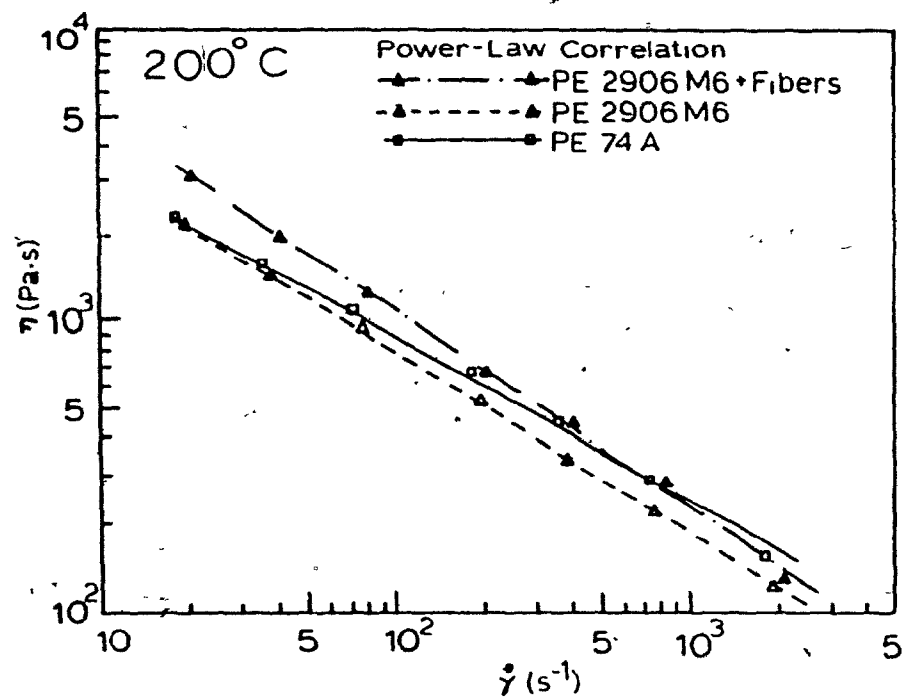


Figure 2.1 Viscosity-Shear Rate Relationship for the Materials Used

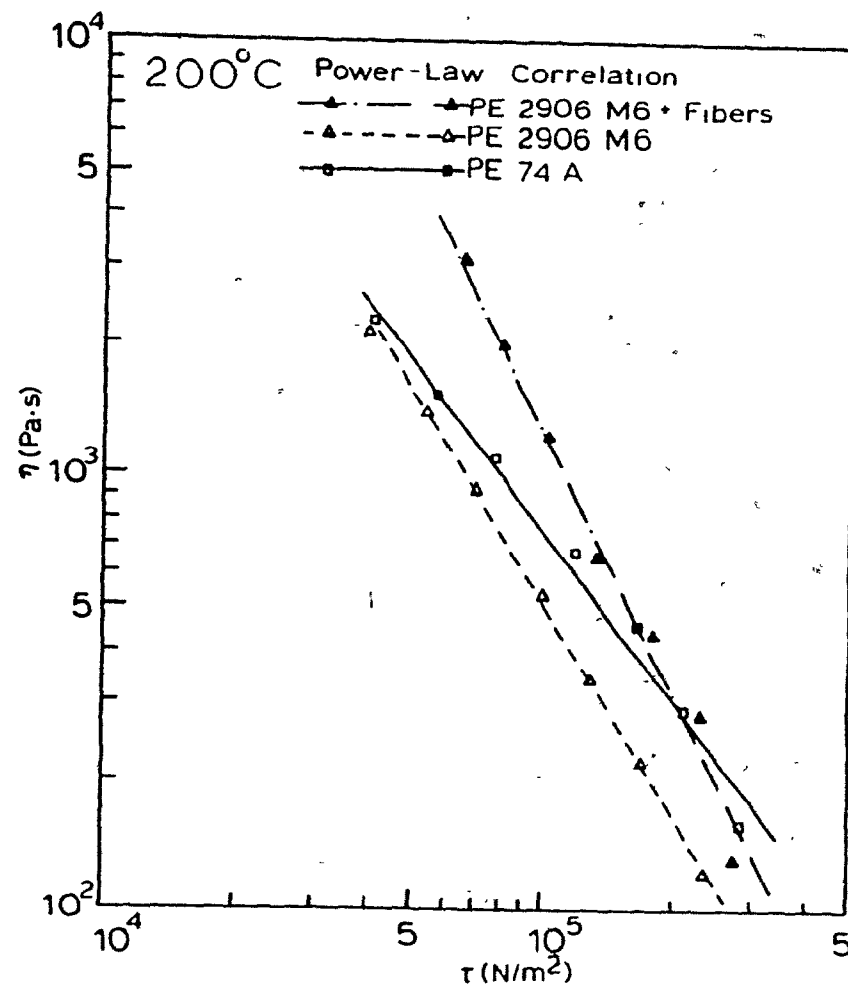


Figure 2.2 Viscosity-Shear Stress Relationships for the Materials Used

data are provided in Appendix 2.1. As expected (27), the SF RTP melt has a viscosity somewhat higher than that of the high density unreinforced resin (PE 2906 M6) over the range of shear rates studied. In the low shear rate region, the viscosity of the low density unreinforced resin (PE 74A) is close to that of the high density unreinforced resin, as indicated by their similar MFI values. At higher shear rates, however, its viscosity is higher than that of the high density unreinforced resin and close to that of the SF RTP melt. In Figure 2.2, the viscosities of the three materials are plotted as functions of the shear stress. These results are utilized for the flow analysis of coextrusion experiments in Chapter 4.

In order to check whether the compounding operation for the preparation of the SF RTP had caused any degradation of the resin, the molecular weight distribution measurements were carried out by size exclusion chromatography (SEC) with the help of Dr. P. C. Wong of Du Pont Canada, Inc. The molecular weight distributions for the matrix resin of the SF RTP system and the high density unreinforced resin (PE 2906 M6) are shown in Figure 2.3. Various molecular weight averages and polydispersity indices determined from these distributions are given in Table 2.2. The slight differences in the molecular weight distributions do not seem to indicate a resin degradation and may be a reflection of the usual fluctuations in the process parameters during resin manufacture and/or batch to batch variations in additive concentrations (28).

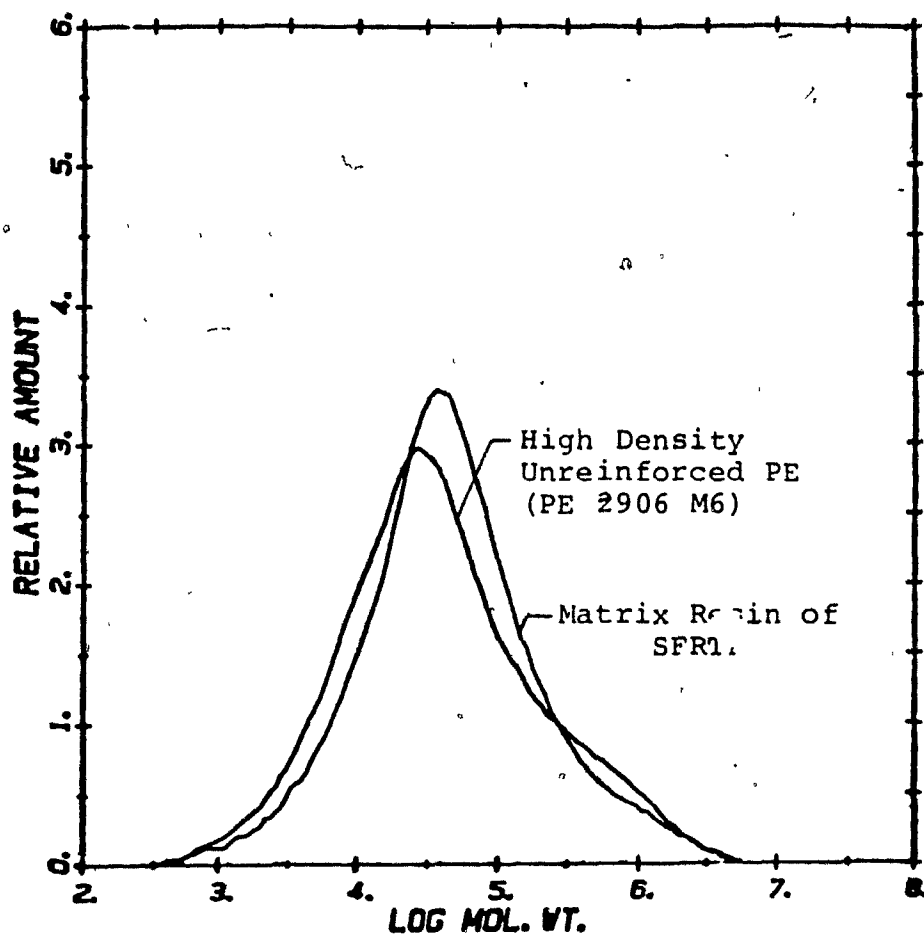


Figure 2.3 Molecular Weight Distributions of the High Density Unreinforced PE (PE 2906 M6) and the Matrix Resin of SFRT.

Table 2.2

Molecular Weight Averages and Polydispersity Indices

	Matrix Resin of SFRT	High Density Unreinforced Resin
No. Avg. Molecular Wt. (M_N)	16714	12661
Wt. Avg. Molecular Wt. (M_W)	136236	150941
z - Avg. Molecular Wt. (M_z)	942588	1064849
Polydispersity Index (M_W/M_N)	8.15	11.92

The characterization of the mechanical properties of the materials was carried out by measuring their flexural moduli and yield strengths. The measurements for the unreinforced polymers were carried out by the procedure described in Chapter 5. The measurements for the SF RTP system were carried out by the supplier of the compound using injection molded test pieces. The data are given in Table 2.3. The measured yield strengths of the unreinforced resins (Table 2.3) are somewhat lower than those reported by the manufacturer (Table 2.1). This may be due to differences in the method of test piece preparation and in the rate of testing. The measured flexural moduli (Table 2.3), on the other hand, are higher because they are based on the initial slope of the force-displacement records, whereas the values reported in Table 2.1 are based on the slope of the secants corresponding to 1% strain. In the case of SF RTP, the properties are known to be strongly dependent on the fiber orientation pattern. The injection molded test pieces usually feature a predominant orientation in the flow direction, which is the direction of testing for the results reported in Table 2.3.

SIMPLE EXTRUSION STUDIES

In this chapter, theoretical and experimental studies carried out to examine the feasibility of promoting hoop orientation of fibers by simple extrusion of SF RTP through a die incorporating a special geometry is discussed. Before discussing these studies, a brief review of the processing-structure-properties relationships of the SF RTP composites is presented.

3.1 SF RTP Composites and Their Processing-Structure-Properties Relationships

A composite material can be defined as a combination of several distinct phases selected to enhance or modify certain properties of one or several of them (24). In the case of SF RTP, the polymer forms a matrix or continuous phase in which the discrete short fibers are dispersed.

The reinforcement of plastics by discontinuous fibers or other orientable particles results in improvements in several mechanical properties such as stiffness and strength and generally provides better performance at lower cost (29,30). The enhancement of various mechanical properties, however, is strongly dependent on the structure of the composite part. In particular, the following structural parameters are important in

determining the properties of a composite:

- 1) Fiber loading.
- 2) Fiber orientation distribution or spatial arrangement.
- 3) Fiber shape and dimensions (aspect ratio).
- 4) Fiber-matrix adhesion.

Fiber loading, usually specified in terms of mass fraction or volume fraction, determines the overall degree of reinforcement. An increase in fiber loading usually leads to an increase in stiffness and strength up to the point where the matrix acts only as a binding medium, the applied load being almost completely carried by the reinforcing fibers. At a very high fiber loading poor bonding and, hence, a weaker material can result.

An increase in the fiber loading also leads to an increase in melt viscosity and a reduction in the ease of processing. Ease of processing is of particular importance for an extrusion product such as pipe and puts an upper limit on the fiber content that can be used. Thus, it is necessary to strike a balance between improved mechanical properties and decreased processability when selecting the fiber content.

The fiber orientation distribution is a very important structural parameter, because the enhancement of mechanical properties is normally associated with the direction of preferential fiber orientation. In a SFRTTP part, the fibers may be oriented in a random fashion leading to a macroscopically

isotropic material, or they may exhibit a preferential orientation in a given direction leading to significantly higher properties in that direction and hence an anisotropic material. These aspects are demonstrated, for a SF RTP system similar to that used in this study, in Figure 3.1 where L and T refer to the direction of preferential fiber orientation and the direction normal to it, respectively (31). In the case of a reinforced pipe, where the primary objective is to improve the hoop properties, it is thus desirable to promote fiber orientation in the hoop direction.

Particle aspect ratio (ratio of a major to a minor dimension of the particle) is another important structural parameter. In general, mechanical properties such as stiffness and strength in the direction of orientation increase with an increase in the fiber aspect ratio up to a certain value.

Fiber-matrix adhesion is also important in improving composite properties. Good bonding between the two phases is desirable for efficient load transfer across the interface.

One disadvantage of incorporating a brittle discrete phase such as glass fibers in a ductile polymeric matrix such as polyethylene is the associated embrittlement and consequent deterioration of the impact and fracture properties. An increase in the fiber content usually causes a reduction in the impact strength. Also, from a fracture mechanics point of view, it may

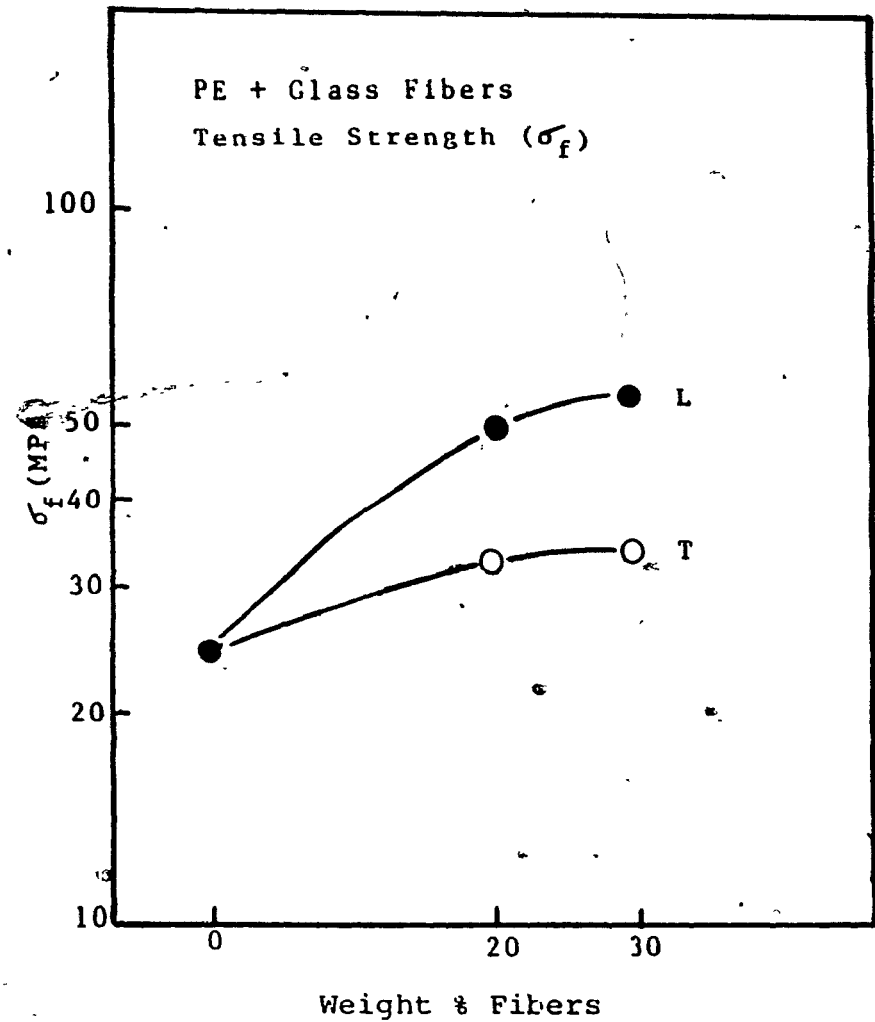
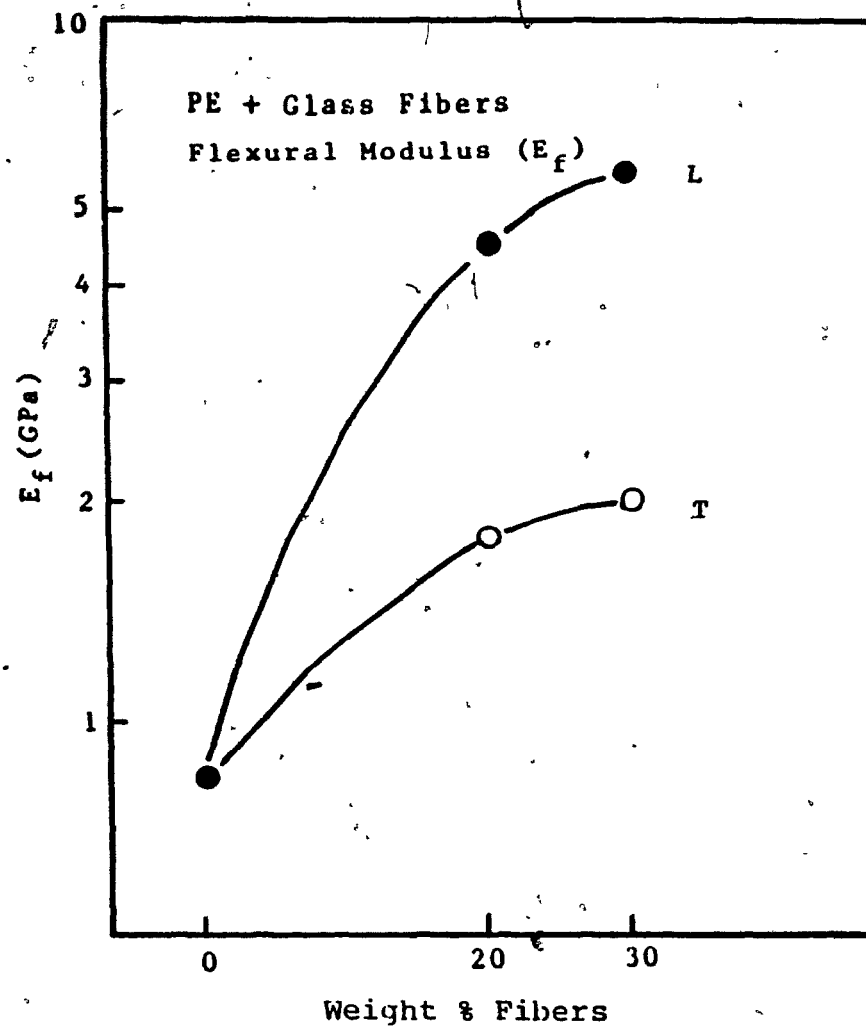


Figure 3.1 Variation of Flexural Modulus (E_f) and Tensile Strength (σ_y) of PE + Glass Fibers Composite with Fiber Fraction (31)

be undesirable to have excessively long fibers or exceptionally strong fiber-matrix bonding as both can suppress fiber pull out during the crack propagation in favor of fiber breakage and, thus, can cause a reduction in the absorption of energy during fracture (32).

3.1.1 Processing of SF RTP and its Influence on Properties

The greatest advantage of SF RTP over, for example, continuous filament reinforced materials, is their ease of processing. SF RTP materials can usually be processed using the same processing methods that are used for unreinforced thermoplastics. These include injection, transfer and compression molding, extrusion and thermoforming. Processing, however, involves flow of the material through complex geometries and gives rise to a complex orientation distribution of the suspended fibers. It also causes a significant fiber attrition. Since these are very important structural parameters governing the properties of the composite part, processing details strongly influence the final properties.

Fiber attrition corresponding to a reduction of the mean aspect ratio, though undesirable, is often unavoidable, and hence may be considered somewhat uncontrollable. Recently, several investigators have reported attempts to minimize such attrition (33,34).

Fiber orientation is strongly affected by several material and processing parameters. These include the rheological characteristics of the matrix, the processing equipment geometry and melt flow rate, which may give rise to a complex superposition of shear and extensional strain components during the flow. The fiber loading and aspect ratio govern the fiber-fiber and fiber-wall interactions and, thus, strongly influence the fiber orientation. The exact mechanism of fiber orientation is not fully understood, although several experimental and theoretical investigations of this phenomenon have been reported, as discussed below.

3.1.1.1 Experimental Studies

The studies reported in the literature have been carried out using widely differing composite systems, flow geometries and conditions. Many of these studies are qualitative in nature, since a convenient and rapid technique of quantitatively characterizing the fiber orientation distribution is not yet available (35,36).

An early attempt to study flow induced fiber orientation was that of Bell (37), who used a converging channel followed by a capillary die. Using an epoxy resin with 50% (by weight) fiber loading, he reported that the elongational flow in the converging section was important in aligning the fibers in the flow direction at the entrance to the capillary. Subsequent shearing

in the capillary did not affect the orientation significantly. Bell reported that the angle of convergence and the flow rate did not have a significant influence on fiber orientation.

On the other hand, Takano (38), using a rectangular channel converging into a slit die and an epoxy matrix based short fiber filled system reported that the degree of fiber orientation in the flow direction was strongly dependent on the angle of convergence and increased with flow rate. He also observed that the suspended fibers travelled with velocities somewhat lower than the local fluid velocities, depending on the fiber concentration.

Bright et al. (39) made qualitative observations of the effect of injection speed on the fiber orientation in rectangular injection molded bars of polypropylene containing 20% (by weight) glass fibers. At a low injection speed, the molded bars had a core having good fiber alignment in the flow direction and skins having a random fiber orientation. At a higher injection speed, the core had a predominantly transverse orientation and the skin had a significant fiber orientation in the flow direction. The authors attributed this to: (1) the divergent flow upon entry from the narrow gate into the wider mold cavity causing an extensional flow field, and (2) the shear thinning characteristics of the melt limiting the shear effects close to the surfaces. In another study, they used a capillary rheometer to show that the fiber alignment in the entrance region of the

capillary increases with an increase in flow rate and that the fibers can become almost perfectly aligned at a sufficiently high flow rate (40).

Goettler (41) suggested that the effect of fill rate on fiber orientation in his transfer molding experiments was strongly dependent on the gate geometry, which, in turn, controlled the degree of expansion upon entry into the mold cavity, and the matrix resin viscosity. The shear-thinning characteristics of the resin determined the thickness of the aligned fiber layers at the surfaces.

The extrusion of SF RTP systems has not been studied extensively. Knuttson et al. (42) reported that the degree of fiber orientation in the flow direction in a capillary flow increases with the wall shear rate, slowly at low shear rates but more sharply above a certain shear rate. --

Extrudates of SF RTP materials usually have a very rough surface. Wu (43) has reported that the surface texture of the extrudates obtained from a capillary die is dependent on the wall shear rate in the capillary. At low extrusion rates, the surface of the extrudates is relatively smooth, but becomes rough at higher rates. At even higher rates the surface again becomes smooth. He attributed this phenomenon to fiber migration in the extrudates.

Goettler (44) has reported that in the case of rubber stocks with cellulose fibers extruded through a slit die, the surface was smoother at low rates of extrusion becoming rougher at higher rates due to the fibers protruding out of the surface.

Favis et al. (45) have shown that the extrudates of mica-reinforced polypropylene are generally rough at low rates of extrusion. However, the degree of particle orientation in the direction of extrusion and the associated surface smoothness can be significantly improved under conditions of "starve feeding" to the extruder. Rémillard et al. (46) have explained this in terms of a reduction or elimination of voids in the extrudate produced under these conditions.

Cole et al. (47) have attributed the rough extrudate surface to the post-extrusion reorientation of the fibers initially oriented in the flow direction in the die, as a result of the molecular relaxation in the matrix resin.

3.1.1.2 Theoretical Studies

Despite the technological importance of concentrated particle suspensions in fluid media having various rheological characteristics, little success has been achieved in the theoretical treatment of their flow due to the mathematical complexity involved.

In a classical work, Jeffrey (48) related the torque resulting from the hydrodynamic stresses on a suspended rigid spheroid of revolution to its motion for an incompressible Newtonian medium in a creeping flow (low Reynold's number), neglecting the particle-particle and particle-boundary interactions and Brownian motion. His basic equations describing the particle motion, transformed to a fixed Cartesian frame of reference, have been given by Goldsmith and Mason (49) as follows:

$$\begin{aligned} \frac{d\phi}{dt} = & Z_1 - Z_2 \cos\phi \cot\psi - Z_3 \sin\phi \cot\psi + B [-D_{12} \sin\phi \cot\psi \\ & + D_{23} \cos 2\phi + D_{31} \cos\phi \cot\psi - \frac{1}{2} (D_{22} - D_{33}) \sin 2\phi] \end{aligned} \quad (3.1)$$

and

$$\begin{aligned} \frac{d\psi}{dt} = & -Z_2 \sin\phi + Z_3 \sin 2\phi + B [D_{12} \cos\phi \cos 2\psi + \\ & + \frac{1}{2} D_{23} \sin 2\phi \sin 2\psi + D_{31} \sin\phi \cos 2\psi \\ & + \frac{1}{4} (D_{22} - D_{33}) \cos 2\phi \sin 2\psi + \frac{3}{4} (D_{22} + D_{33}) \sin 2\psi] \end{aligned} \quad (3.2)$$

where ϕ and ψ are the angles defining the spatial orientation of the fibers, as shown in Figure 3.2. In the above equations, D_{ij} are the components of the stretch rate tensor, $\underline{D}$, given in the fixed Cartesian frame of reference as follows:

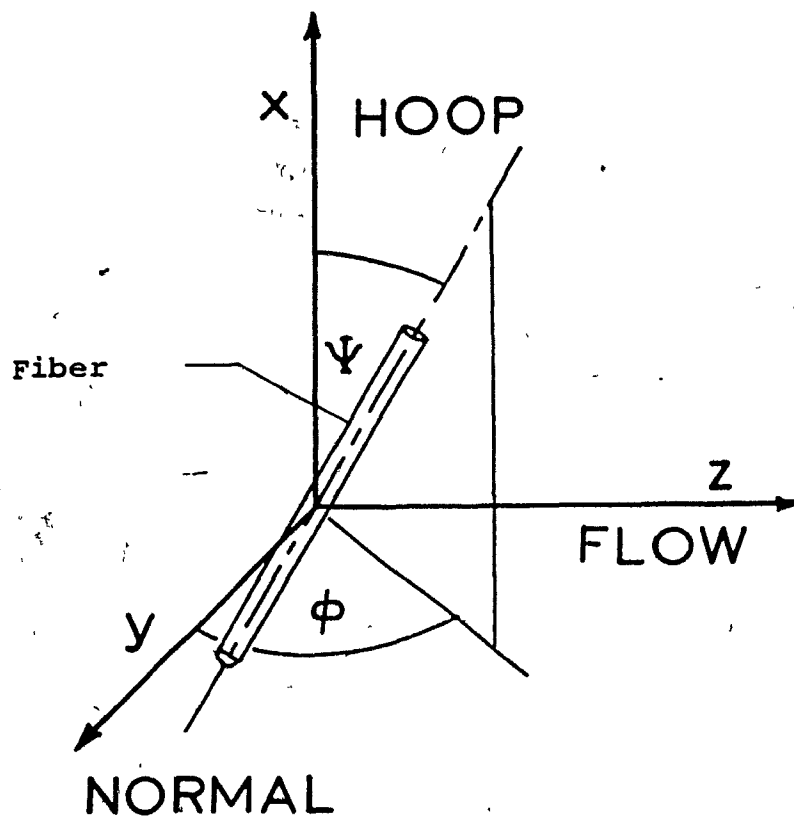


Figure 3.2 Angles ϕ and ψ Describing Fiber Orientation with respect to the Cartesian Frame of Reference.

$$\underline{D} = \frac{1}{2} \begin{bmatrix} 2 \frac{\partial u_x}{\partial x} & \frac{\partial u_x}{\partial y} + \frac{\partial u_y}{\partial x} & \frac{\partial u_x}{\partial z} + \frac{\partial u_z}{\partial x} \\ \frac{\partial u_x}{\partial y} + \frac{\partial u_y}{\partial x} & 2 \frac{\partial u_y}{\partial y} & \frac{\partial u_y}{\partial z} + \frac{\partial u_z}{\partial y} \\ \frac{\partial u_x}{\partial z} + \frac{\partial u_z}{\partial x} & \frac{\partial u_y}{\partial z} + \frac{\partial u_z}{\partial y} & 2 \frac{\partial u_z}{\partial z} \end{bmatrix} \quad (3.3)$$

and Z_k are related to the components of the vorticity tensor as follows:

$$\begin{aligned} Z_1 &= \frac{1}{2} \left(\frac{\partial u_z}{\partial y} - \frac{\partial u_y}{\partial z} \right) \\ Z_2 &= \frac{1}{2} \left(\frac{\partial u_x}{\partial z} - \frac{\partial u_z}{\partial x} \right) \\ Z_3 &= \frac{1}{2} \left(\frac{\partial u_y}{\partial x} - \frac{\partial u_x}{\partial y} \right) \end{aligned} \quad (3.4)$$

Also, B is a shape parameter related to the particle aspect ratio, r_p , as follows:

$$B = \left(\frac{r_p^2 - 1}{r_p^2 + 1} \right) \quad (3.5)$$

It may be noted that a short cylindrical fiber can be approximated as a slender prolate spheroid of revolution with an aspect ratio equal to the ratio of its length to diameter.

For a steady simple shear flow,

$$\vec{u} = (0, 0, \dot{\gamma}y) \quad (3.6)$$

Therefore, Equations (3.1) and (3.2) can be integrated to give

$$\tan \phi = r_p \tan\left(\frac{\dot{\gamma} t}{r_p + 1/r_p}\right) \quad (3.7)$$

and

$$\tan \psi = \frac{C \cdot r_p}{(r_p^2 \cos^2 \phi + \sin^2 \phi)^{1/2}} \quad (3.8)$$

where C is a constant of integration. Thus, in a shear flow, a prolate spheroid of revolution ($r_p > 1$) rotates spending more time orienting in the flow direction. The period of rotation depends on the particle aspect ratio, r_p .

For a planar elongational flow,

$$\vec{u} = (0, -\dot{\epsilon} y, \dot{\epsilon} z) \quad (3.9)$$

and, therefore, the fiber motion is described as follows:

$$\ln \tanh \phi = 2\dot{\epsilon} B(t - t_0) \quad (3.10)$$

$$\ln \tan \psi = - (1/2) \ln \sin^2 \phi + C' \quad (3.11)$$

C' being a constant of integration. Thus, a prolate spheroid tends to get oriented in the direction of stretching, z . Similarly, Takserman-Krozer and Ziabicki (50) have shown that for a uniaxial extensional flow field, elongated particles tend to get preferentially oriented in the principal direction of stretching.

Recently, Givler et al. (51,52) and Gillespie et al. (53) have employed Jeffrey's equations for predicting the planar (two-dimensional) orientation of suspended particles in a variety of complex flow situations such as fountain flow phenomenon in

injection molding, expansion at the entrance of a large cavity, and the flow around mold inserts.

Jeffrey's equations, however, are intended for purely viscous, dilute suspensions. The complexity of the constitutive equations and related computational difficulties make the development of a theory for the non-Newtonian fluids and concentrated suspensions very difficult (54-56).

Various studies described above appear to be somewhat inconclusive. Some general remarks can, however, be made. Extensional flow fields tend to orient suspended fibers in the direction of stretching, while shear fields tend to rotate them with a period dependent on the particle aspect ratio and the shear rate. Also, during this rotation, particles spend more time aligned close to the direction of shear than in the direction normal to that. Therefore, in the case of particles having sufficiently large aspect ratio, a large degree of fiber orientation in the flow direction can be expected in a shear field. The effects of the rheological characteristics of the matrix are not clearly understood, although shear-thinning appears to be useful if shear effects are to be suppressed. The effects of fiber loading, fiber interactions and migration and strain rate on the fiber orientation distribution are not clearly understood either.

An important point to be noted is that practical situations

such as the flow through dies and in mold cavities usually involve a complex superposition of shear and extensional flows. It is, therefore, important to understand the combined effects of these flow fields on fiber orientation so that the processing-structure-properties relationships of SF RTP composites can be used to an advantage.

3.2 Special Pipe Extrusion Die for the Control of Fiber Orientation Distribution

As noted in the above review, the flow field strongly affects the fiber orientation in SF RTP melt. In designing a die, it is therefore important to generate a flow field that promotes a desired orientation of suspended fibers.

Goettler (57,58) has taken such an approach in designing the extrusion dies for production of short-fiber-reinforced rubber hose. He reasoned that in the case of a highly concentrated suspension of cellulose fibers in a rubber stock, the shearing effects of the flow through the die are limited to the region near the wall due to the highly pseudoplastic nature of the suspension. The central core of the material, hence, flows essentially as a plug. Under these conditions, the fiber orientation in the core is highly responsive to the extensional flow field which can be generated by changing the cross-section in the direction of flow. Based on these arguments, Goettler has suggested the use of three specific dies, which he referred to as

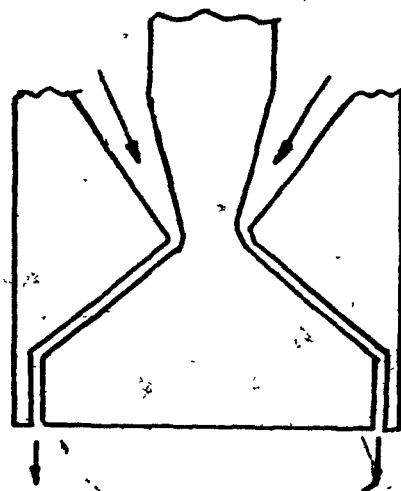
the expanding mandrel die, the obstructed flow die and the combination flow die, each giving rise to a different type of fiber orientation in the extrudate (59,60).

The geometry of the expanding mandrel die is shown in Figure 3.3a. The die walls in the conically expanding annular gap are parallel to each other, and the channel gap remains constant. However, the cross-sectional area normal to the flow increases, giving rise to a deceleration in the flow direction and an associated stretching in the circumferential or hoop direction. Such a die is claimed to produce a preferential hoop orientation of the suspended fibers (59,60).

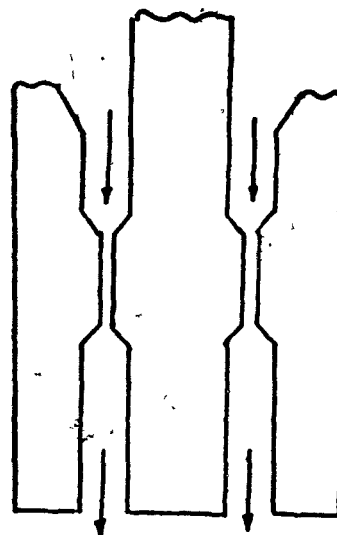
The obstructed flow die is shown schematically in Figure 3.3b. The expansion in the thickness direction of the channel following an obstruction is claimed to orient the fibers preferentially in the radial direction (60).

The combination die, shown in Figure 3.3c, incorporates expansion in both the thickness and the circumferential directions (60). Such a geometry gives rise to combined radial and hoop orientation of fibers.

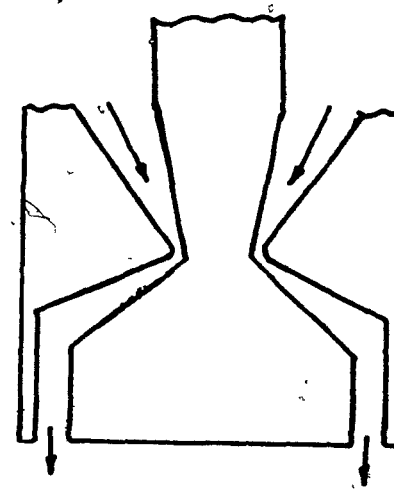
In the present study, it was decided to use an expanding channel die similar to that suggested by Goettler (Figure 3.3a) for promoting the hoop orientation of fibers in the SFRTM melt during pipe extrusion. Other approaches such as the rotating



Expanding Mandrel Die
(a)



Obstructed Flow Die
(b)



Combination Die
(c)

Figure 3.3 Various Die Geometries Proposed by Goettler for Preferential Fiber Orientation.

mandrel die can also be considered for this purpose, however, the expanding channel die was selected for its simplicity of design and operation. It must be noted that there are several differences between the rheological characteristics of the cellulose fiber filled rubber stock used by Goettler and those of the SF RTP used in the present study. The fiber aspect ratio and fiber concentration are also significantly lower in the case of SF RTP. Since these factors can influence fiber orientation distribution significantly, it was decided to study first the fiber orientation developed by simple extrusion of SF RTP through an expanding channel die both theoretically and experimentally.

In the following, a theoretical model is developed in order to predict the fiber orientation distribution at the die exit. The general conclusions of this model are subsequently verified by the results of an experimental study of extrusion through such a die.

3.3 Theoretical Analysis of Flow-Induced Fiber Orientation in an Expanding Channel Tubing Die

As mentioned in Section 3.1.1.2, a rigorous simulation of the flow of a concentrated suspension of nonspherical particles in a viscoelastic medium such as a polymer melt is not possible at the present time, although several models have been developed by making certain simplifying assumptions. Quantitative treatments are generally based on the equations derived by Jeffrey for

describing the motion of a spheroid suspended in a Newtonian medium subjected to an arbitrary homogeneous flow field, i.e., a flow field in which the velocity gradients are spatially uniform. Goldsmith and Mason have, however, demonstrated experimentally that these equations can also be used for nonhomogeneous flow fields such as Poiseuille flow, provided that the local values of the components of the rate of deformation tensor are used, and that the particle dimensions are small compared to the flow channel (61).

Several investigators have used Jeffrey's equations to predict fiber orientation in complex flow situations (51-53,62,63). These analyses usually neglect particle-particle and particle-wall interactions, and often assume the polymer melt as a Newtonian fluid. Models attempting to incorporate fiber interactions make use of certain empirically derived parameters that are process and composite system dependent, and thus are unable to provide generally valid predictions (64,65). In the following, the flow of a polymer melt through the expanding channel die is analyzed, and the fiber orientation distribution at the die exit is predicted on the basis of Jeffrey's equations.

Three basic assumptions are made at the outset. The fiber is modelled as a highly elongated, axisymmetric ellipsoid (slender prolate spheroid). Particle-particle and particle-wall interactions are neglected. Finally, on a global scale, the suspension is assumed to be a power-law fluid described by the

following tensorial constitutive equation.

$$\underline{\tau} = K(I\dot{I}/2)^{(n-1)/2} \cdot \underline{\dot{A}} \quad (3.12)$$

where K and n are the consistency and power-law index, respectively, and $\underline{\tau}$ and $\underline{\dot{A}}$ are the stress and rate of deformation tensors, respectively. In the vicinity of a particle, the medium is considered to be Newtonian. This somewhat crude assumption, referred to as the "pseudo non-Newtonian" assumption, allows a realistic estimation of the effect of shear flow on the particle motion.

The analysis consists of three steps: the derivation of the flow field, the solution of Jeffrey's equations for the derived flow field to describe the motion of an individual fiber along the flow path, and the determination of the fiber orientation distribution at the die exit for a given initial orientation distribution at the entrance. Some additional assumptions have been made in the course of development of the model. The implications of these assumptions are discussed in the Appendix 3.1.

3.3.1 Derivation of the Flow Field

The flow geometry considered here is shown schematically in Figure 3.4. The mean die radius is R_0 at the entrance and R_1 at the exit. The angle of expansion is α , and the die gap, $2H$, is constant. A Cartesian frame of reference is chosen with its

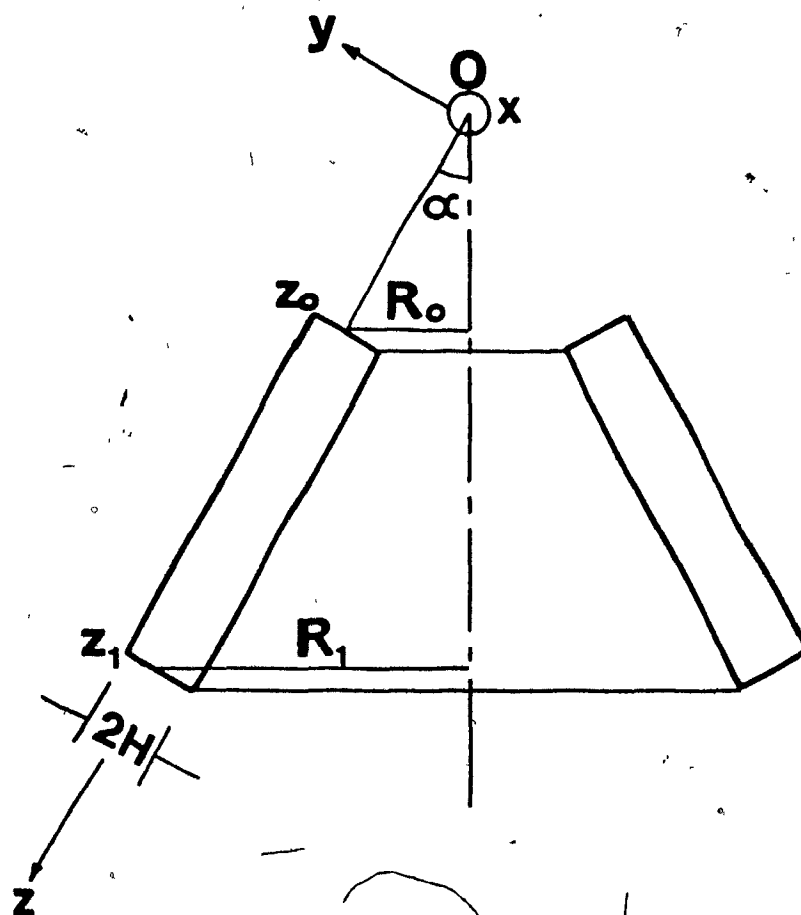


Figure 3.4 Axial Section of the Expanding Channel Die.

origin at point 0 (Figure 3.4). The x-axis is in the hoop direction, the y-axis is normal to the channel wall, and the z-axis is along the channel center line in the flow direction. $z=z_0$ corresponds to the entry to the expanding section, while $z=z_1$ corresponds to the exit from it.

The lubrication approximation is used to derive the following expression for the z-direction velocity, u_z .

$$u_z = \left[\frac{H}{K} \left(-\frac{dP}{dz} \right) \right]^{1/n} \left(\frac{H}{n+1} \right) \left[1 - \left(\frac{y}{H} \right)^{1+1/n} \right] \quad (3.13)$$

It must be noted that in using the symmetric velocity profile given by the above equation, the curvature of the annular geometry has been ignored. This can be justified on the grounds that the thickness of the channel is small compared to the channel radius. Also, the pressure gradient $(-dP/dz)$ in the above equation is the local pressure gradient. It is not constant, but varies with z due to the expansion in the flow area along the die.

The overall mass balance across the die gap is given as:

$$Q = \int_A u_z dA = 4\pi H^2 z \sin \alpha \left(\frac{n}{2n+1} \right) \left[\frac{H}{K} \left(-\frac{dP}{dz} \right) \right]^{1/n} \quad (3.14)$$

Elimination of the pressure gradient term between Equations 3.13 and 3.14 gives the following expression for the velocity u_z as a function of the distances along the channel (z) and across the die gap (y).

$$u_z = \left[\frac{Q}{4\pi H z \sin \alpha} \right] \left(\frac{2n+1}{n+1} \right) \left[1 - \left(\frac{y}{H} \right)^{1+1/n} \right] \quad (3.15)$$

It should be noted that the flow field given by the above equation involves a superposition of shear and extensional deformation components. The rate of deformation tensor is as follows:

$$\underline{\Delta} = \begin{bmatrix} 2 \frac{\partial u_x}{\partial x} & 0 & 0 \\ 0 & 0 & \frac{\partial u_z}{\partial y} \\ 0 & \frac{\partial u_z}{\partial y} & 2 \frac{\partial u_z}{\partial z} \end{bmatrix} \quad (3.16)$$

where,

$$\frac{\partial u_z}{\partial z} = - \frac{\partial u_x}{\partial x} = - \left[\frac{Q}{4\pi H z^2 \sin \alpha} \right] \left(\frac{2n+1}{n+1} \right) \left[1 - \left(\frac{y}{H} \right)^{1+1/n} \right] \quad (3.17)$$

and

$$\frac{\partial u_z}{\partial y} = - \left[\frac{Q}{4\pi H^2 z \sin \alpha} \right] \left(\frac{2n+1}{n} \right) \left(\frac{y}{H} \right)^{1/n} \quad (3.18)$$

3.3.2 Individual Fiber Motion

The orientation of an individual fiber in space is characterized by two angles, ϕ and ψ , which are defined in Figure 3.2. Jeffrey's equations, as given by Mason et al. (49), describe the motion of a suspended particle in terms of the rates of change of angles ϕ and ψ as functions of the parameters of the

flow field. These equations, which have been given earlier, are repeated here for convenience.

$$\frac{d\phi}{dt} = Z_1 - Z_2 \cos\phi \cot\psi - Z_3 \sin\phi \cot\psi + B \left[-D_{12} \sin\phi \cot\psi + D_{23} \cos 2\phi + D_{31} \cos\phi \cot\psi - \frac{1}{2} (D_{22} - D_{33}) \sin 2\phi \right] \quad (3.1)$$

$$\frac{d\psi}{dt} = -Z_2 \sin\phi + Z_3 \sin 2\phi + B \left[D_{12} \cos\phi \cos 2\psi + \frac{1}{2} D_{23} \sin 2\phi \sin 2\psi + D_{31} \sin\phi \cos 2\psi + \frac{1}{4} (D_{22} - D_{33}) \cos 2\phi \sin 2\psi + \frac{1}{4} (D_{22} + D_{33}) \sin 2\psi \right] \quad (3.2)$$

For the flow field given by Equation 3.15, Jeffrey's equations may be transformed into the relations describing the rates of change of the angles ϕ and ψ along the flow streamlines, which in the present case are parallel to the z-axis.

$$\frac{d\phi}{d(z/H)} = - \left[\left(\frac{n+1}{2n} \right) \frac{(y/H)^{1/n}}{1 - (y/H)^{1+1/n}} (1 + B \cos 2\phi) + \frac{B \sin 2\phi}{2 (z/H)} \right] \quad (3.19)$$

$$\frac{d\psi}{d(z/H)} = - \frac{B}{4} \left[\left(\frac{n+1}{n} \right) \frac{(y/H)^{1/n}}{1 - (y/H)^{1+1/n}} \sin 2\phi \sin 2\psi + \frac{\cos 2\phi \sin 2\psi}{(z/H)} + \frac{3 \sin 2\psi}{(z/H)} \right] \quad (3.20)$$

For a given initial orientation, (ϕ_0, ψ_0) , at the entrance, the change of orientation along a streamline, and particularly, the orientation at the exit, (ϕ_1, ψ_1) , can be determined by solving the above equations numerically. It may be noted that neither Equation 3.19 nor 3.20 contains the flow rate Q . Thus, this model predicts that the fiber orientation is independent of the flow

rate.

3.3.3 Fiber Orientation Distribution

An actual suspension contains a large number of fibers, each fiber having its own orientation at the entrance, denoted here by the subscript "0", and as a result of the flow it achieves a final orientation at the exit, denoted here by the subscript "1". For an initial orientation distribution at the entrance, $f_0(\phi_0, \psi_0)$, the corresponding final orientation distribution at the exit, $f_1(\phi_1, \psi_1)$, can be determined as follows.

The angle ϕ describes the plane in which fibers are lying with respect to the y-axis and, thus, with respect to the shear plane, while the angle ψ describes the fiber orientation with respect to the hoop direction. The range of values for ϕ is from 0 to 2π , while that for ψ is from 0 to $\pi/2$. Equation 3.19 is independent of ψ and, therefore, all the fibers having a certain value of angle ϕ_0 at the entrance will have the same value of angle ϕ_1 at the exit. In order to evaluate the 3-dimensional orientation distribution, it is convenient to consider several planes corresponding to various initial values, $\phi_0(i)$ ($i=1, N$). For each of these planes, an initial distribution $f_0(\phi_0, \psi_0)$ with respect to angle ψ_0 can be considered. All fibers corresponding to this plane end up lying in a plane $\phi_1(i)$ in accordance with Equation 3.19. The final orientation distribution of these fibers, $f_1(\phi_1, \psi_1)$, in the plane $\phi_1(i)$ can then be determined using the

following procedure.

For a selected plane, $\phi_0(i)$, the range of values of ψ is divided into several intervals as follows:

$$\Delta \psi_0(j) = \psi_0(j) - \psi_0(j-1) \quad (j=2, M) \quad (3.21)$$

where M is the number of interval. For each interval, a representative value $\psi_0(j)$ is chosen. For the given initial orientation $(\phi_0(i), \psi_0(j))$, the final orientation $(\phi_1(i), \psi_1(j))$ is determined by solving Equations 3.19 and 3.20 numerically. The procedure is repeated for each interval $\Delta \psi_0(j)$. The corresponding final intervals $\Delta \psi_1(j)$ at the exit are determined as follows:

$$\Delta \psi_1(j) = \psi_1(j) - \psi_1(j-1) \quad (j=2, M) \quad (3.22)$$

The final orientation distribution $f_1(\psi_1)$ is then obtained as follows:

$$f_1(\psi_1) = \frac{\Delta \psi_0(j)}{\Delta \psi_1(j)} f_0(\psi_0) \quad (3.23)$$

This distribution function describes the orientation with respect to the hoop direction, at the exit, of those fibers that lie at the entrance in the plane described by a certain value of $\phi_0(i)$, and at the exit in the plane described by the corresponding value of $\phi_1(i)$. This procedure is repeated for all values of $\phi_0(i)$, and a quantitative 3-dimensional description of the fiber orientation distribution is thus obtained.

In the results presented below, the primary goal is to obtain the orientation distribution with respect to the hoop direction described by angle ψ at the exit. Moreover, the calculations

indicate that irrespective of the initial plane of orientation ($\phi_0(i)$), almost all the fibers end up lying nearly parallel to the shear plane. Consequently, it is useful to calculate the following overall distribution function with respect to ψ .

$$\bar{f}(\psi) = \frac{\int f_1(\phi, \psi) d\phi}{\int d\phi} \quad (3.24)$$

A high value of $\bar{f}(\psi)$ near $\psi = 0^\circ$ indicates a strong hoop orientation of fibers, while a high value of $\bar{f}(\psi)$ near $\psi = 90^\circ$ indicates a strong axial orientation fibers.

For all the results presented below, the initial orientation distribution at the entrance was assumed to be uniformly random implying that there was an equal chance of finding a fiber oriented in any given direction at this location, i.e.,

$$f_0(\psi_0) = 1 \quad (3.25)$$

for all $\phi_0(i)$. It may be noted that in an actual die, the melt is subjected to flow before reaching the expanding section giving rise to a fiber orientation distribution that may not be random, particularly near the wall. The above choice was, however, made for the purpose of illustration.

A FORTRAN computer program was developed to carry out the above calculations. A listing of this program is given in Appendix 3.2.

3.3.4 Model Predictions

In all the results presented below, the value of the shape parameter B was chosen as 1, which corresponds to a very high aspect ratio of fibers. Also, two values of the power-law index n are considered; n=0.5, representing a shear-thinning medium, and n=1, corresponding to a Newtonian medium. Figures 3.5 and 3.6 correspond to the following die design parameters:

$$R_0/H = 12, \quad R_1/R_0 = 8/3, \quad \alpha = 60^\circ$$

Figures 3.7 through 3.10 show the effect of variations in these parameters on the fiber orientation at the exit.

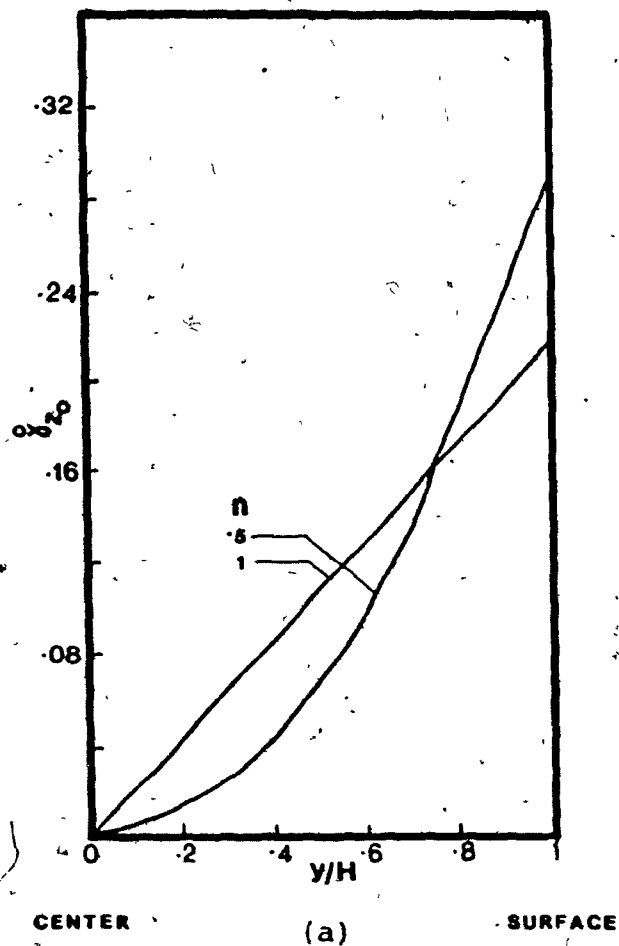
The shear and extensional strain rates across the die gap are given by Equations (3.17) and (3.18). These can be de-dimensionalized with respect to the flow rate as follows:

$$\left(\frac{\partial u_z}{\partial z}\right)^* = \frac{4\pi H^2 z \sin \alpha}{Q} \left(\frac{\partial u_z}{\partial z}\right) = \frac{1}{(z/H)} \left(\frac{2n+1}{n+1}\right) \left[1 - \left(\frac{y}{H}\right)^{1+1/n}\right] \quad (3.26)$$

$$\left(\frac{\partial u_z}{\partial y}\right)^* = \frac{4\pi H^2 z \sin \alpha}{Q} \left(\frac{\partial u_z}{\partial y}\right) = - \left(\frac{2n+1}{n}\right) \left(\frac{y}{H}\right)^{1/n} \quad (3.27)$$

Figure 3.5a shows a representative shear rate profile across the die gap at the entry to the expanding section, where $z=z_0$. For both the shear-thinning and the Newtonian cases, the shear rate is zero at the centerline and increases toward the die wall. Figure 3.5b shows the corresponding profiles for the extensional strain rate in the hoop direction. The extensional strain rate is maximum at the centerline and reduces to zero at the die wall. It is thus expected that the effect of hoop stretching will be dominant in the core region whereas that of shearing will

Shear Rate



Extensional Rate

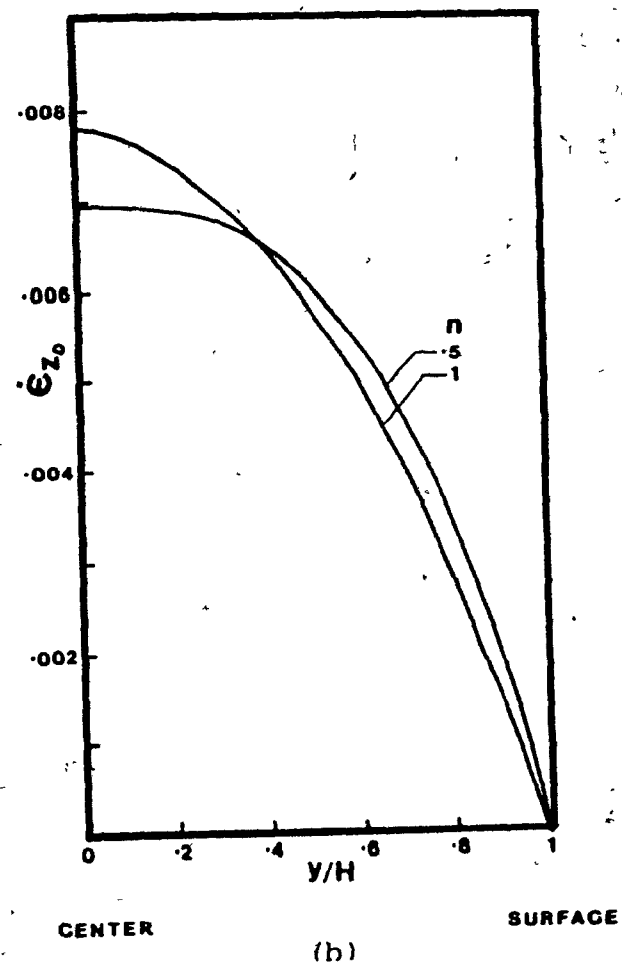


Figure 3.5a Dimensionless Shear Rate Profile across the Die Gap at $z=z_0$
 3.5b Dimensionless Extensional Rate Profile across the Die Gap at $z=z_0$

predominate near the die wall.

For comparison purposes, three locations across the die gap, $y/H=0$ (center line), $y/H=0.25$ (near-center) and $y/H=0.5$ (midway) were selected to present the fiber orientation distribution functions. Figure 3.6 shows the predicted fiber orientation functions $\bar{f}(\psi)$ at these three locations for both the shear-thinning ($n=0.5$) and Newtonian ($n=1$) cases. It can be seen from Figure 3.6a that, at the center line ($y/H=0$), a strong hoop orientation of fibers is developed as indicated by a large peak of the distribution function at $\psi=0^\circ$. Also, there is no difference between the predictions for the shear-thinning and the Newtonian cases, as the shear rate is zero along the center line. On the other hand, as seen in Figure 3.6c, at the midway location ($y/H=0.5$), both the shear-thinning and Newtonian cases show a strong axial fiber orientation indicated by a peak in the distribution function at $\psi=90^\circ$. Due to a somewhat lower shear rate for the shear-thinning case, the degree of axial orientation is also somewhat lower. The near-center location ($y/H=0.25$) represents an intermediate case. As can be seen in Figure 3.6b, while the Newtonian case gives a preferred axial orientation of fibers, the shear-thinning case shows a mild peak in the hoop direction at this location.

Thus, the above results show that in the presence of combined shear and extension, there is a variation in the fiber orientation pattern from the center to the surface of the

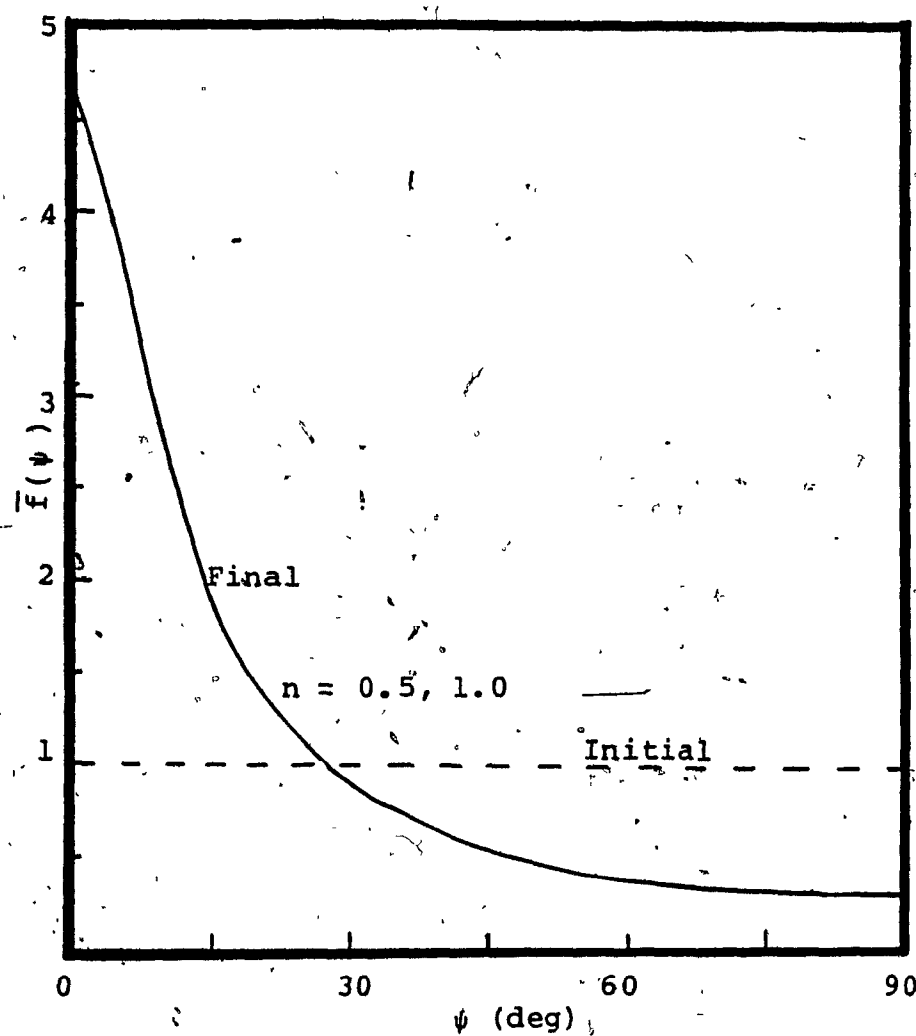


Figure 3.6a Fiber Orientation Distribution at
 $y/H = 0$ (Center Line)
 $(\alpha = 60^\circ, R_1/R_0 = 8/3, R_0/H = 12)$

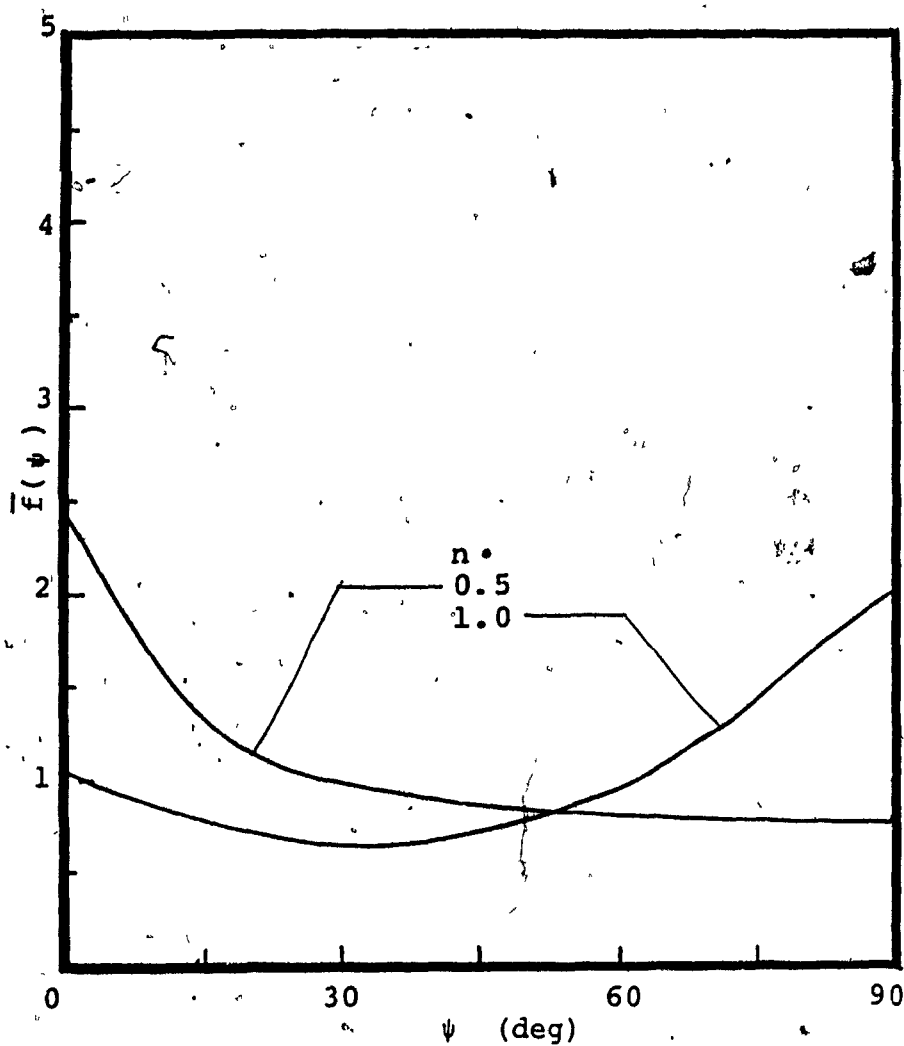


Figure 3.6b Fiber Orientation Distribution at
 $y/H = 0.25$ (Near-Center)
 $(\alpha = 60^\circ, R_1/R_0 = 8/3, R_0/H = 12)$

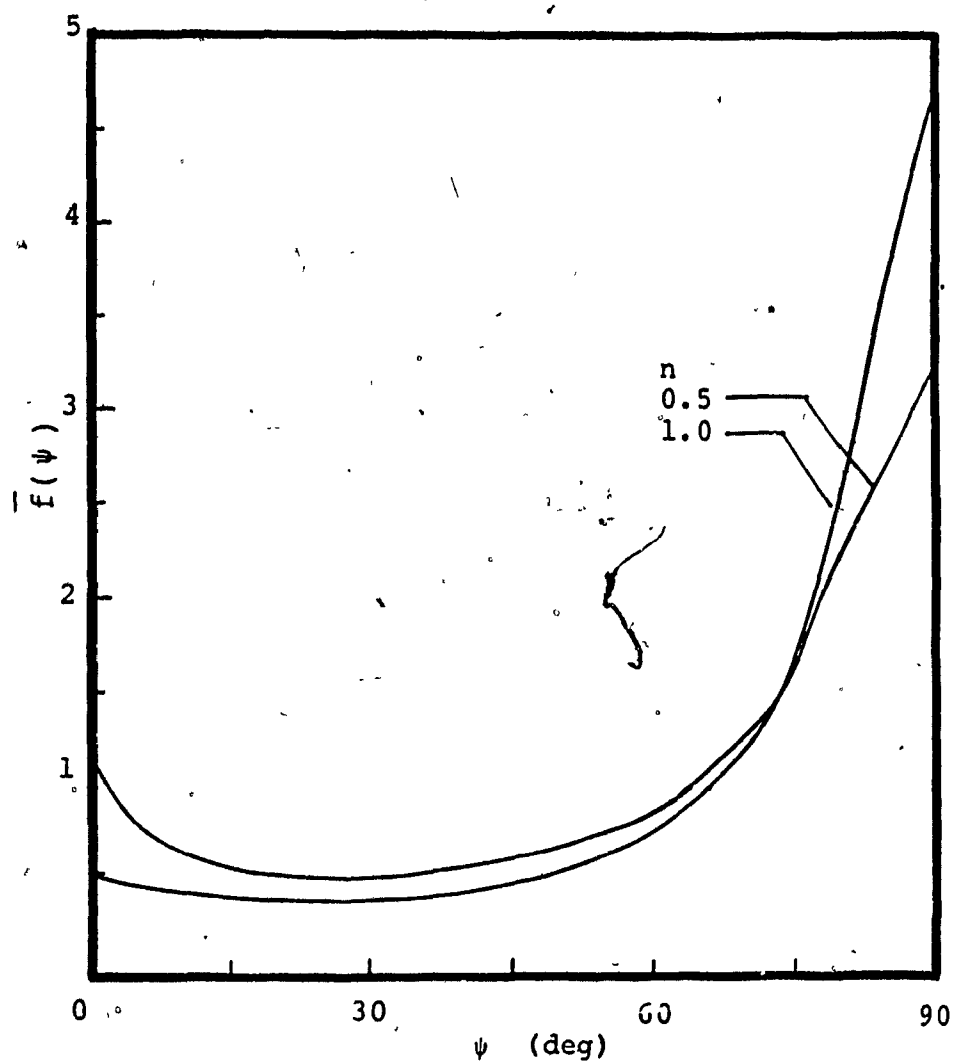


Figure 3.6c Fiber Orientation Distribution at
 $y/H = 0.5$ (Midway)
 $(\alpha = 60^\circ, R_1/R_0 = 8/3, R_0/H = 12)$

extrudate. While a strong hoop orientation of fibers is developed in the core region, the orientation pattern changes toward the surface where a predominantly axial orientation is developed. Also, the relative thickness of the hoop-oriented core depends on the shear-thinning characteristics of the resin.

Besides resin rheology, geometrical parameters also play an important role in determining the overall structure of the extrudate obtained using an expanding channel die. In Figures 3.7 to 3.10, the effect of die geometry on the fiber orientation distribution is illustrated in order of decreasing influence.

Figures 3.7 shows the effect of channel thickness on the fiber orientation distribution for the same degree of expansion ($R_1/R_0=8/3$) and angle of expansion ($\alpha=60^\circ$) as before. A greater degree of hoop orientation at the near-center location, $y/H=0.25$ and a lower degree of axial orientation at the midway location, $y/H=0.5$, are predicted for the thicker channel ($R_0/H=6$), as compared to the thinner channel ($R_0/H=12$). The results for the center line, $y/H=0$, remain unchanged, as the overall degree of expansion is unchanged.

The effect of the change in angle of expansion, α , keeping the degree of expansion ($R_1/R_0=8/3$) and the relative channel thickness ($R_0/H=12$) constant is shown in Figure 3.8. A smaller angle of expansion, α , for a given expansion ratio R_1/R_0 implies a longer flow path and hence more shearing in the region away

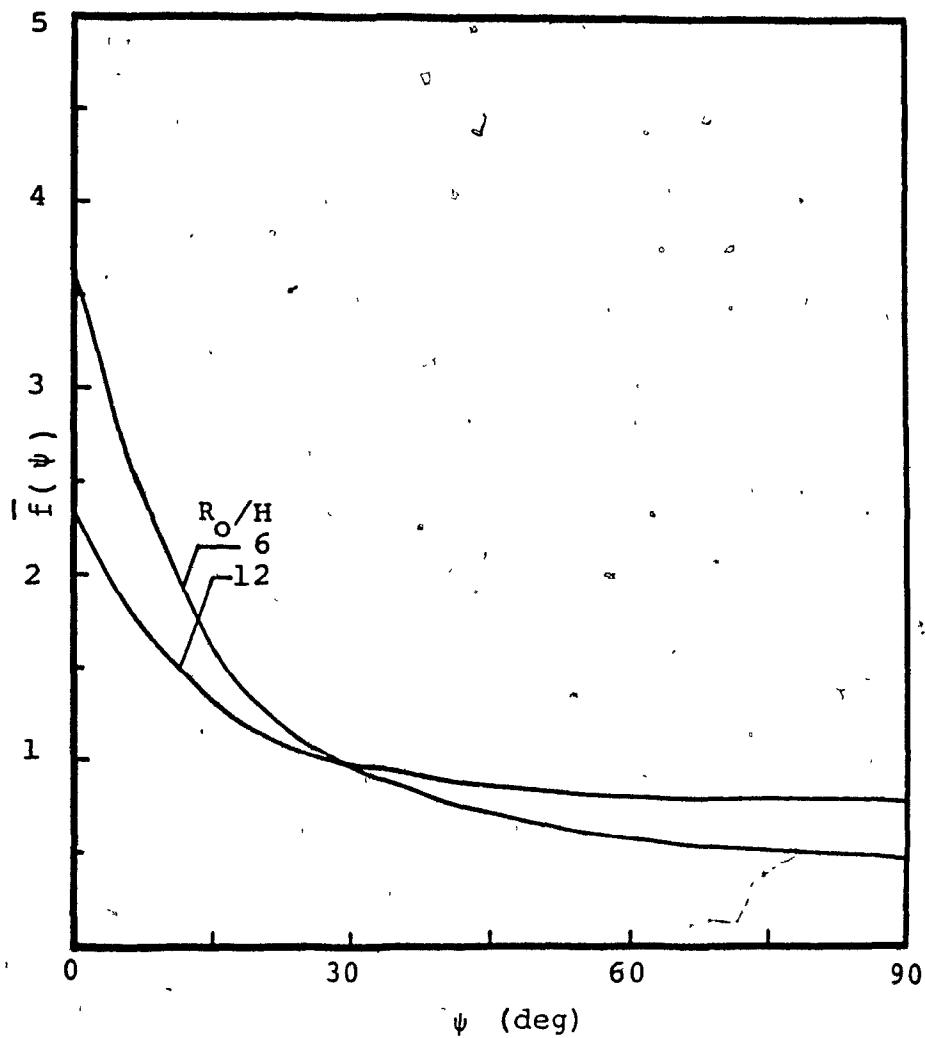


Figure 3.7a Effect of the Change in the Relative Channel Thickness on Fiber Orientation Distribution.

$y/H = 0.25$ (Near-Center)

$(R_1/R_o = 8/3, \alpha = 60^\circ, n = 0.5)$

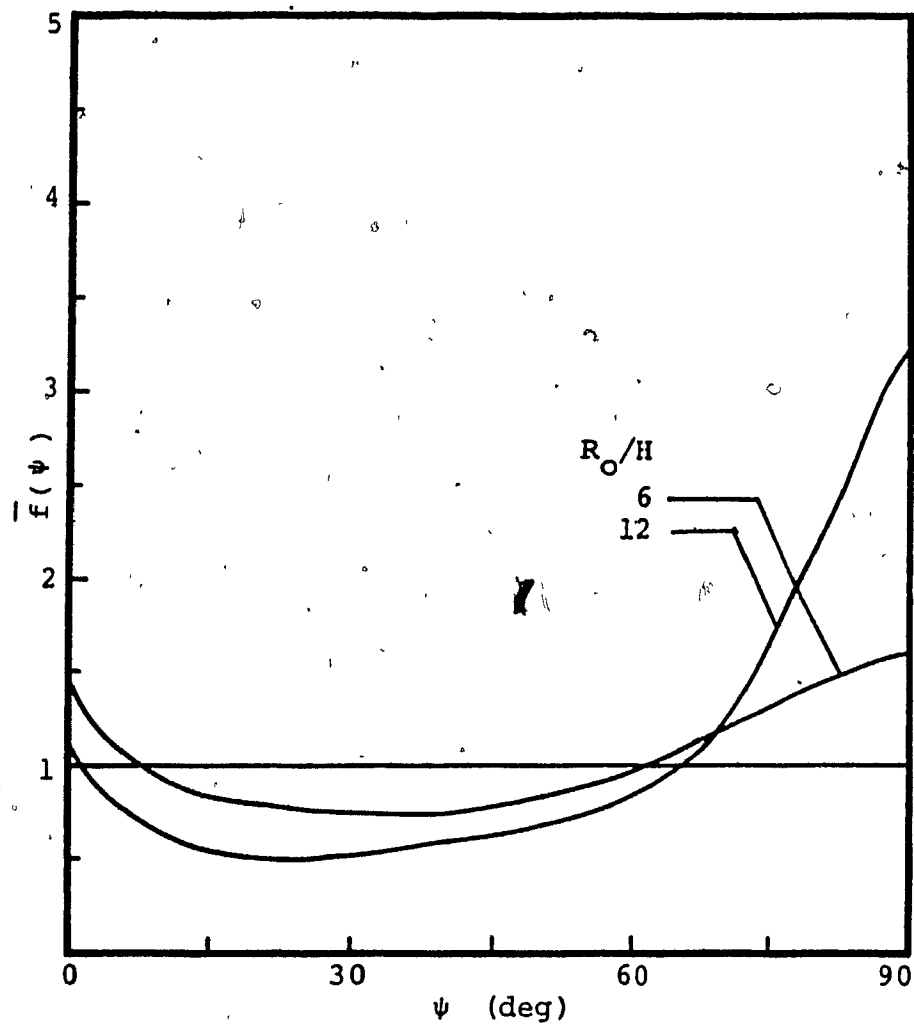


Figure 3.7b Effect of the Change in the Relative Channel Thickness on Fiber Orientation Distribution.

$y/H = 0.5$ (Midway)

$(R_1/R_0 = 8/3, \alpha = 60^\circ, n = 0.5)$

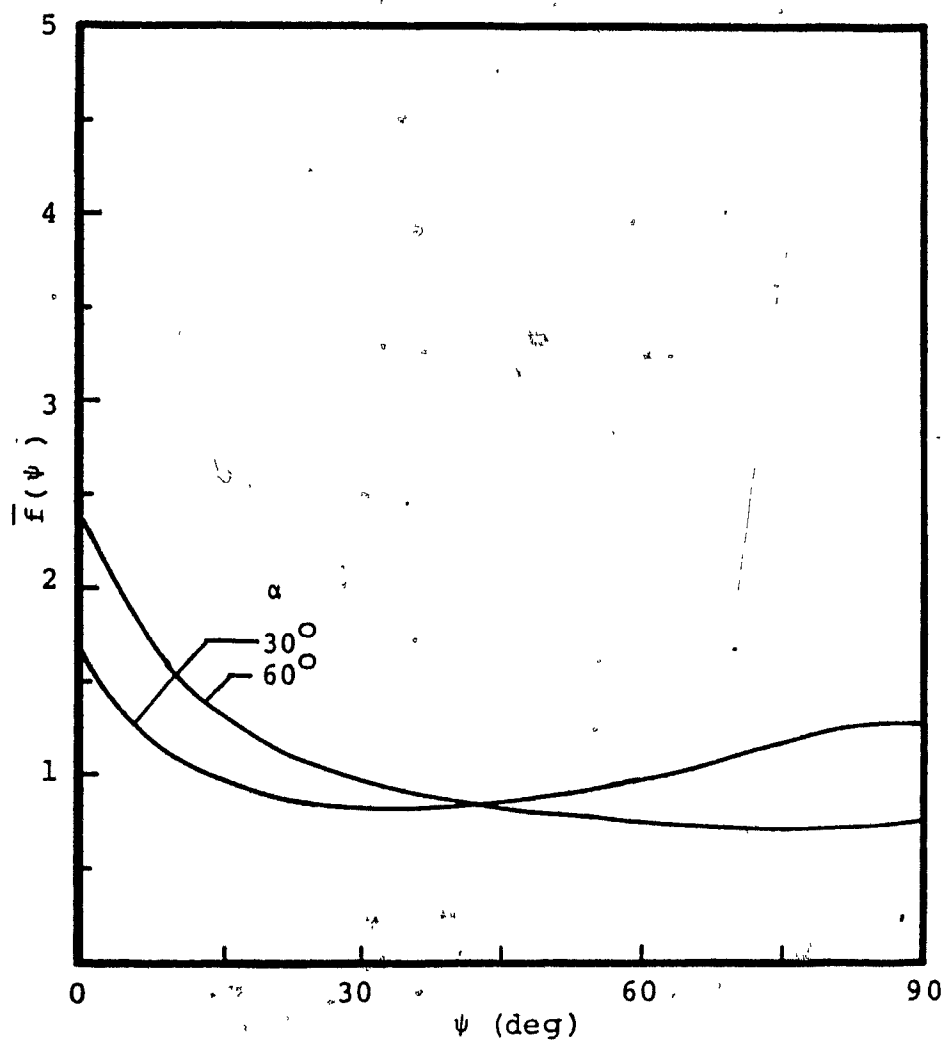


Figure 3.3a Effect of the Change in the Angle of Expansion (α) on Fiber Orientation Distribution.

$y/H = 0.25$ (Near-Center)

$(R_1/R_0 = 8/3, R_0/H = 13, n = 0.5)$

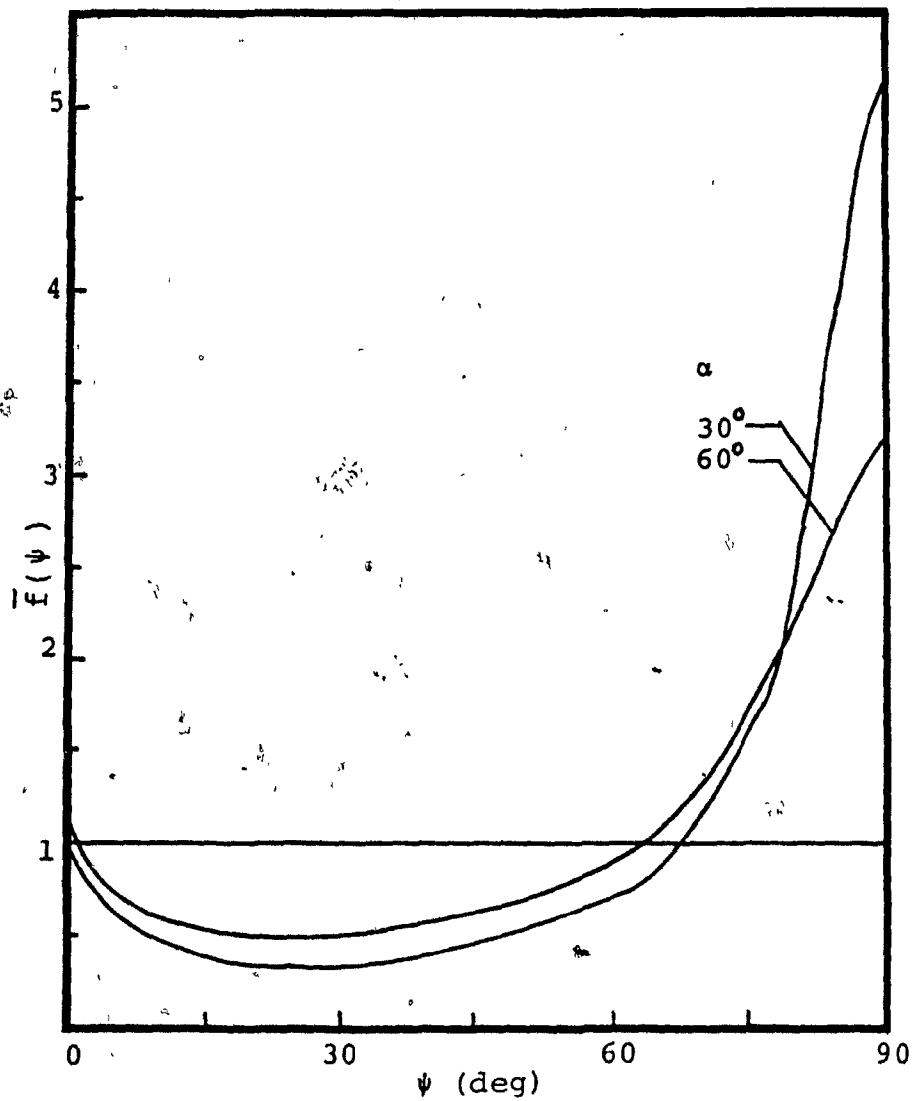


Figure 3.8b Effect of the Change in the Angle of Expansion (α) on Fiber Orientation Distribution.

$y/H = 0.5$ (Midway)

($R_1/R_0 = 8/3$, $R_0/H = 12$, $n = 0.5$)

from the centerline. Consequently, a smaller angle of expansion ($\alpha=30^\circ$) leads to a lower degree of hoop orientation and a higher extent of axial orientation at the near-center and midway locations.

As a point of reference, it is interesting to look at the fiber orientation developed in a shear flow without hoop stretching, such as that produced in a straight die of equivalent flow length (the distance z_0 to z_1). At the center line of the straight die channel, ($y/H=0$), there is no change in the initial fiber orientation due to the absence of shear as well as extensional components of flow (Figure 3.9a). At locations away from the center line, the straight die produces a predominantly axial orientation of fibers, as shown in Figures 3.9b and 3.9c.

Figure 3.10 shows the effect of a change in the degree of expansion (R_1/R_0) on the fiber orientation distribution keeping the other geometrical parameters constant ($\alpha=60^\circ$, $R_0/H=12$). It can be seen that an increase in the degree of expansion by a factor of two from $R_1/R_0=8/3$ to $R_1/R_0=16/3$ does not have a significant effect on the orientation distribution, particularly at locations away from the centerline. This implies that slender particles in a flow involving both shear and extension acquire an ultimate orientation within a certain length dictated by the hydrodynamic forces, and that subsequent flow does not produce any significant further change. A practical consequence of this is that a die with a higher degree of expansion may not produce a

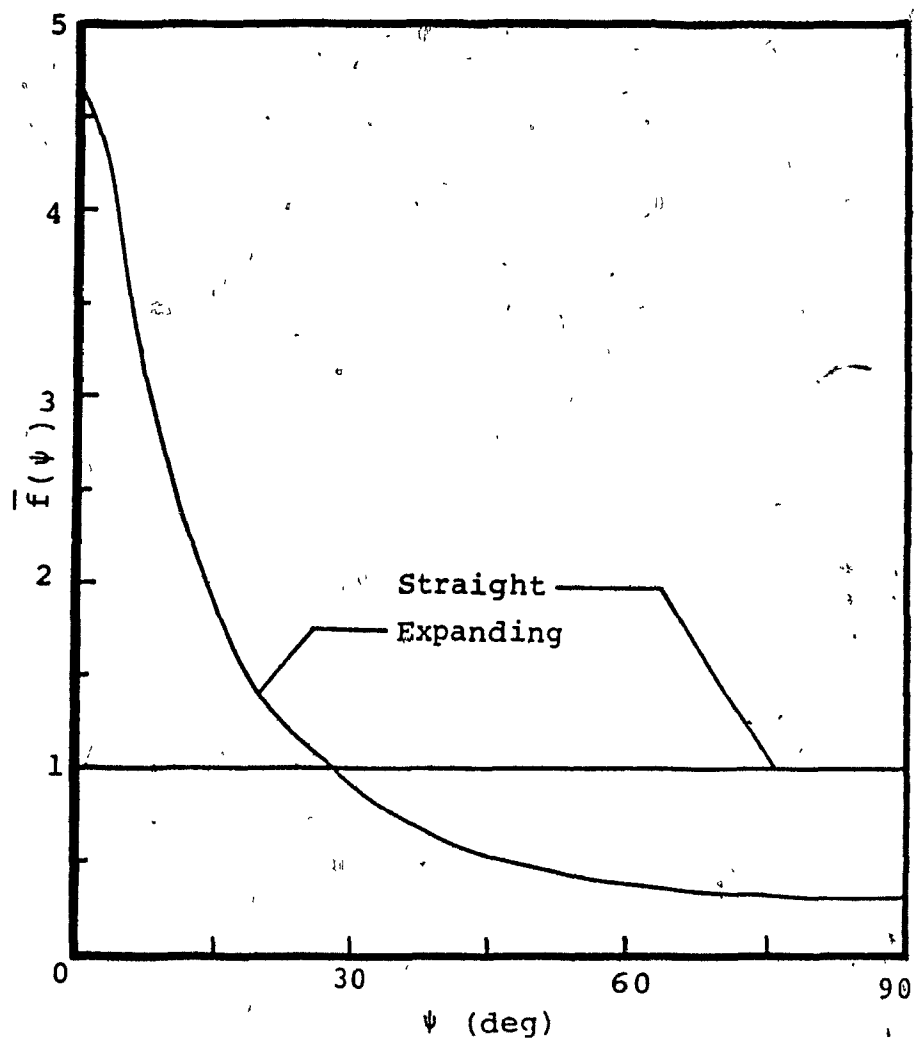


Figure 3.9a Comparison of Fiber Orientation Distributions
Produced by Expanding and Straight Dies.
 $y/H = 0$ (Center Line)

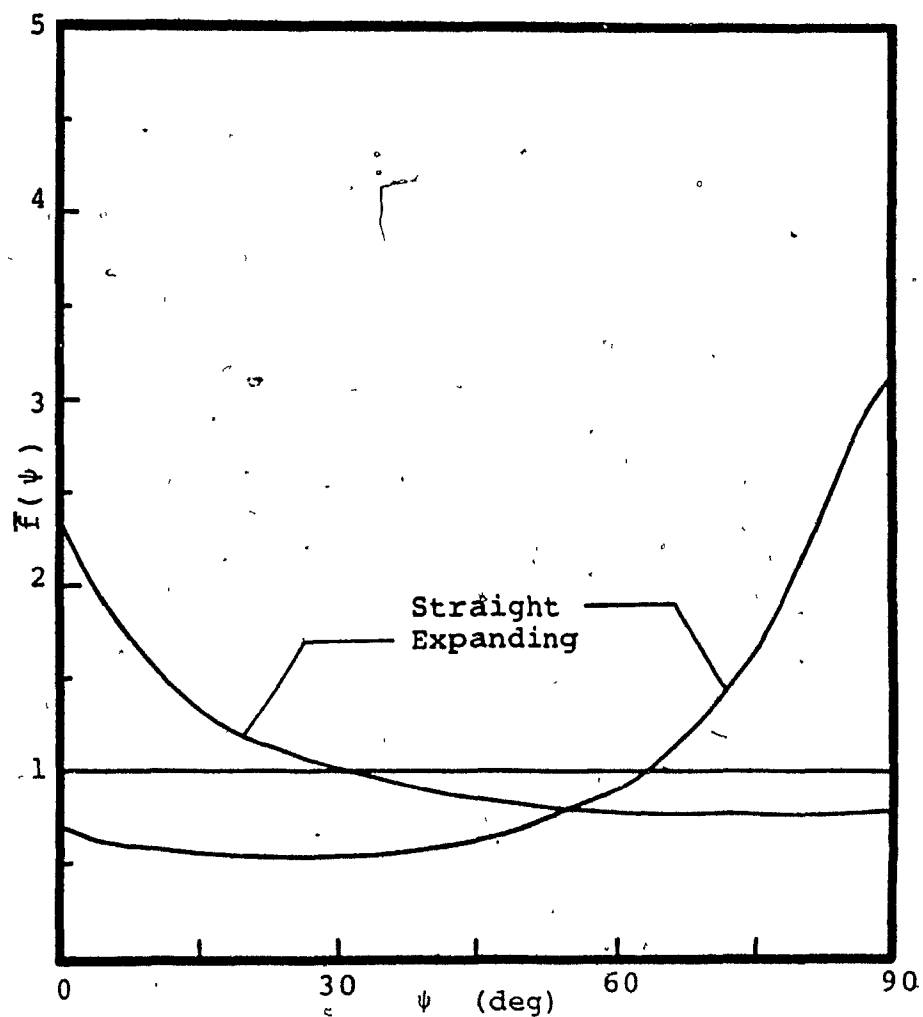


Figure 3.9b Comparison of Fiber Orientation Distributions
Produced by Expanding and Straight Dies.
 $y/H = 0.25$ (Near-Center)

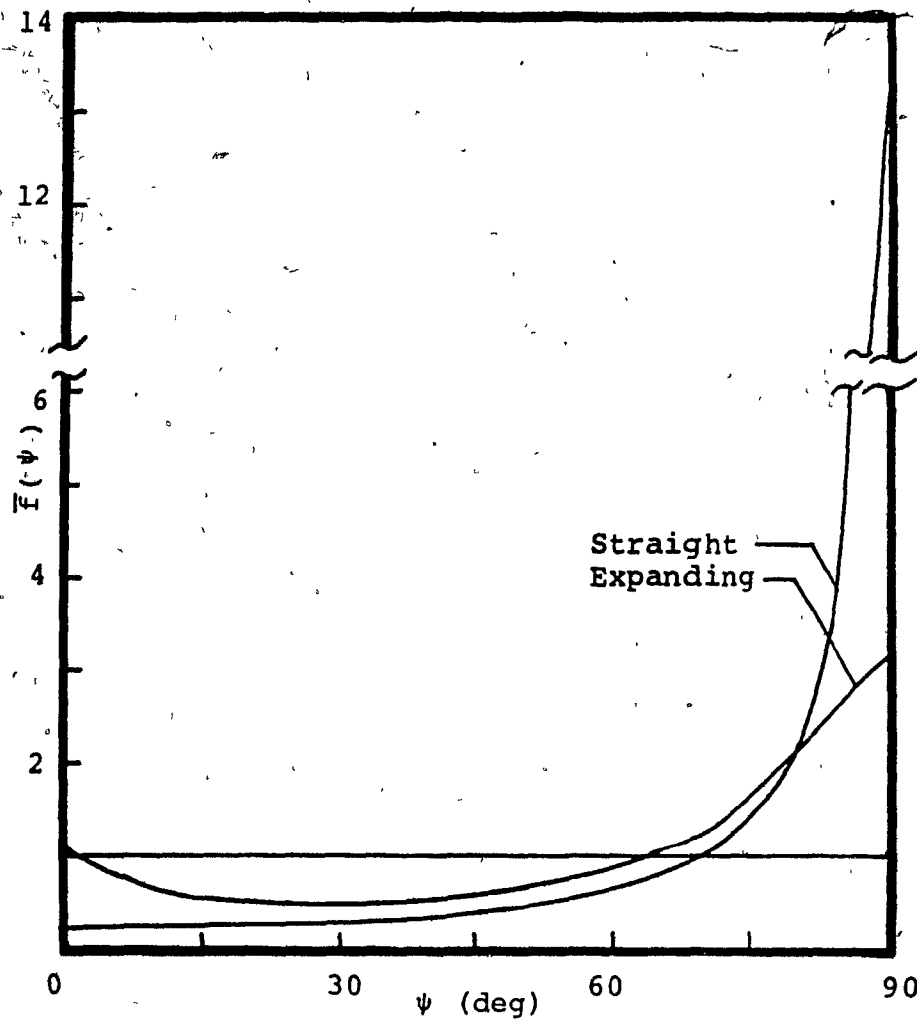


Figure 3.9c Comparison of Fiber Orientation Distributions
Produced by Expanding and Straight Dies.
 $y/H = 0.5$ (Midway)

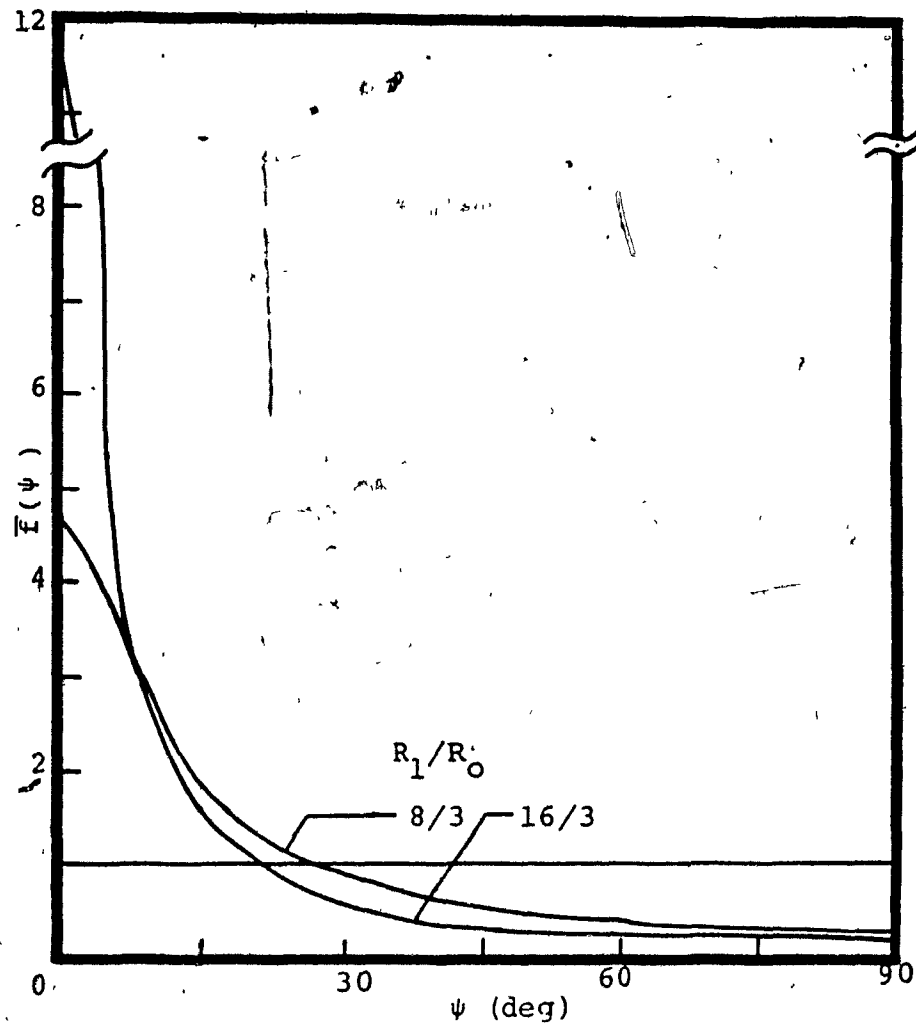


Figure 3.10a Effect of the Change in the Degree of Expansion (R_1/R_0) on Fiber Orientation Distribution
 $y/H = 0$ (Center Line)
 $(R_0/H = 12, \alpha = 60^\circ, n = 0.5)$

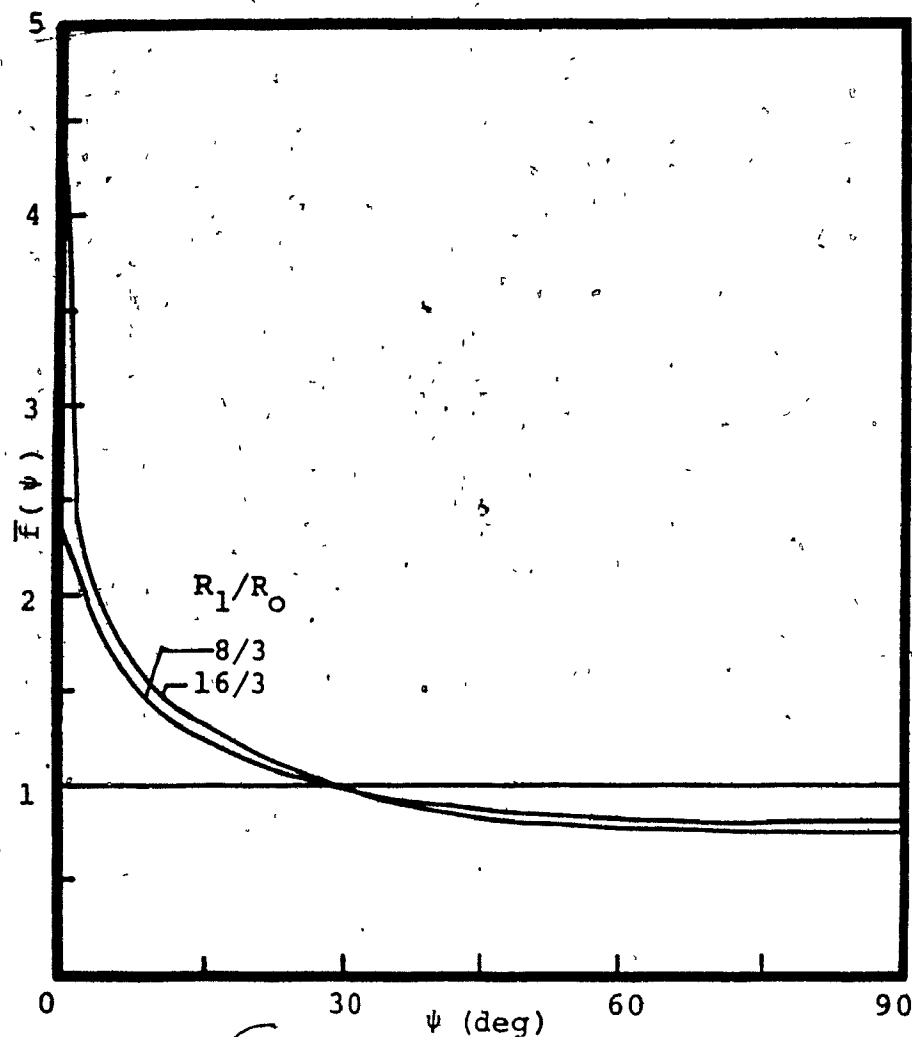


Figure 3.10b Effect of the Change in the Degree of Expansion
 (R_1/R_0) on Fiber Orientation Distribution.
 $y/H = 0.25$ (Near-Center)
 $(R_0/H = 12, \alpha = 60^\circ, n = 0.5)$

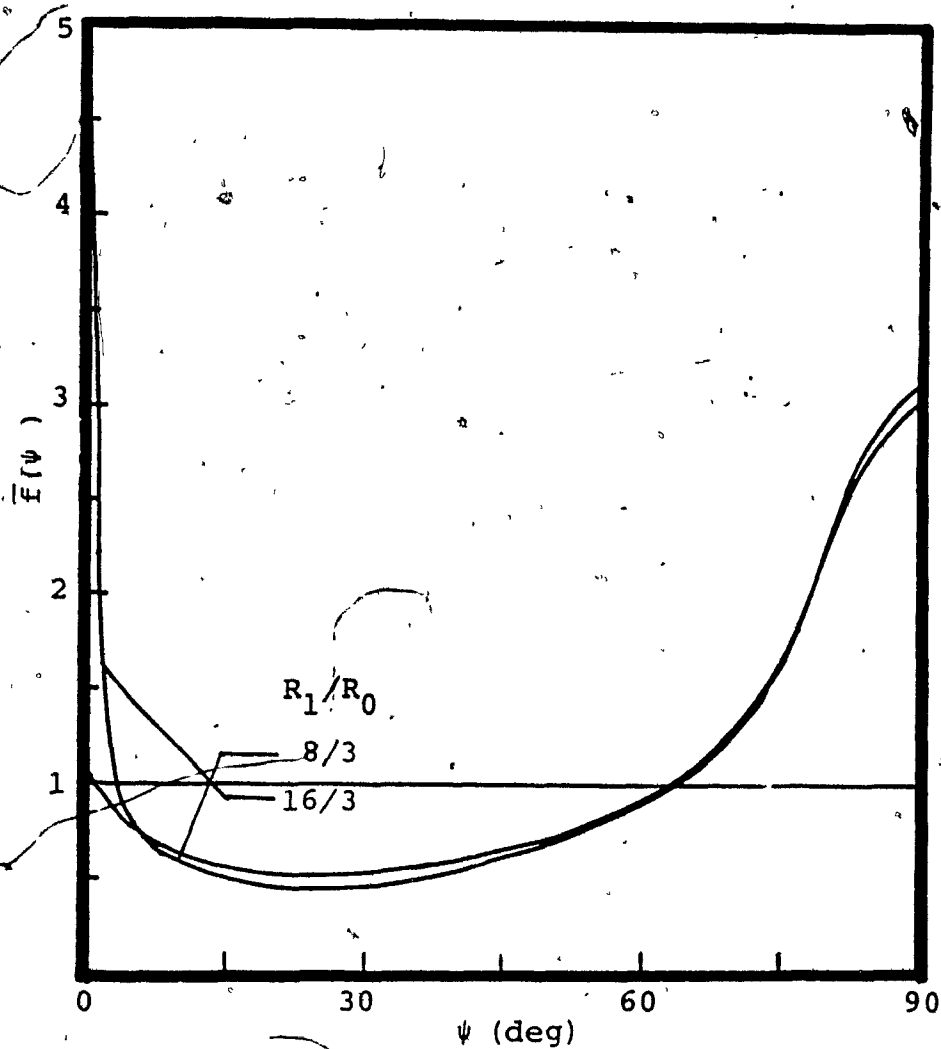


Figure 3.10c Effect of the Change in the Degree of Expansion (R_1/R_0) on Fiber Orientation Distribution.
 $y/H = 0.5$ (Midway)
 $(R_0/H = 12, \alpha = 60^\circ, \eta = 0.5)$

significantly higher overall degree of hoop orientation.

In Figures 3.6 to 3.10, results are presented in terms of the orientation distribution function, $\bar{f}(\psi)$. In order to represent the effects of the various geometrical parameters on the overall degree of orientation at the die exit in a more concise way, it is convenient to define an average angle of orientation with respect to the hoop direction, $\bar{\psi}$, as follows:

$$\bar{\psi} = \frac{\int \psi \bar{f}(\psi) d\psi}{\int \bar{f}(\psi) d\psi} \quad (3.28)$$

The average angle of orientation, $\bar{\psi}$, at the die exit as a function of the three die geometry parameters, the relative channel thickness (R_0/H), the angle of expansion (α) and the overall degree of expansion (R_1/R_0), is plotted in Figure 3.11 at the three locations across the die channel considered above. It may be noted that the minimum value of $\bar{\psi}$ (maximum degree of hoop orientation) predicted at the center line is about 23° . As discussed above, the overall degree of hoop orientation at any location increases with the relative channel thickness, the angle of expansion and the overall degree of expansion.

3.4 Experimental Study

In order to carry out the extrusion experiments, a special expanding channel die was constructed. The die is shown schematically in Figure 3.12a. It essentially consists of three

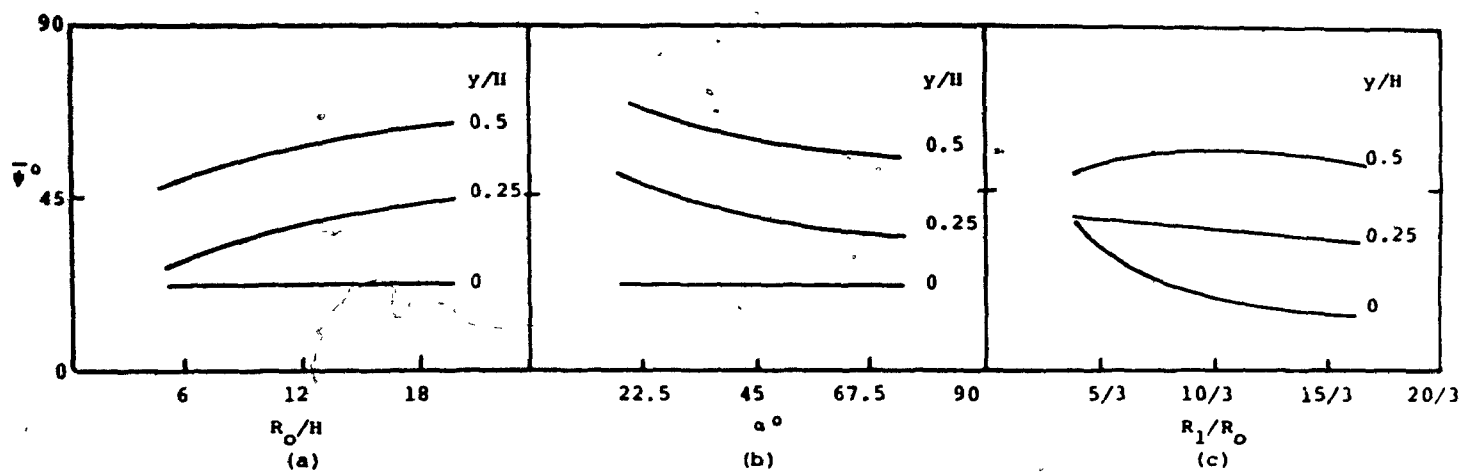


Figure 3.11 ϕ as a Function of Die Geometrical Parameters
 (a) Relative Channel Thickness (R_0/H)
 (b) Angle of Expansion (α)
 (c) Degree of Expansion (R_1/R_0)

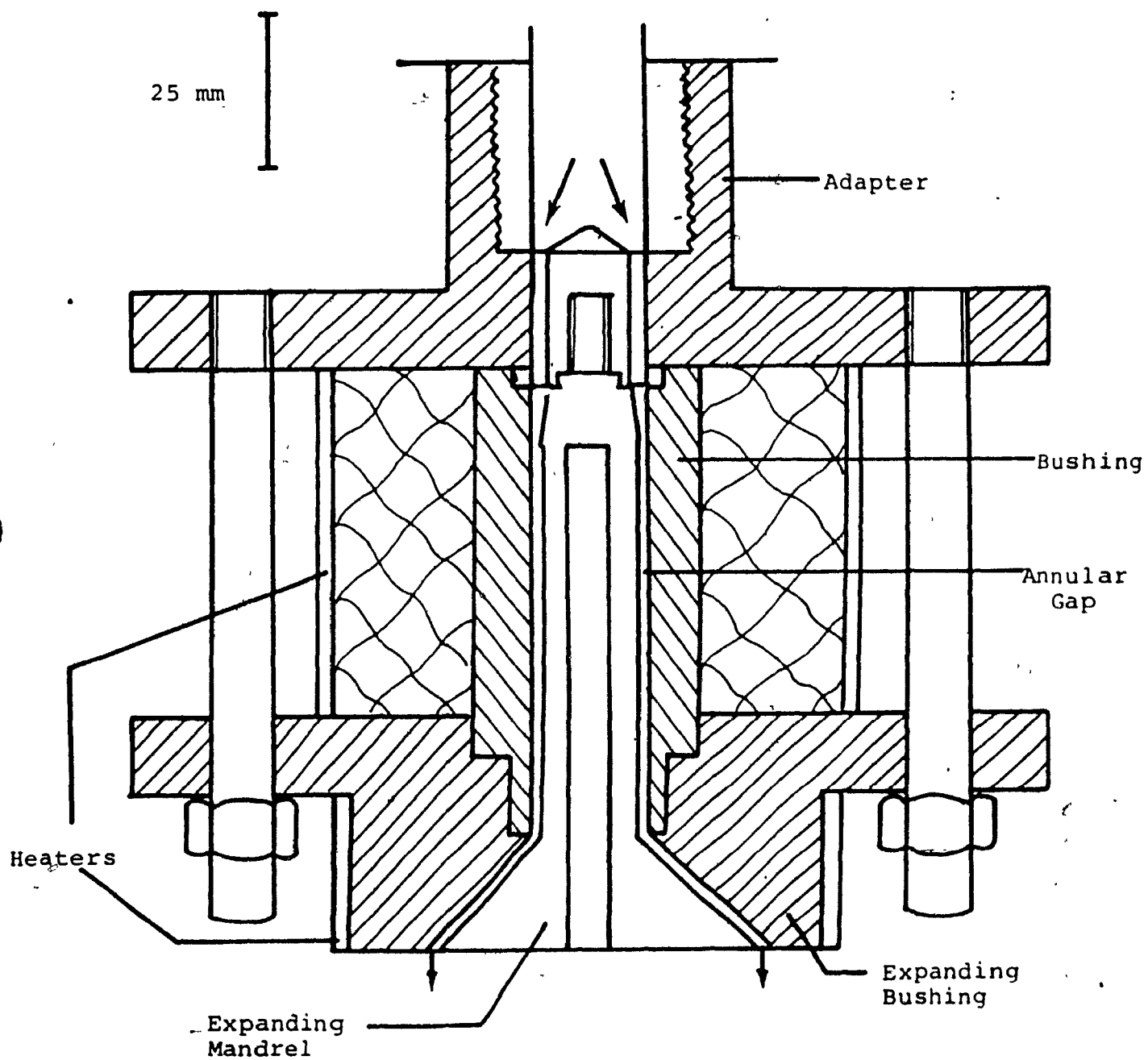


Figure 3.12a Expanding Channel Die for Simple Extrusion (Schematic).

parts: the adapter, the central die body and the expanding section. The adapter is used to attach the die to the end of the barrel of an Instron capillary rheometer. The barrel thus acts as a reservoir for the molten polymer. The adapter features ten symmetrically placed holes for the entry of the polymer melt from the barrel into the annular die gap, as shown schematically in Figure 3.12b. A streamlining cone in the center facilitates the flow of melt into the holes and helps avoid its stagnation. The central die body provides a straight annular passage formed between the straight portion of the mandrel and the outer bushing. It channels the melt into the expanding section of the die formed between the expanding portion of the mandrel and an outer expanding bushing. All die parts are held together by four symmetrically placed studs on the outer periphery of the flanges provided on the adapter and the expanding bushing. Both the expanding mandrel and the expanding bushing of the die can be replaced by a straight mandrel and a flange shown in Figure 3.12c to provide a non-expanding straight annular flow geometry. To maintain the concentricity of the mandrel with respect to the outer bushing during extrusion, it was found necessary to provide three centering pins on each mandrel. The pins were located about 10 mm upstream of the expanding portion of the expanding mandrel and about halfway from the exit on the straight mandrel. The dimensions of the annular geometry of the die are given in Figure 3.12 a.

The die was provided with three heaters, each with its own

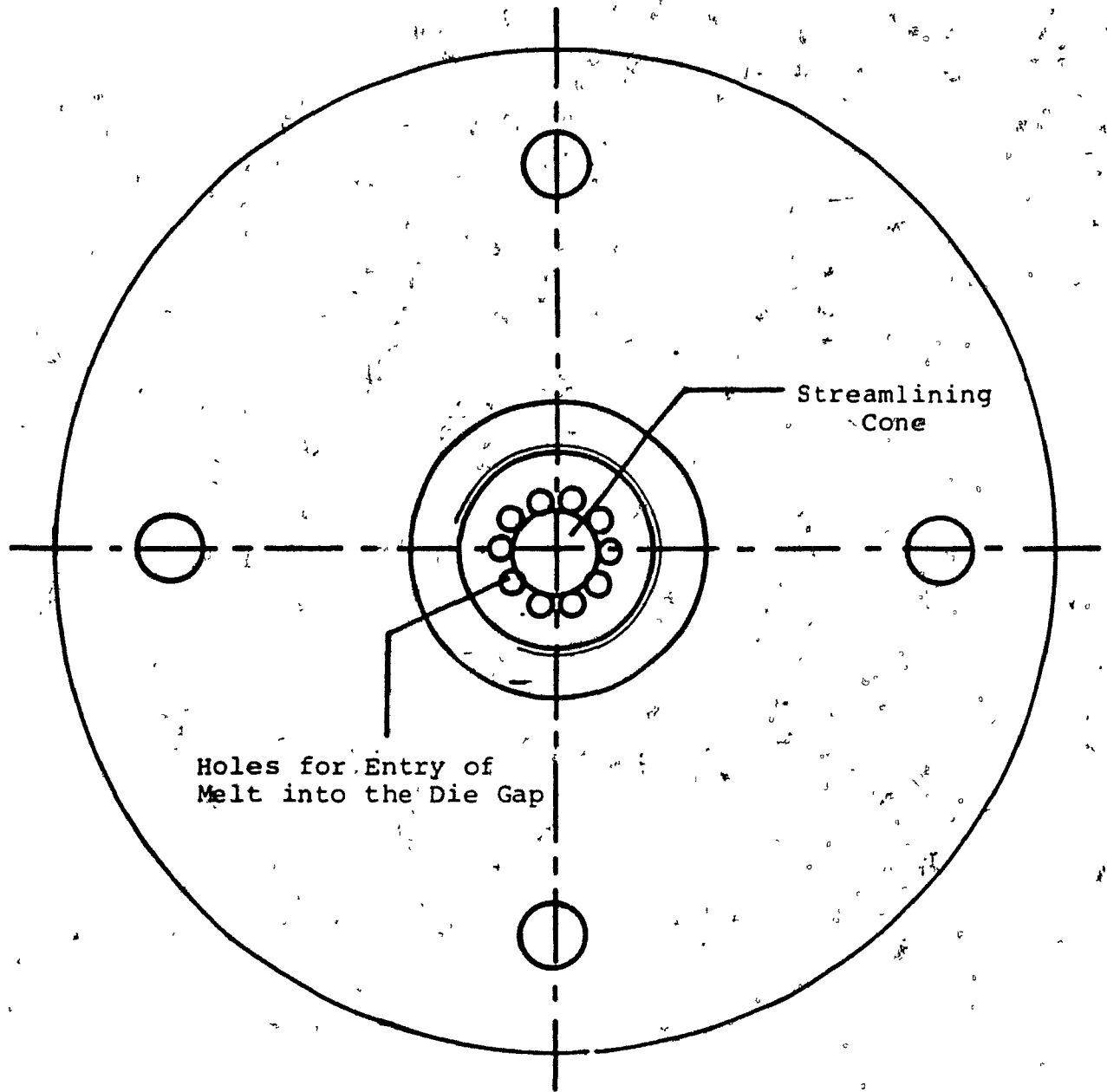


Figure 3.12b Plan View of the Adapter

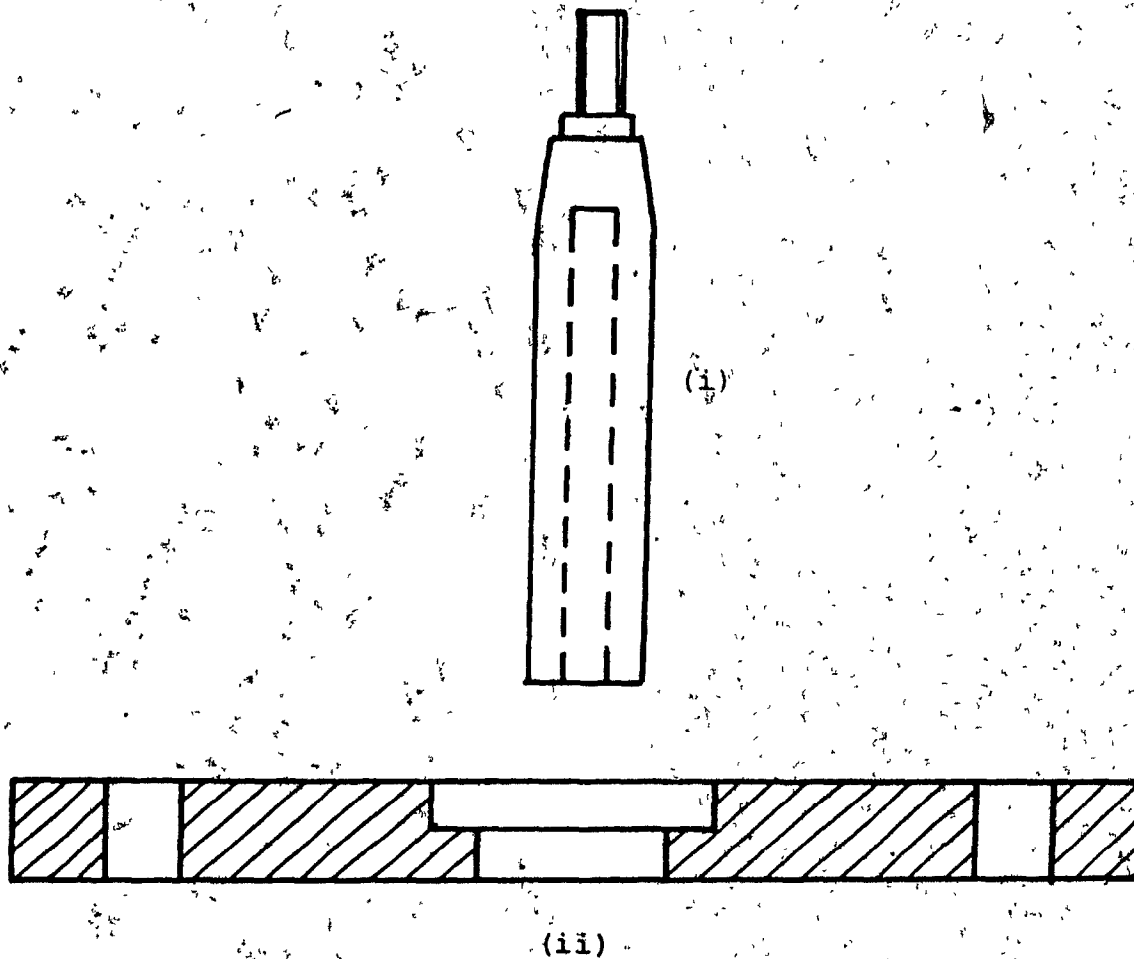


Figure 3.12c (i) Straight Mandrel and (ii) Flange for the Straight Annular Die Configuration.

temperature controller. The central die body was heated by a 600 W band heater, and the expanding section was heated by a 300 W band heater. The mandrel was provided with a 65 W cartridge heater for preheating. This heater was disconnected during extrusion. The die was well insulated to minimize heat losses.

For extrusion the die was attached to the barrel of the capillary rheometer and the assembly was heated to the extrusion temperature of 200° C. The barrel was filled with the SFRTTP resin, pouring a small amount at a time and compacting so as to avoid the trapping of air bubbles in the melt. The material was allowed to melt and attain a reasonably uniform temperature. Extrusion was carried out by a piston attached to the crosshead of the Instron rheometer and lowered at a specified speed into the barrel. An initial portion of the extrudate was discarded until the force to drive the piston into the barrel was steady. After a sufficient length of the extrudate was obtained, the extrusion was stopped and it was allowed to freeze in air before cutting. Experiments were carried out at various extrusion rates in the range from 0.0475 cm³/s to 2.375 cm³/s using both the expanding and the straight channel dies. The collected extrudates were subsequently subjected to mechanical testing and optical microscopy to assess fiber orientation.

3.4.1 Mechanical Testing

Flexural stiffness and tensile strength in the axial and hoop directions of the extrudates were measured in order to evaluate the fiber orientation patterns. Two important factors had to be considered in preparing the test pieces. First, due to the cylindrical geometry of the extrudate, test pieces suitable for standard testing procedures could not be obtained directly. Secondly, weld lines were present along the extrudate due to splitting and recombination of the melt at the holes provided in the adapter. While some healing of the weld lines did occur during the subsequent flow along the die, their complete elimination was not possible, particularly at the higher extrusion rates. The centering pins present on the mandrel also gave rise to three additional weld lines located very close to the three of the weld lines resulting from the holes in the adapter. It is likely that the fiber orientation pattern in the vicinity of a weld line may be different from that in the region away from a weld line.

To alleviate the first problem, strips were cut from extruded tubes in the axial and hoop directions and then flattened between parallel platens to obtain usable samples. The flattening procedure consisted of heating two highly polished steel plates along with a spacer of the appropriate thickness to a temperature of $105 \pm 5^\circ \text{C}$ on a laboratory press. Strips cut from the extrudates were placed between the heated plates, as

illustrated in Figure 3.13. The portion of the strip in contact with the plate was thus heated to a rubbery state, and was slowly flattened under the moderate force exerted by the weight of the top plate. In this way, the entire length of the strips could be flattened in about 25 to 30 minutes. The presence of the spacer prevented compression of the strips. Due to the moderate temperature and force applied during flattening, the amount of secondary flow occurring during the process was rather insignificant, and the fiber orientation in the strips was preserved. The flattened strips were cooled to room temperature by water circulation in the platens of the press.

The weld lines mentioned above were also present in the flattened strips and the test procedure had to take this into account. For flexural stiffness determination by the three point bending method, test pieces of dimensions 75x12.5 mm (3x0.5 in) were cut from the flattened strips, as shown in Figure 3.14a. In the case of the expanding die extrudates, the test pieces in the axial direction could be cut from a region away from weld lines, but weld lines could not be avoided in the case of test pieces in hoop direction. However, care was taken so that the point of maximum deflection in the hoop test pieces never corresponded to a weld line. The extrudates obtained from the straight annular die had a nominal diameter of only 19 mm (0.75 in). Hence, hoop test pieces were slightly shorter than 60 mm in length and the weld lines were very close in both the axial and hoop direction test pieces.

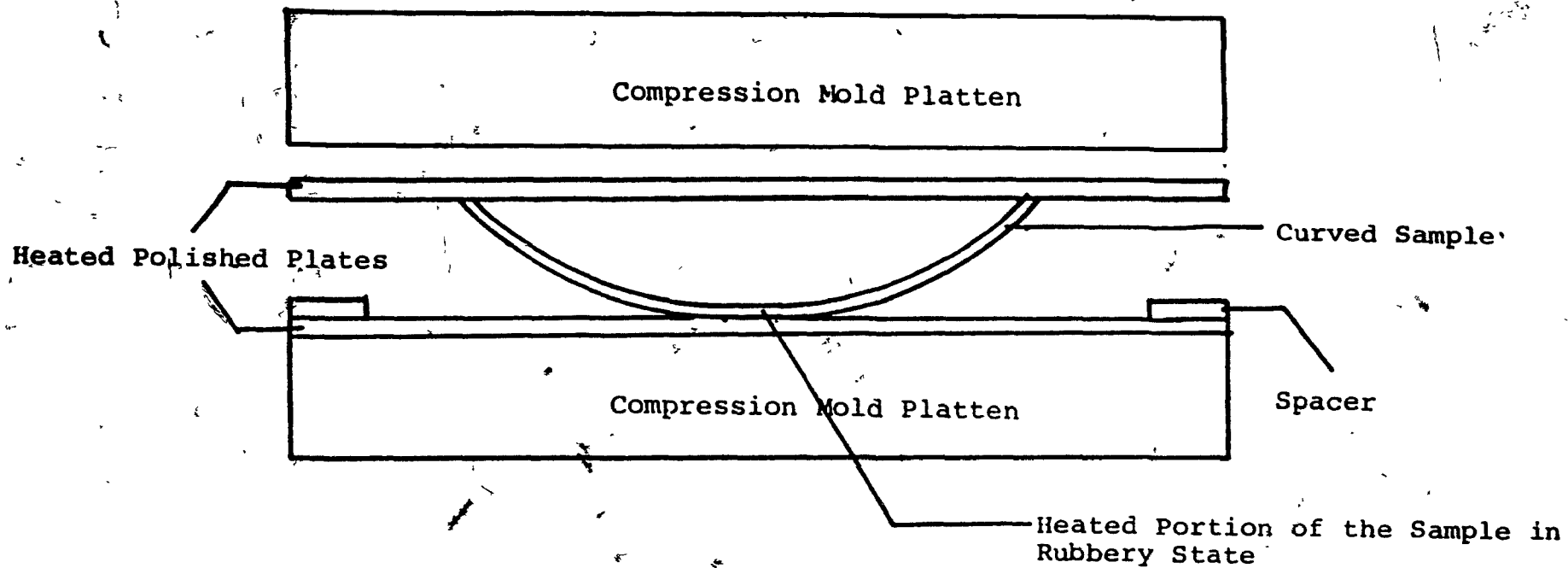


Figure 3.13 Flattening of Curved Sample from Extrudate.

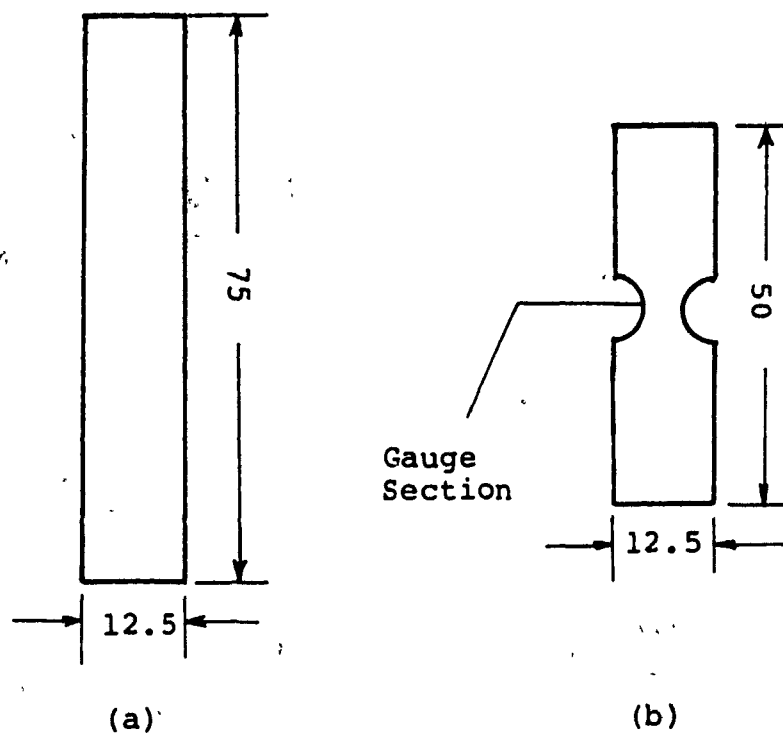


Figure 3.14 Test Piece Geometry
(a) Flexural (b) Tensile
(Dimensions in mm)

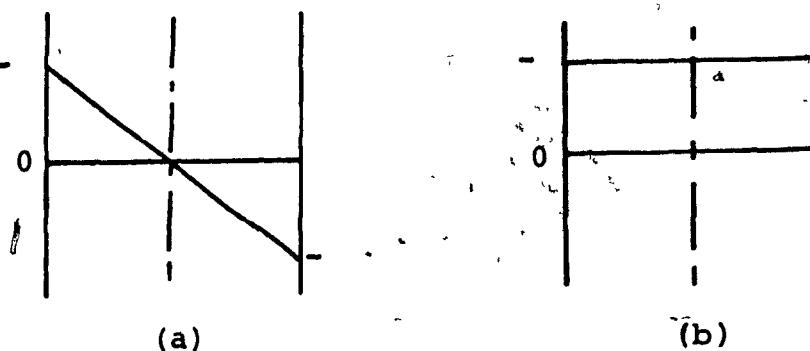


Figure 3.15 Strain Distribution across Test
Piece Thickness
(a) Flexural Testing
(b) Tensile Testing

The flexural stiffness was determined by the three point bending method with a span of 50 mm (2 in) using the Instron Universal Tester at a crosshead speed of 0.5 mm/min. The initial slope of the load deflection test record was used to calculate the flexural stiffness E_f as follows.

$$E_f = \frac{1}{4} \left(\frac{F}{y} \right) \frac{S^3}{B D^3} \quad (3.29)$$

where (F/y) is the initial slope of the load-deflection record, S is the support span (50 mm), and B and D are the width and thickness of the test piece, respectively.

For tensile strength measurements, test pieces with a length of 50 mm (2 in) were used. Again, the test pieces in the axial direction of the expanding channel extrudates could be obtained while avoiding the weld lines. In order to avoid weld lines in the gauge section of the hoop test pieces cut from the expanding die extrudates, a special test piece geometry with a shorter gauge section was used as is illustrated in Figure 3.14b. Using such a geometry, the failure always occurred in the region away from the weld lines. Again, it was not possible to avoid the weld lines in the test pieces derived from the straight annular die extrudates. The same geometry as shown in Figure 3.14 b was used for both the axial and hoop direction test pieces.

The tensile strength was also determined on the Instron Universal Tester at the same crosshead speed of 0.5 mm/min. The

tensile strength, σ_y , was calculated as follows:

$$\sigma_y = \frac{F_m}{B D} \quad (3.30)$$

where F_m is the maximum force corresponding to the test piece failure
and B and D are the width and thickness of the test piece in the gauge section, respectively.

3.4.2 Results and Discussion

Average flexural stiffness and tensile strength data are presented in Tables 3.1 and 3.2, respectively. The individual values are tabulated in Appendix 3.3 and 3.4 respectively. Four or more test pieces were used for each case and scatter about the mean value was less than 10% for flexural stiffness and 6% for tensile strength. The results show little systematic effect of the rate of extrusion on the measured properties, and hence the fiber orientation distribution, which is in accord with the model prediction.

The flexural moduli are higher in the axial direction for both the expanding and the straight channel die geometries. However, the anisotropy ratio (defined as the ratio of the axial to hoop direction properties) for the expanding geometry is much lower than that for the straight geometry (1.2 versus 3). A comparison of the results clearly shows that while a high degree of axial orientation is developed in the straight channel die,

Table 3.1
Flexural Stiffness

Rate of Extrusion (cm ³ /s)	Flexural Stiffness	
	MPa	
	Axial	Hoop
(a) Expanding Die		
0.0475	2984 $\pm$ 219	2336 $\pm$ 205
0.2375	2721 $\pm$ 364	2345 $\pm$ 185
0.475	2817 $\pm$ 155	2469 $\pm$ 209
0.95	2801 $\pm$ 190	2314 $\pm$ 145
2.375	2580 $\pm$ 215	2476 $\pm$ 92
Average	2800	2400

Average Anisotropy Ratio (A/H) = 1.2

(b) Straight Die		
0.0475	4276 $\pm$ 104	1360 $\pm$ 81
0.2375	4562 $\pm$ 320	1522 $\pm$ 86
0.475	4104 $\pm$ 226	1408 $\pm$ 79
0.95	4250 $\pm$ 298	1543 $\pm$ 68
2.375	3819 $\pm$ 179	1496 $\pm$ 91
Average	4200	1400

Average Anisotropy Ratio (A/H) = 3.0

(c) Core		
Ground test pieces	1800 $\pm$ 346	2700 $\pm$ 440

Average Anisotropy Ratio (A/H) = 0.7

Table 3.2

Tensile Strength

Rate of Extrusion (cm ³ /s)	Tensile Strength	
	MPa	
	Axial	Hoop
(a) Expanding Die		
0.0475	25.0 $\pm$ 1.3	30.5 $\pm$ 1.8
0.2375	24.2 $\pm$ 2.4	28.4 $\pm$ 1.4
0.475	24.2 $\pm$ 0.8	30.5 $\pm$ 3.8
0.95	23.2 $\pm$ 0.8	28.6 $\pm$ 1.2
2.375	21.9 $\pm$ 0.8	27.5 $\pm$ 1.9
Average	24	29

Average Anisotropy Ratio (A/H) = 0.8^B

(b) Straight Die		
0.0475	38.6 $\pm$ 2.4	17.7 $\pm$ 0.5
0.2375	34.4 $\pm$ 1.9	18.3 $\pm$ 0.6
0.475	33.6 $\pm$ 3.3	17.5 $\pm$ 0.6
0.95	35.8 $\pm$ 2.4	18.6 $\pm$ 0.4
2.375	34.8 $\pm$ 1.8	17.0 $\pm$ 1.5
Average	35	18

Average Anisotropy Ratio (A/H) = 2

(c) Core

Ground test pieces	23 $\pm$ 2.6	31 $\pm$ 3.5

Average Anisotropy Ratio (A/H) = 0.75

the expanding channel die succeeds in producing a fairly balanced orientation of fibers between the axial and hoop directions.

It should be noted that in a flexural test, a nonuniform strain is imposed across the test piece thickness. The strain is zero at the neutral axis and varies linearly toward the surface, as illustrated in Figure 3.15a. As a result, the near surface region dominates the mechanical response in a flexural test. The overall flexural stiffness, thus, reflects the fiber orientation near the surface more than that in the core (31,66). It may be noted that the theoretical analysis predicted a strong variation in the orientation pattern across the extrudate thickness. In order to assess this experimentally, several test pieces were carefully ground to remove surface layers on both sides reducing the overall thickness by about 30%. These ground test pieces were tested in flexure and were expected to better reflect the "core" structure. Test results for the ground test pieces are presented in Tables 3.1 c and 3.2 c. It is seen from these tables that the average hoop stiffness is higher than the average axial stiffness indicating a higher degree of hoop orientation of fibers in the core.

Tensile strength measurements impose a uniform strain across the test piece cross-section and hence reflect more balanced contributions from the surface and core regions (Figure 3.15b). Results presented in Table 3.2 indicate that the axial tensile strength is lower than the hoop tensile strength for the

expanding channel die extrudates (average anisotropy ratio being less than 1.0), while the inverse is true for the straight channel die extrudates (average anisotropy ratio being more than 1.0).

3.4.3 Optical and Scanning Electron Microscopy

Optical techniques were used to obtain a direct evidence of the fiber orientation and its variation across the thickness of the extrudates produced using the expanding and straight channel dies. Specifically, low magnification transmission light microscopy (TLM) of thin microtomed slices and scanning electron microscopy (SEM) of the fracture surface of tensile test pieces were employed for this purpose.

Thin slices of 20 to 30 μm were microtomed from regions near the surface, near the center, and at some intermediate location. Light passing through such a slice undergoes significant scattering due to the fibers. To avoid this, the microtomed slices were placed on glass plates and immersed in benzene, which has nearly the same refractive index as glass. The slices were covered with thin glass plates and photographed through a transmission light microscope. Figure 3.16 shows the change in the fiber orientation pattern from the surface to the core of the extrudate produced using the expanding channel die. These pictures show a predominantly axial orientation near the surface changing to a strong hoop orientation near the center.



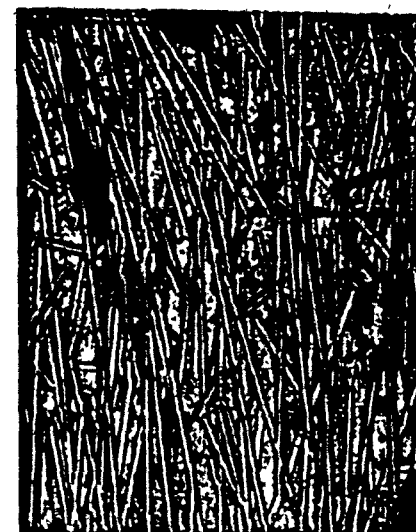
(a)

Near Outer Surface



(b)

Intermediate



(c)

Core

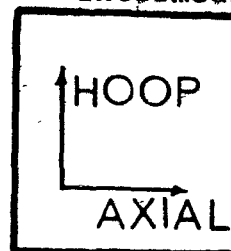
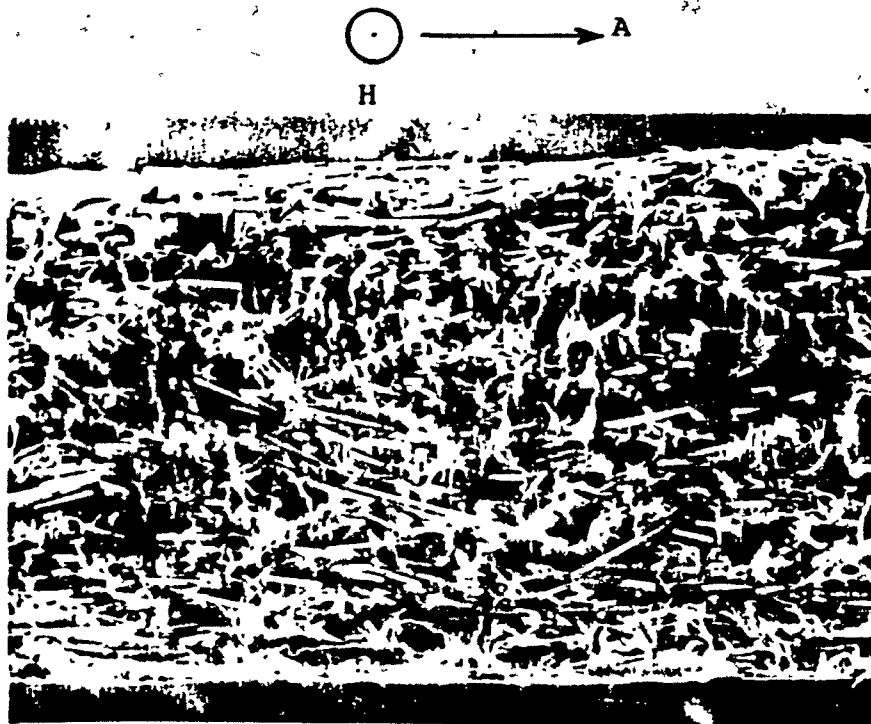


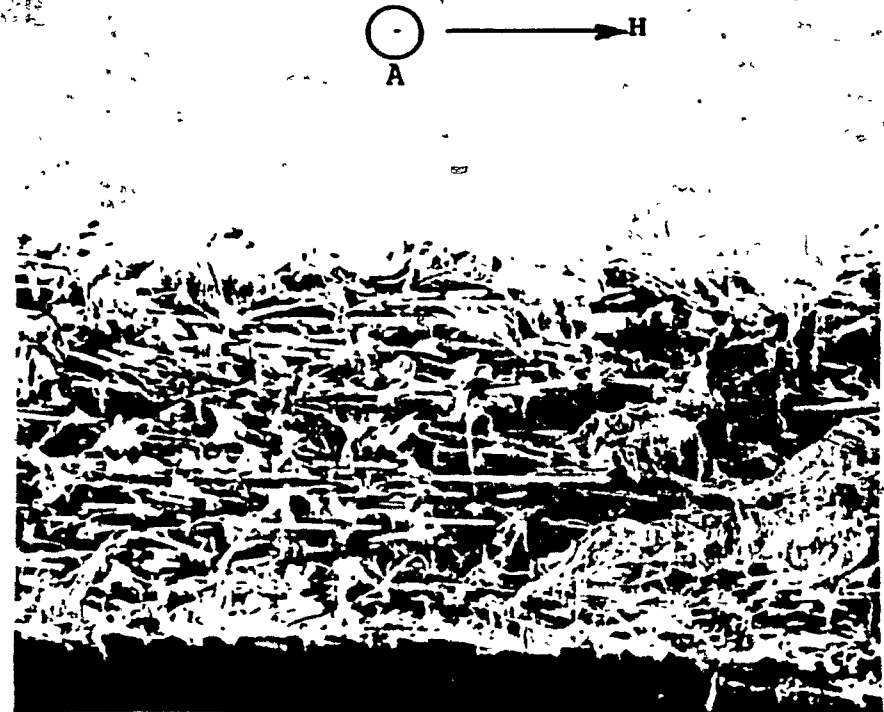
Figure 3.16 Optical Micrographs Showing Change in the Fiber Orientation Pattern across the wall of the Expanding Channel Die Extrudates. (75x)

Examination of the fracture surfaces of the tensile test pieces by scanning electron microscopy further demonstrates this point. Figure 3.17a is a scanning electron micrograph of the fracture surface of a hoop test piece obtained from an expanding channel extrudate. (The hoop direction is normal to the plane of the picture and the axial direction is along this plane.) It is seen from this micrograph that in the central region of the test piece the fibers are oriented normal to the plane of the picture and hence in the hoop direction. Near the surfaces, however, the fibers are oriented in the axial direction. Figure 3.17b shows the fracture surface of an axial test piece obtained from an expanding channel extrudate. (The axial direction is normal to the plane of the picture and the hoop direction is along the plane.) A significant degree of hoop orientation of fibers in the core of the test piece is clearly demonstrated in this micrograph.

Figure 3.18 shows the fiber orientation pattern across the thickness of the extrudate obtained with the straight channel die. Here the orientation is predominantly axial near the surface and somewhat random near the center. Figure 3.19a is a scanning electron micrograph showing the fracture surface of a hoop test piece obtained from a straight channel die extrudate. The axial direction is in the plane of the picture, and a large number of fibers are seen to be oriented in that direction. Figure 3.19b shows the fracture surface of an axial test piece. A large number of fibers are oriented perpendicular to the plane of the picture,



(a)

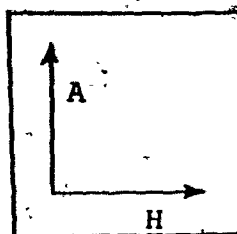


(b)

Figure 3.17 Scanning Electron Micrographs of the Fracture Surfaces of Tensile Test Pieces Derived from the Expanding Channel Die Extrudates (51 x).
(a) Hoop Test Piece (b) Axial Test Piece



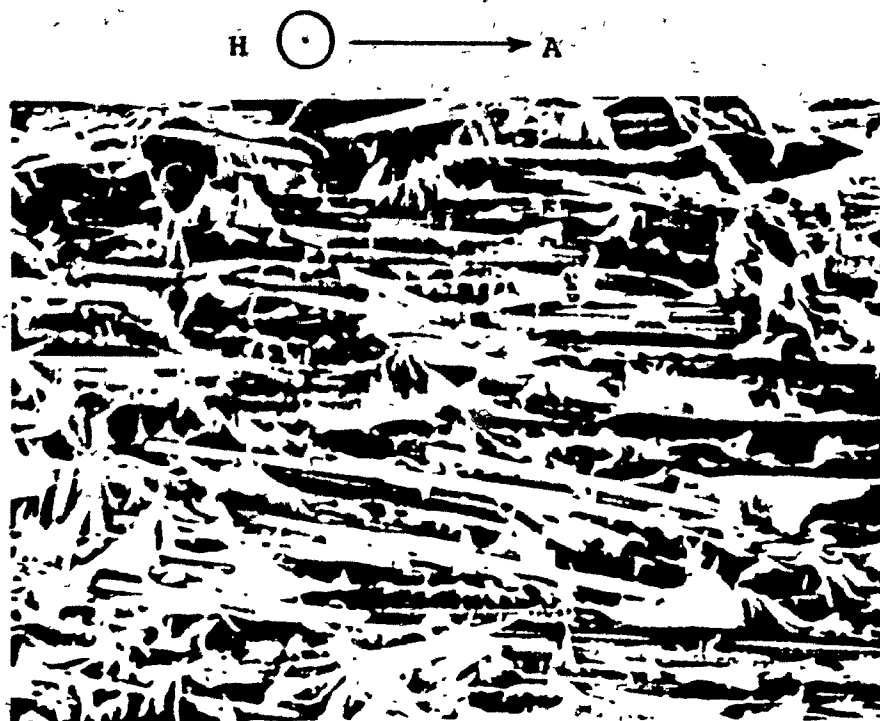
(a)



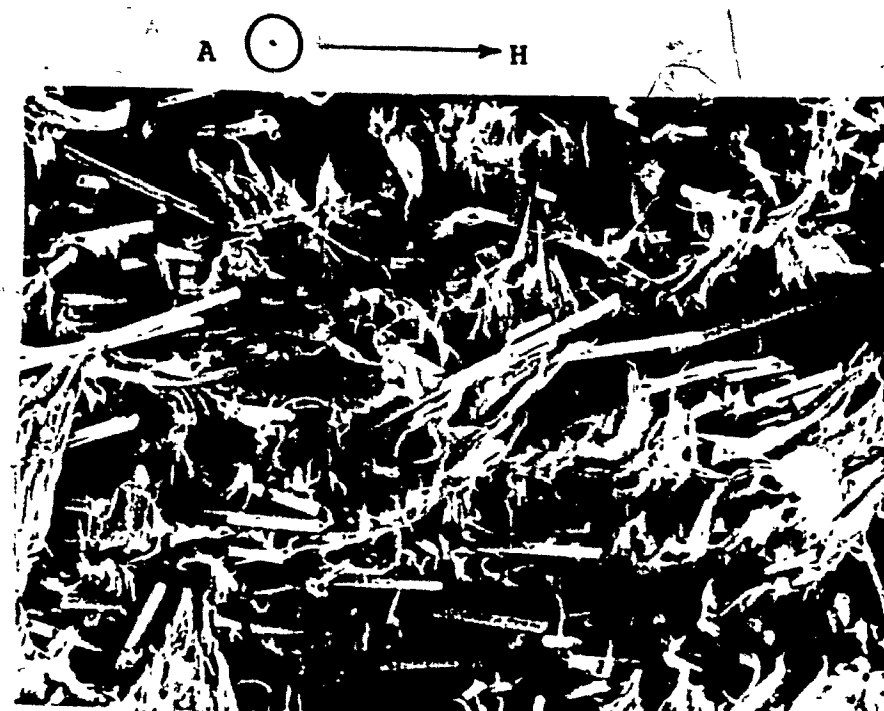
(b)

Figure 3.18 Optical Micrographs Showing Fiber Orientation in the Straight Annular Die Extrudates (100x).

(a) Surface (b) Center



(a)



(b)

Figure 3.19 Scanning Electron Micrographs of the Fracture Surfaces of Tensile Test Pieces Derived from the Straight Annular Die Extrudates (105x).
 (a) Hoop Test Piece (b) Axial Test Piece

i.e., close to the axial direction.

The results obtained from both the mechanical testing and microscopic methods thus demonstrate that while the shear flow in the straight channel die produces a predominantly axial orientation of fibers, the extensional flow generated in the expanding channel die promotes the hoop orientation of fibers, particularly in the central region away from the walls. These results are all in qualitative agreement with the results of the theoretical analysis presented in Section 3.3.4.

While the experimental results were not intended for quantitative comparison with the theoretical predictions, both of these indicate that even in the expanding channel die, the higher shear rate occurring near the die walls does not permit hoop orientation of fibers to be promoted near the surface of the extrudate. It is hence desirable to find ways of reducing the effect of shear near the wall.

This consideration led to the design of a coextrusion die to produce a three-layer pipe while overcoming the effect of shear near the wall as discussed in Chapter 4.

Chapter 4

COEXTRUSION STUDIES

As mentioned in Chapter 1, the use of reinforcing fibers in an extrusion product such as pipe has the disadvantage of causing a deterioration of the surface quality as a result of protruding fibers. The process of coextrusion to produce a three-layer pipe was suggested as a practical solution to this problem in Chapter 1. In the coextruded pipe (Figure 1.2), the inner and outer surface layers are of unreinforced polymer to provide a smooth surface finish, and the middle layer is of SF RTP having a preferential orientation of fibers in the hoop direction. As discussed in Chapter 3, an expanding channel die can be used to promote hoop orientation of fibers in the SF RTP layer during extrusion. The flow through such a die involves a superposition of shear and extensional components of deformation. While the extensional component tends to orient the fibers in the hoop direction, the shear component tends to orient them in the axial direction. Also, the effect of shear is higher near the walls, while that of extension is higher in the core region.

If a uniform preferential hoop fiber orientation is desired over most of the thickness of the SF RTP layer, shearing in this layer during flow through an expanding channel of a coextrusion die must be minimized. This can be achieved by forming a three-layer melt system consisting of two unreinforced surface layers

and a SF RTP middle layer, upstream of the expanding channel. During the flow through the expanding channel, the unreinforced surface layers keep the SF RTP layer away from the regions of high shear rate near the wall (Figure 4.1). In addition, if they are of a resin having a relatively low melt viscosity and suitable shear-thinning characteristics, they will "lubricate" the flow of the middle SF RTP layer through the die, and thus minimize its shearing, and promote the hoop orientation of fibers over most of its thickness.

In order to evaluate the feasibility of this concept to produce a fairly uniform preferential hoop orientation of fibers throughout the thickness of the SF RTP layer, a theoretical analysis of the coextrusion process was carried out. This analysis is based on the theoretical model developed in Chapter 3, with the addition of coextrusion, and it provides a basis for the design of the coextrusion die. Coextrusion experiments were carried out using a die constructed in accordance with the results of this analysis.

4.1 Theoretical Analysis of the Flow-Induced Fiber Orientation During Coextrusion through an Expanding Channel Die.

The analysis of the coextrusion process involves an extension of the model developed in Section 3.3 for predicting the flow-induced fiber orientation distribution resulting from simple extrusion through an expanding channel pipe die. Some

details of the previous analysis are repeated here for the sake of continuity and completeness.

The essential steps of the analysis are the same as before, i.e., the derivation of the flow field, the solution of Jeffrey's equations for the derived flow field to describe the motion of an individual fiber along the flow channel, and the determination of the final fiber orientation distribution at the die exit for a given initial orientation distribution at the entrance. However, as will be shown below, the derivation of the flow field is much more involved for the coextrusion case than for the simple extrusion case. This, in turn, makes the solution of Jeffrey's equations somewhat more complex.

The assumptions made previously are also used for the present analysis. The fiber is modelled as an elongated spheroid (prolate spheroid of revolution) with a high aspect ratio, and fiber-fiber and fiber-wall interactions are neglected. Also, in the vicinity of a fiber the SF RTP melt is assumed to follow Newtonian behavior, while on a global scale it is assumed to follow power-law behavior with a consistency K_1 and an index n_1 . The unreinforced melt for the surface layers is also assumed to follow power-law behavior with a consistency K_2 and an index n_2 . Thus the rheological constitutive equations for the SF RTP and the unreinforced melts are as follows.

$$\text{SF RTP : } \underline{\tau} = K_1 \left(\frac{\dot{\gamma}}{2} \right)^{(n_1-1)/2} \underline{\Delta} \quad (4.1)$$

$$\text{Unreinforced : } \underline{\tau} = K_2 (II_{\underline{\Delta}}/2)^{(n_2-1)/2} \underline{\Delta} \quad (4.2)$$

where $\underline{\tau}$ and $\underline{\Delta}$ are the stress and the rate of deformation tensors respectively.

4.1.1 Derivation of the Flow Field

The flow geometry in the expanding section of the die is shown in Figure 4.1. The mean die radius is R_0 at the entrance and R_1 at the exit. The angle of expansion is α , and the die gap, $2H$, is constant. In order to derive the velocity profile in the channel, the lubrication approximation is used by assuming that the flow in the channel is locally equivalent to fully developed flow between two parallel plates. Also, the effect of the curvature of the flow geometry is neglected on the grounds that the channel thickness, $2H$, is much smaller than the die radius, R . Under these assumptions, the flow geometry of interest can be represented as a flat wedge or pie-shaped region between parallel plates as shown in Figure 4.2. It must be noted that the two side edges A and B of this "lay flat" geometry, shown in the figure, are not free boundaries, but represent coincident planes of the original channel. Thus, the velocity, rate of deformation and shear stress must be continuous across these two planes. This concept of "lay-flat" geometry was also used for the simple extrusion analysis discussed in Section 3.3, but here it also provides a convenient basis for establishing a coordinate system for describing the location of interface during coextrusion.

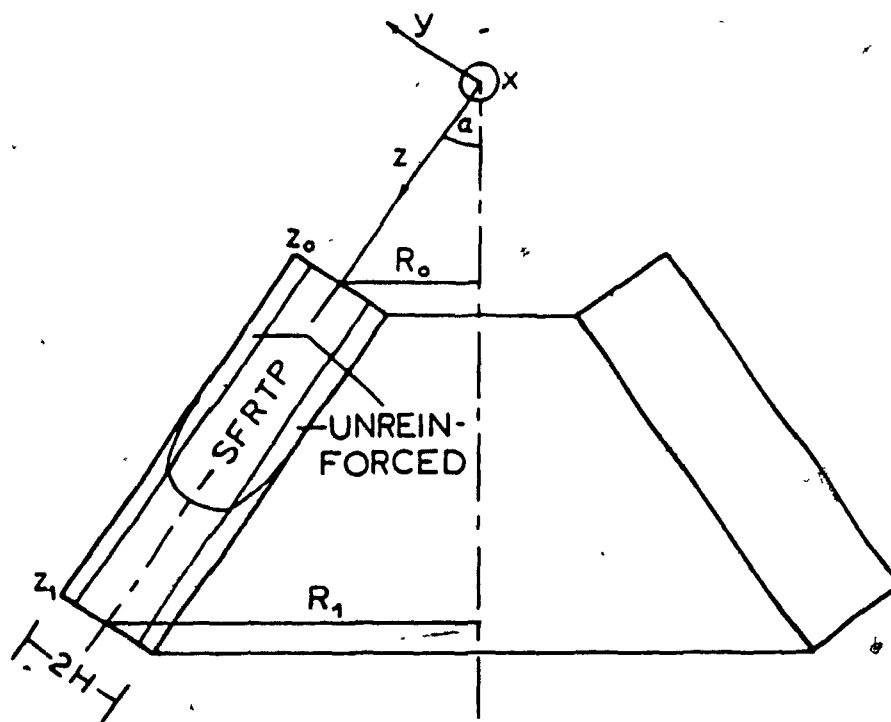


Figure 4.1 Axial Section of the Expanding Channel Coextrusion Die.

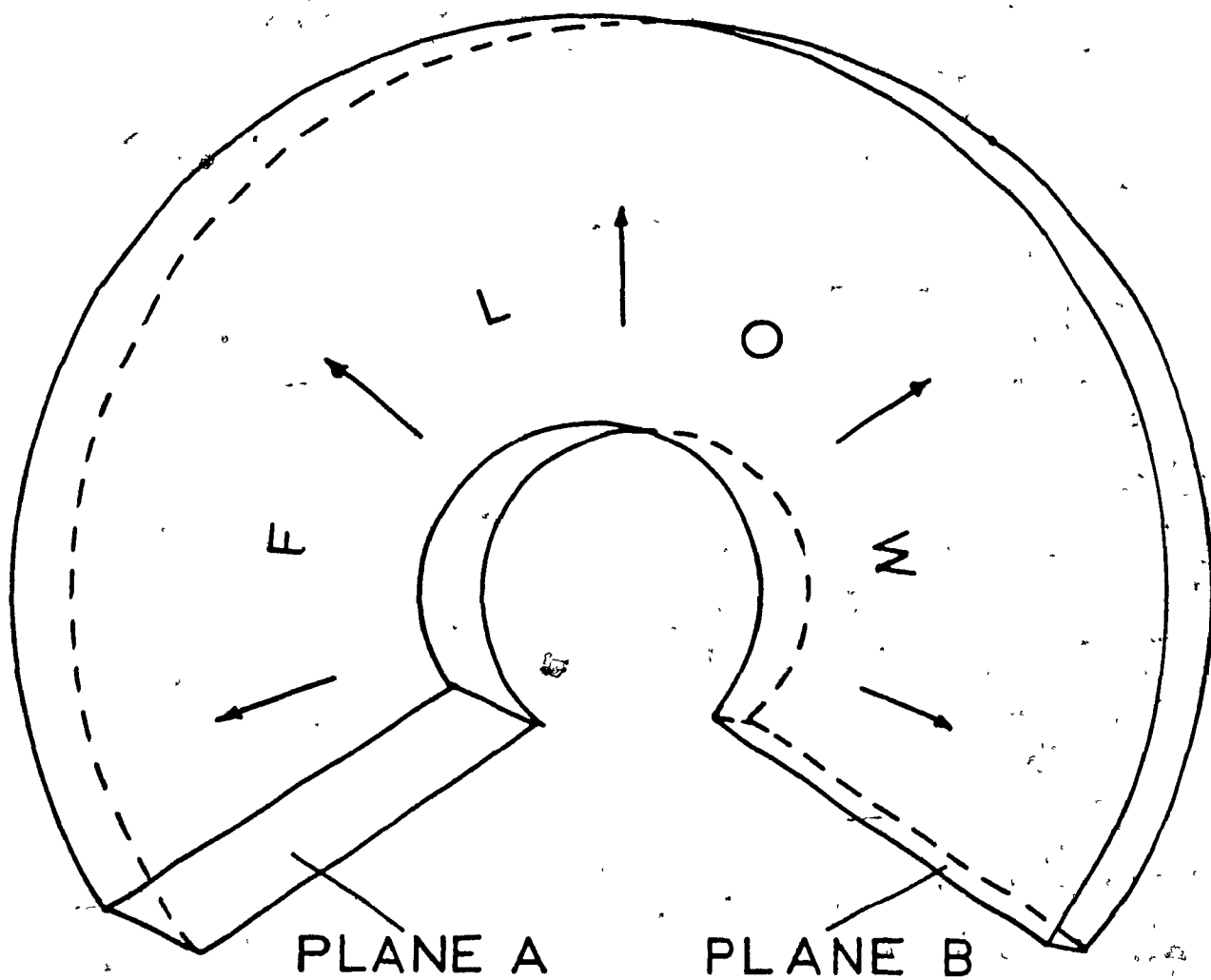


Figure 4.2 "Lay Flat" Geometry for the Expanding Channel Coextrusion Die.

The cylindrical coordinate system (r, θ, n) is shown in Figure 4.3. The r -axis is in the flow direction, the θ -axis represents the hoop direction of the original channel, and the n -axis is normal to the channel wall. As a result of the lubrication approximation, the velocity profile is symmetric about the channel center plane represented by $n=0$. The interface is located at a distance $n=y_1$ from either side of the center plane. The middle layer of SFRTF will be referred to as "layer I", while the inner or outer surface layer of the unreinforced polymer will be referred to as "layer II". The derivation of the velocity profile involves the solution of the dynamic equation in the r direction along with the constitutive relationships given by Equations 4.1 and 4.2. Neglecting the acceleration terms, the dynamic equation in the r direction can be written as follows.

$$\frac{d\tau_{rn}}{dn} - \frac{dp}{dr} = 0 \quad (4.3)$$

where τ_{rn} is the shear stress and $(-dp/dr)$ is the local pressure gradient. The following boundary conditions must be satisfied by the solution:

- i) At $n = 0$, $\tau_{rn} = 0$
- ii) At $n = \pm y_1$, $(\tau_{rn})_I = (\tau_{rn})_{II}$
- iii) At $n = \pm y_1$, $(u_r)_I = (u_r)_{II}$
- iv) At $n = \pm H$, $u_r = 0$

Boundary condition (i) results from the symmetry of the velocity profile about the channel center plane, and (iv) implies no slip

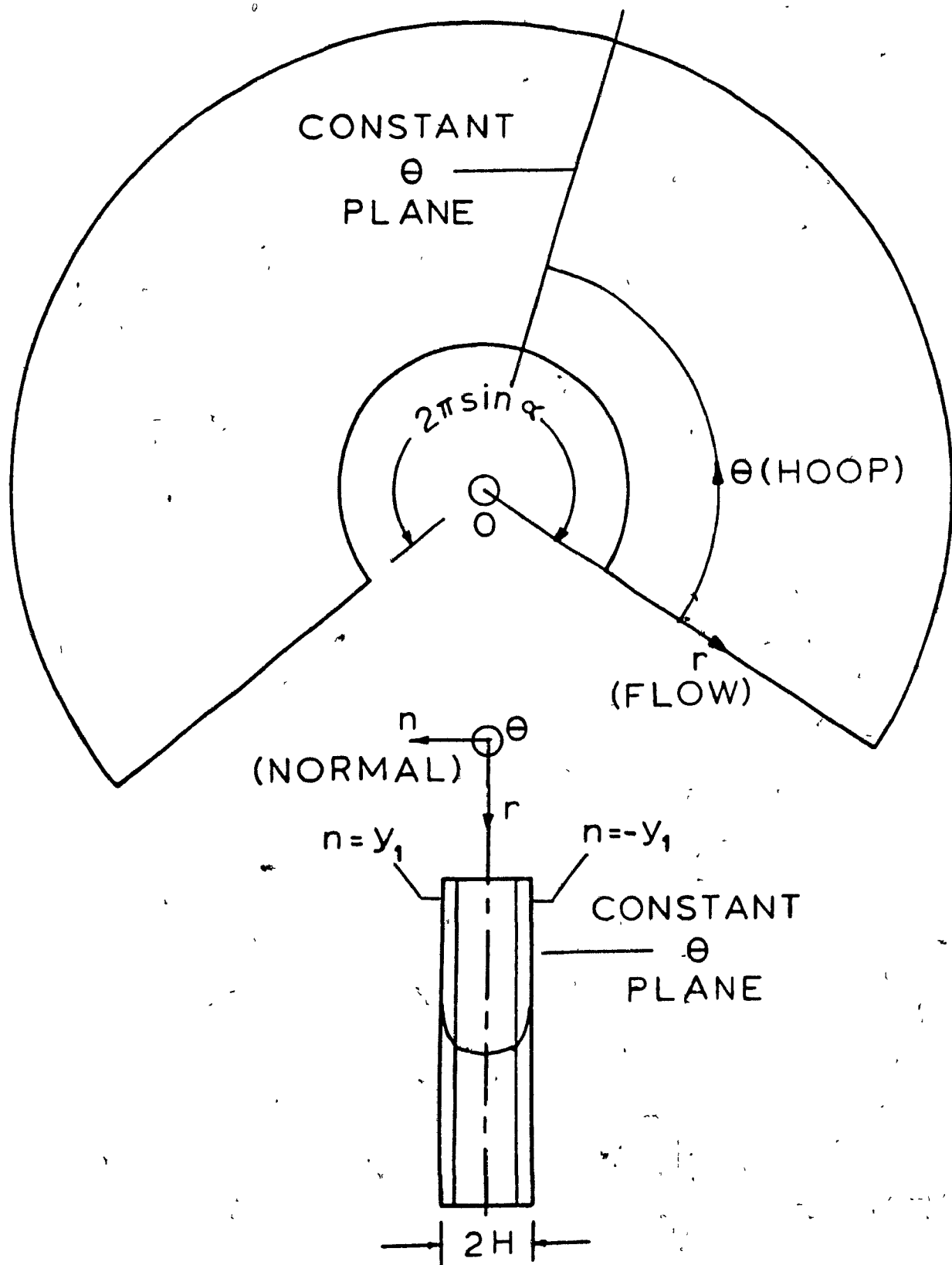


Figure 4.3 Cylindrical Coordinate System for the "Lay Flat" Geometry.

at the channel walls. Boundary conditions (ii) and (iii) ensure the continuity of the shear stress and velocity across the melt interfaces. Integration of Equation (4.3) gives the following expressions for the velocity profiles in layers I and II respectively.

$$\text{Layer I : } u_r = \left[\frac{y_1}{K_1} \left(-\frac{dP}{dr} \right) \right]^{1/n_1} \left(\frac{y_1 n_1}{n_1+1} \right) \left[1 - \left(\frac{n}{y_1} \right)^{1+1/n_1} \right] + \left[\frac{H}{K_2} \left(-\frac{dP}{dr} \right) \right]^{1/n_2} \left(\frac{H n_2}{n_2+1} \right) \left[1 - \left(\frac{y_1}{H} \right)^{1+1/n_2} \right] \quad (4.4)$$

$$\text{Layer II : } u_r = \left[\frac{H}{K_2} \left(-\frac{dP}{dr} \right) \right]^{1/n_2} \left(\frac{H n_2}{n_2+1} \right) \left[1 - \left(\frac{n}{H} \right)^{1+1/n_2} \right] \quad (4.5)$$

The pressure gradient $(-dP/dr)$ in the above equations is the local pressure gradient, which varies with r along the flow channel due to the expanding geometry. The volumetric flow rates for the individual layers, however, must remain constant. The volumetric flow rate Q_I of the SF RTP melt is as follows.

$$Q_I = \int_{-y_1}^{y_1} 2\pi r \sin \alpha u_r \, dn \quad (4.6)$$

$$= 4\pi r \sin \alpha \left[\frac{y_1}{K_1} \left(-\frac{dP}{dr} \right) \right]^{1/n_1} \left(\frac{n_1}{2n_1+1} \right) y_1^2 + 4\pi r \sin \alpha \left[\frac{H}{K_2} \left(-\frac{dP}{dr} \right) \right]^{1/n_2} \left(\frac{n_2}{n_2+1} \right) \left[1 - \left(\frac{y_1}{H} \right)^{1+1/n_2} \right] y_1 H$$

Due to the symmetry of the velocity profile, the flow rates of the inner and outer unreinforced surface layers are equal. The total flow rate Q_{II} for both of these layers is as follows.

$$\begin{aligned}
 Q_{II} &= \int_{-H}^{-y_1} 2\pi r \sin\alpha u_r \, dn + \int_{y_1}^H 2\pi r \sin\alpha u_r \, dn \quad (4.7) \\
 &= 4\pi r \sin\alpha \left[\frac{H}{K_2} \left(-\frac{dP}{dr} \right) \right]^{1/n_2} \left(\frac{H^{2n_2}}{n_2+1} \right) \times \\
 &\quad \left[1 - \frac{y_1}{H} - \left(\frac{n_2}{2n_2+1} \right) \left(1 - \left(\frac{y_1}{H} \right)^{2+1/n_2} \right) \right]
 \end{aligned}$$

Upon elimination of the pressure gradient term $(-dP/dr)$ between the Equations (4.6) and (4.7), the following relationship between the flow rates Q_I and Q_{II} is obtained.

$$\begin{aligned}
 Q_I &= H^3 \left(\frac{K_2}{K_1} \right)^{1/n_1} \left[\left(\frac{n_2+1}{n_2} \right) \frac{Q_{II}/H^3}{\left[1 - \frac{y_1}{H} - \left(\frac{n_2}{2n_2+1} \right) \left(1 - \left(\frac{y_1}{H} \right)^{2+1/n_2} \right) \right]^2} \right]^{n_2/n_1} \times \\
 &\quad \left(\frac{4\pi r \sin\alpha}{H} \right)^{1-(n_2/n_1)} \left(\frac{n_1}{2n_1+1} \right) \left(\frac{y_1}{H} \right)^{2+1/n_1} + \\
 &\quad \left[\frac{Q_{II} (y_1/H) \{ 1 - (y_1/H)^{1+1/n_2} \}}{1 - \frac{y_1}{H} - \left(\frac{n_2}{2n_2+1} \right) \left(1 - \left(\frac{y_1}{H} \right)^{2+1/n_2} \right)} \right] \quad (4.8)
 \end{aligned}$$

De-dimensionalizing Equation (4.8) by use of the channel half-thickness H and the rheological parameters K_1 , K_2 , n_1 and n_2 yields the following equation.

$$Q_I^* = \left[\left(\frac{n_2+1}{n_2} \right) \frac{Q_{II}^*}{\left[1 - \frac{y_1}{H} - \left(\frac{n_2}{2n_2+1} \right) \left(1 - \left(\frac{y_1}{H} \right)^{2+1/n_2} \right) \right]} \right]^{n_2/n_1} x$$

$$\left(\frac{4\pi r \sin \alpha}{H} \right)^{1-(n_2/n_1)} \left(\frac{n_1}{2n_1+1} \right) \left(\frac{y_1}{H} \right)^{2+1/n_1} +$$

$$\frac{Q_{II}^* (y_1/H) \{ 1 - (y_1/H)^{1+1/n_2} \}}{\left[1 - \frac{y_1}{H} - \left(\frac{n_2}{2n_2+1} \right) \left(1 - \left(\frac{y_1}{H} \right)^{2+1/n_2} \right) \right]} \quad (4.9)$$

where $Q_I^* = \left(\frac{Q_I}{H^3} \right) \left(\frac{K_2}{K_1} \right)^{1/(n_2-n_1)}$

and $Q_{II}^* = \left(\frac{Q_{II}}{H^3} \right) \left(\frac{K_2}{K_1} \right)^{1/(n_2-n_1)}$

Q_I^* and Q_{II}^* can be considered to be the dimensionless volumetric flow rates of SF RTP and unreinforced melts, respectively. Knowing these flow rates, Equation (4.9) can be solved iteratively in order to determine the location of the interface y_1 at any distance r from the origin. Once the interface location is known, the local pressure gradient $(-dp/dr)$ at that location can be determined using Equation (4.6) or (4.7). Finally, knowing the local pressure gradient $(-dp/dr)$ and y_1 at any r , the velocity profiles in the middle SF RTP layer and the surface unreinforced layers can be determined using Equations (4.4) and (4.5) respectively.

It is important to note that the location of the interface y_1 varies along the channel in the flow direction, as Equation

(4.9) contains a term in r . Only when the power-law indices of the SF RTP and the unreinforced melt are equal, does Equation (4.9) become independent of r , and y_1 does not vary along the flow path. The variation of the interface location along the flow direction implies that there is a component of the velocity vector, u_n , in the normal direction, n , in addition to that in the main flow direction, u_r . The streamlines are hence not exactly parallel to the channel walls throughout the die gap as was the case for simple extrusion. Since in the subsequent analysis of the fiber motion using Jeffrey's Equations, it is assumed that the fibers travel with the local fluid velocity, it is necessary to determine the magnitude and direction of the velocity vector at each point of interest.

A rigorous derivation of the flow field would involve a simultaneous solution of the dynamic equations in the r and n directions. This is, however, extremely involved. In such cases, it is often convenient to follow the two-dimensional parabolic method of solution suggested by Patankar and Spalding (69). According to this procedure, when there is a predominant component of velocity in a given direction in a two dimensional flow field, an approximate numerical solution for the velocity field can be obtained as follows. First the dynamic equation in the main flow or streamwise direction is solved to derive the velocity component in that direction. The variation of pressure in the cross stream direction (direction n in the above case) is assumed to be negligible. The cross stream velocity component can

then be obtained by solving the continuity equation applied in the vicinity of the point of interest.

In the above analysis, the velocity component in the r direction, u_r , is the predominant one and is given by Equations 4.4 and 4.5. At any point, the continuity equation is:

$$\frac{1}{r} \frac{\partial}{\partial r}(ru_r) + \frac{\partial u_n}{\partial n} = 0 \quad (4.10)$$

This equation was solved by the numerical discretization scheme described in Appendix 4.1. The following boundary condition has to be satisfied.

$$u_n = 0 \quad \text{at } n = \pm H$$

Once the components u_r and u_n are known at the point of interest, the magnitude and direction of the velocity vector, $\vec{u}_s$ at that point can be determined as follows.

$$|u_s| = \sqrt{(u_r^2 + u_n^2)} \quad (4.11)$$

$$\tan \beta = \frac{u_n}{u_r} \quad (4.12)$$

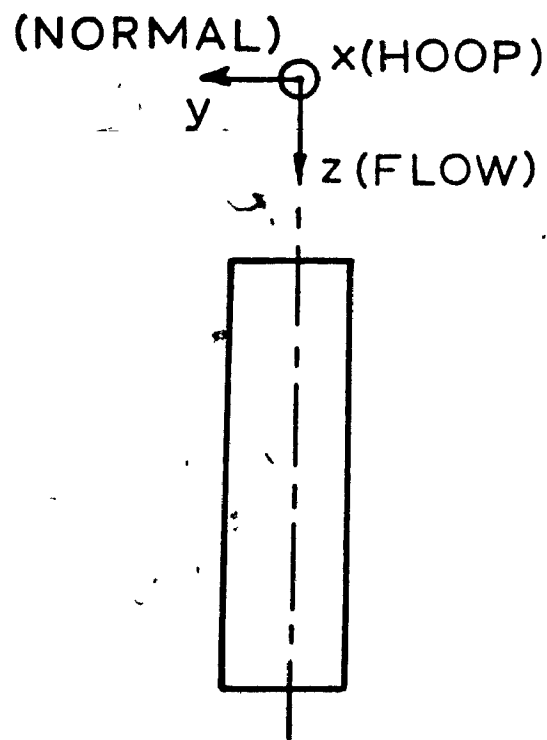
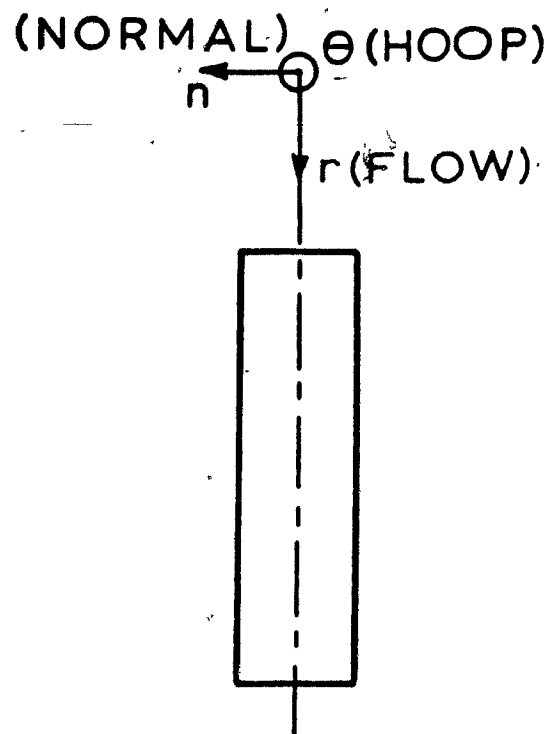
where $|u_s|$ is the magnitude of the velocity vector and β is the angle it makes with the r -axis. The gradients of the velocity components at the point of interest can be determined as discussed in Appendix 4.1. It should be noted that according to the above analysis, there is no component of velocity in the θ direction so that the streamlines originating in a given θ -constant plane remain in that plane along the entire flow path.

4.1.2 Individual Fiber Motion

Once the velocity field is determined, Jeffrey's equations (Equations 3.1 and 3.2) can be used to describe the travel of an individual fiber through the channel of the die. Jeffrey's equations are given in a Cartesian frame of reference, and it is convenient to use them in that form. A Cartesian frame of reference is hence considered in a constant θ plane normal to the channel wall, as shown in Figure 4.4. The z-axis is in the flow direction along the channel center line, and hence is coincident with the r-axis of the previously defined cylindrical coordinate system. The y-axis is normal to the channel wall, and hence is coincident with the n-axis of the cylindrical coordinate system. The x-axis is in the local hoop direction, and hence corresponds to the θ -axis. The velocity components u_r and u_n and their various gradients are thus equivalent to the velocity components u_z and u_y and their corresponding gradients respectively. The rate of deformation tensor in the Cartesian frame of reference is hence given as follows:

$$\underline{\Delta} = \begin{bmatrix} 2 \frac{\partial u_x}{\partial x} & 0 & 0 \\ 0 & 2 \frac{\partial u_y}{\partial y} & \frac{\partial u_z}{\partial y} \\ 0 & \frac{\partial u_z}{\partial y} & 2 \frac{\partial u_z}{\partial z} \end{bmatrix} \quad (4.13)$$

The components of the stretching rate tensor and the vorticity



CONSTANT θ PLANE

Figure 4.4 Cartesian Frame of Reference in a Constant θ Plane.

tensor appearing in Jeffrey's equations are as follows.

$$\begin{aligned}
 D_{22} &= \frac{\partial u_y}{\partial y} & D_{33} &= \frac{\partial u_z}{\partial z} \\
 D_{12} &= 0 & D_{23} &= \frac{1}{2} \frac{\partial u_z}{\partial y} & D_{13} &= 0 \\
 Z_1 &= \frac{1}{2} \frac{\partial u_z}{\partial y} & Z_2 &= 0 & Z_3 &= 0
 \end{aligned} \tag{4.14}$$

These components have to be evaluated as described above. Their substitution into Jeffrey's equations gives the following relationships.

$$\frac{d\phi}{dt} = u_s \frac{d\phi}{ds} = Z_1 + B \left[D_{23} \cos 2\phi - \frac{1}{2} (D_{22} - D_{33}) \sin 2\phi \right] \tag{4.15}$$

$$\begin{aligned}
 \frac{d\psi}{dt} = u_s \frac{d\psi}{ds} = B \left[\frac{1}{2} D_{23} \sin 2\phi \sin 2\psi + \frac{1}{4} (D_{22} - D_{33}) \cos 2\phi \sin 2\psi \right. \\
 \left. + \frac{3}{4} (D_{22} + D_{33}) \sin 2\psi \right] \tag{4.16}
 \end{aligned}$$

where u_s represents the magnitude of the velocity vector at a given point and ds is an elemental displacement in the direction of this vector. The above equations must be solved numerically in order to describe the change in fiber orientation along the flow path and, in particular, its orientation at the die exit.

4.1.3 Fiber Orientation Distribution

The fiber orientation distribution can be derived by the same procedure as that described in Section 3.3.3. The ranges of

the angles ϕ and ψ are divided into a number of intervals, and a representative initial orientation (ϕ_0, ψ_0) is selected for each of the intervals. The components of the velocity vector u_z and u_y and their required gradients at the point occupied by a given fiber are calculated by the solution of the momentum, continuity and constitutive equations as described above. The magnitude (u_s) and direction (β) of the velocity vector $\vec{u}_s$ at that point are then determined. Jeffrey's equations are then solved numerically to calculate the change in the fiber orientation occurring during an elemental displacement ds of the fiber in the direction of the velocity vector. The computations are carried out from the entrance ($z=z_0$) to the exit ($z=z_1$) of the expanding section. An overall orientation distribution $\bar{f}(\psi)$ with respect to the hoop direction is derived at the die exit by averaging over the range of values of ϕ according to Equation 3.24. It may be remembered that a high value of $\bar{f}(\psi)$ near $\psi=0^\circ$ indicates a strong hoop orientation of fibers, while a high value of the function near $\psi=90^\circ$ indicates a strong axial orientation of fibers. In all the results presented below, the initial orientation distribution is assumed to be uniformly random in space, i.e.,

$$f_0(\psi_0) = 1 \quad \text{for all } \phi_0(i) \quad (4.17)$$

A computer listing of the FORTRAN program used to carry out the computations is provided in Appendix 4.2.

4.1.4 Results and Discussion

The shape parameter B was set equal to 1 for all the results

presented below. The power law indices of the SF RTP and the unreinforced melts were chosen to be $n_1=0.5$ and $n_2=0.7$, respectively, implying that the SF RTP melt is more shear-thinning than the unreinforced resin. The following die parameters were chosen.

$$R_0/H = 6 \quad R_1/R_0 = 8/3 \quad \alpha = 60^\circ$$

Dimensionless flow rates of $Q_I^* = 6.0$ and $Q_{II}^* = 2.0$ were selected for the SF RTP and the unreinforced melts, respectively for the purpose of illustration. The above set of values of the coextrusion parameters is referred to as "condition I" in the following discussion. Due to the assumption of symmetry of the flow profile about the channel center line, the dimensionless flow rates for the inner and outer unreinforced surface layers are both equal to 1.0.

As mentioned above, the interface location y_1 varied somewhat along the flow direction. The variation, however, was not large, as the selected power-law indices for the SF RTP and the unreinforced melts were not very different. The thickness of the middle layer was hence approximately equal to $2y_{10}$ along the entire flow channel, where y_{10} is the location of the interface at the entrance to the expanding section of the die ($z=z_0$). For comparison purposes, the case of simple extrusion of SF RTP was also considered, with the channel thickness equal to $2y_{10}$ and other geometrical parameters the same as for the coextrusion case ($R_1/R_0 = 8/3$, $\alpha = 60^\circ$ and $n_1 = 0.5$), and is referred to as extrusion under "condition II" in the following. This case

represents a simple extrusion process for SF RTP pipe with the same consumption of cost-controlling SF RTP resin as the coextrusion process under condition I.

The velocity profiles for coextrusion (condition I) and simple extrusion (condition II) at the channel entrance ($z = z_0$) are shown in Figure 4.5. The shear rate profiles at the entrance for both the cases are shown in Figure 4.6. It may be noted that the shear rate has been de-dimensionalized with respect to the flow rate Q_I and the interface location y_1 as follows.

$$\text{Coextrusion : } \left(\frac{\partial u_z}{\partial y} \right)^* = \frac{4\pi z \sin \alpha y_1^2}{Q_I} \left(\frac{\partial u_z}{\partial y} \right) \quad (4.18)$$

$$\text{Simple Extrusion : } \left(\frac{\partial u_z}{\partial y} \right)^* = \frac{4\pi z \sin \alpha y_1^2}{Q_I} \left(\frac{\partial u_z}{\partial y} \right) \quad (4.19)$$

It is seen that the presence of the surface layers in the case of coextrusion (condition I) significantly reduces the shear rate in the middle layer of SF RTP. In the case of simple extrusion (condition II), the velocity goes from a maximum at the center line to zero at the surface giving rise to a significantly higher shear rate, particularly in the vicinity of the wall. It may be noted from Figure 4.6 that in the case of coextrusion, there is an abrupt change in the shear rate between the middle layer and the surface layer at the interface due to the corresponding change in the power-law index. The shear stress is, however, continuous across the interface as implied by Equation 4.4 and shown schematically in Figure 4.7.

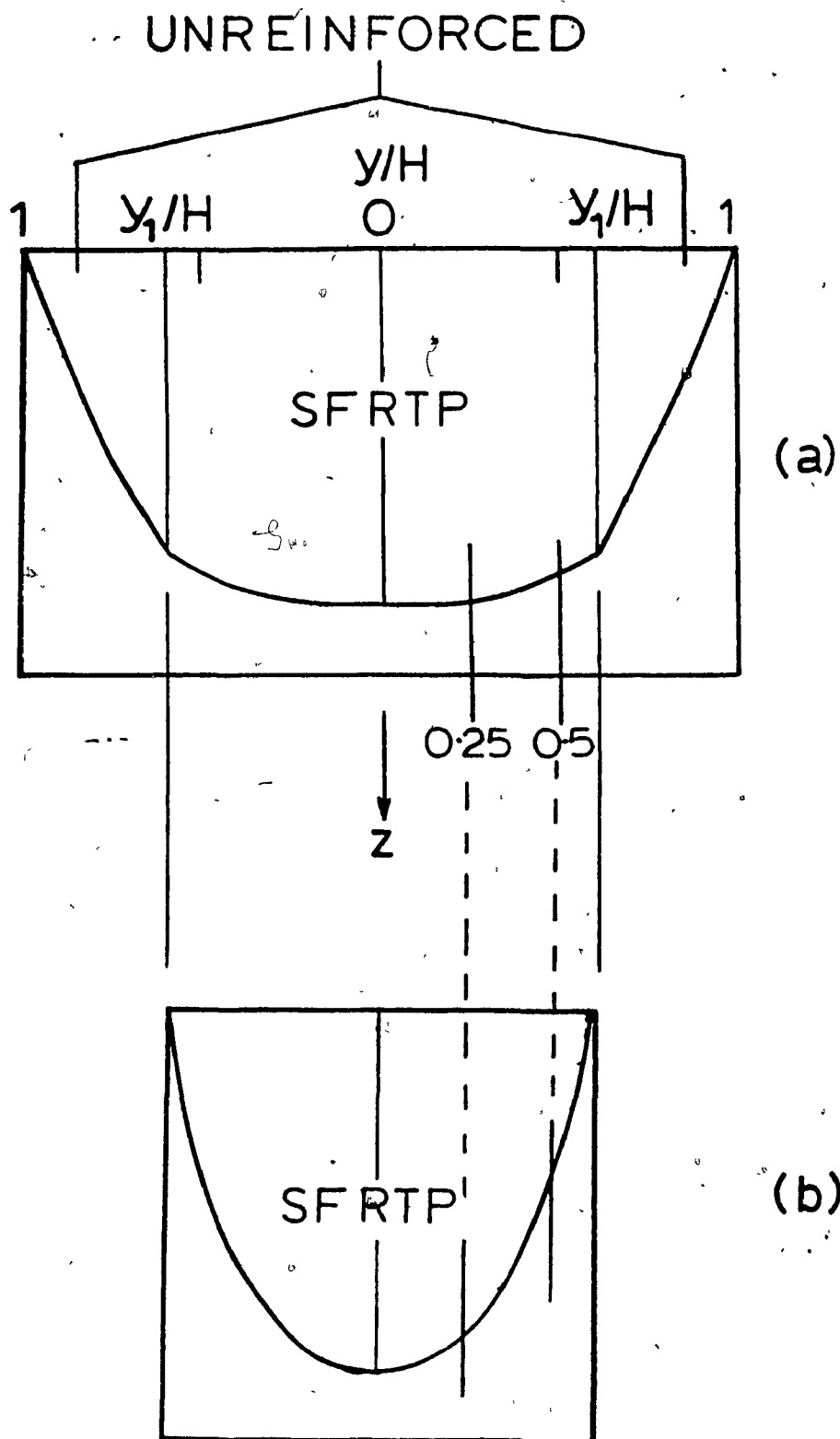


Figure 4.5 Velocity Profiles in the Expanding Channel Die
at $z = z_0$
(a) Coextrusion and (b) Simple Extrusion.

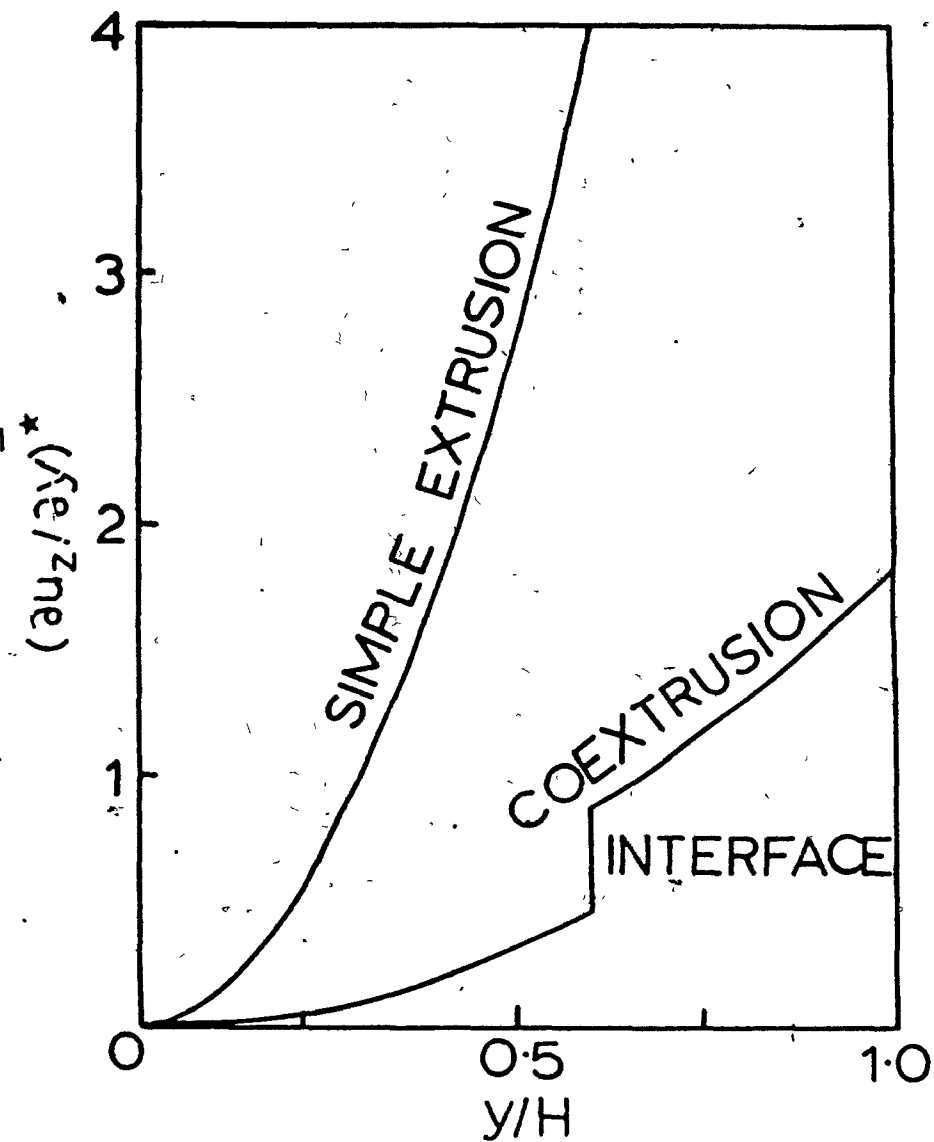


Figure 4.6 Dimensionless Shear Rate Profile at $z = z_0$.

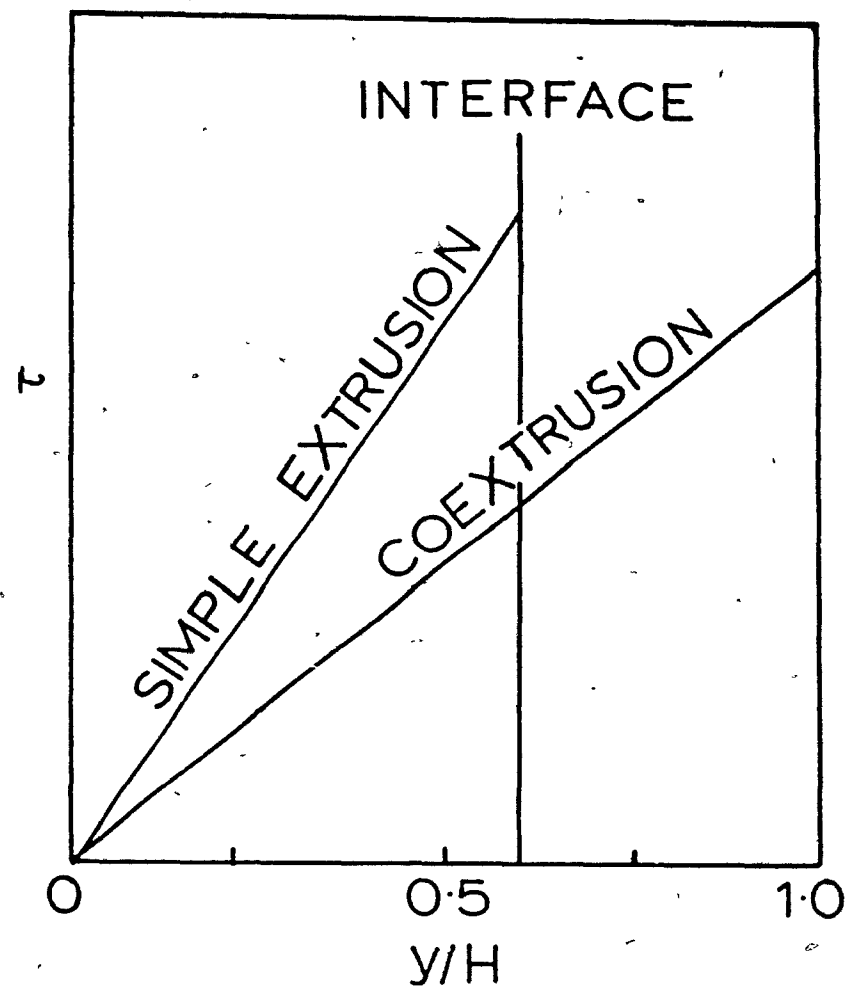


Figure 4.7 Shear Stress Profile across Die Channel (Schematic).

Due to the increase in the channel cross sectional area along the flow path, the average velocity in the flow direction decreases from $z=z_0$ to $z=z_1$. Table 4.1 gives the interface location (y_1/H) and the velocity at the interface as fractions of the maximum velocity at the center line (u_{zy1}/u_{zm}) at the entrance and at the exit of the expanding section for coextrusion (condition I). The fractional velocity at the interface indicates the degree of lubrication of the middle layer by the surface layers. As mentioned above, the interface moves slightly outward toward the wall along the flow channel. Also, the fractional velocity at the interface (u_{zy1}/u_{zm}) increases somewhat from the entrance to the exit.

It must be noted that entrance and exit effects have been neglected in the above analysis, although they may influence the interface location and the velocity profile. Also, once the melt leaves the die, the velocity profile must eventually become flat as illustrated in Figure 4.8. A redistribution of the layer thicknesses, therefore, must occur with the thicknesses of the individual layers becoming proportional to the respective flow rates.

Three specific locations in the middle SFRTF layer, viz., center line ($y/H = 0$), near-center ($y/H = 0.25$) and midway ($y/H = 0.5$), were considered for evaluating the fiber orientation distribution functions for the case of coextrusion (condition I).

Table 4.1

Results of the Theoretical Analysis

	Coextrusion	Simple Extrusion
Power-Law Parameters	$n_1 = 0.5$ $n_2 = 0.7$	$n_1 = 0.5$ -
Interface Location (y_1/H)		
at i) $z = z_0$ (Entrance)	0.59	-
ii) $z = z_1$ (Exit)	0.60	-
Fractional Interfacial Velocity (u_{zy1}/u_{zm}) at		
i) $z = z_0$ (Entrance)	0.85	-
ii) $z = z_1$ (Exit)	0.89	-
Mean Angle of Orientation w.r.t. Hoop Direction at		
i) $y/H = 0$	23°	23°
ii) $y/H = 0.25$	26°	51°
iii) $y/H = 0.5$	40°	72°

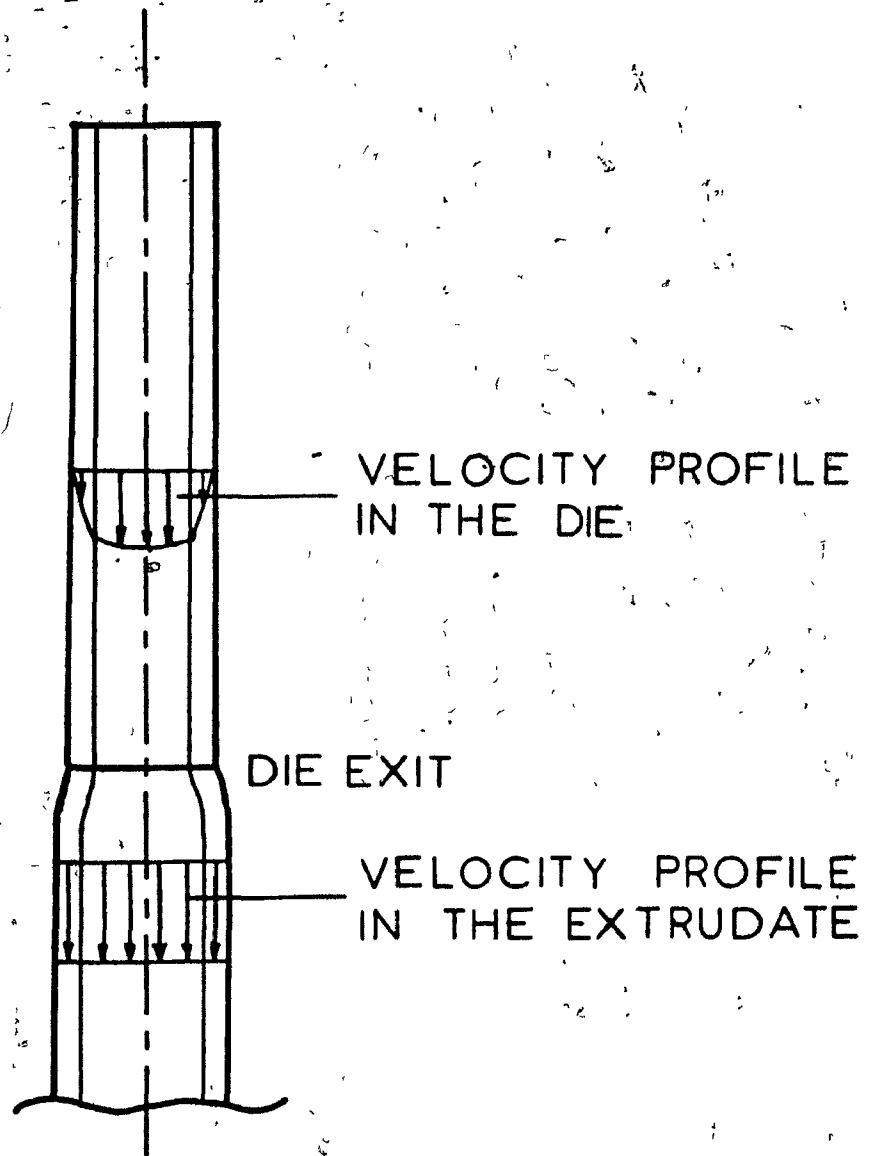


Figure 4.8 Change in the Velocity Profile at the Exit from the Die.

The distribution functions were also calculated at the corresponding locations for the case of simple extrusion (condition II) as illustrated in Figure 4.5. It may be noted that the midway location is equidistant from the center line and the die wall in the case of coextrusion (condition I), and is thus close to the interface. In the case of simple extrusion (condition II), the corresponding location is close to the die wall.

Figure 4.9a shows the fiber orientation distribution function $\bar{f}(\psi)$ at the center line ($y/H = 0$) for the cases of coextrusion and simple extrusion. A rather strong hoop orientation of fibers is developed, as indicated by a sharp peak of the distribution function at $\psi = 0^\circ$. Also, there is no difference between the results for the coextrusion and simple extrusion cases, as the shear rate is zero at this location. The fiber orientation is thus completely determined by the hoop stretching of the melt due to the expanding geometry. The ψ -distribution at the near-center location ($y/H = 0.25$) is shown in Figure 4.9b. A stronger hoop orientation of fibers is predicted for the coextrusion case. For the case of simple extrusion, on the other hand, a mild peak at $\psi = 90^\circ$ is predicted implying that a preferential axial orientation of fibers is developed. The effect of surface "lubrication" through coextrusion is most visible at the midway location ($y/H = 0.5$). The distribution functions at this location are shown in Figure 4.9c. For the simple extrusion case, a predominantly axial orientation of fibers is predicted,

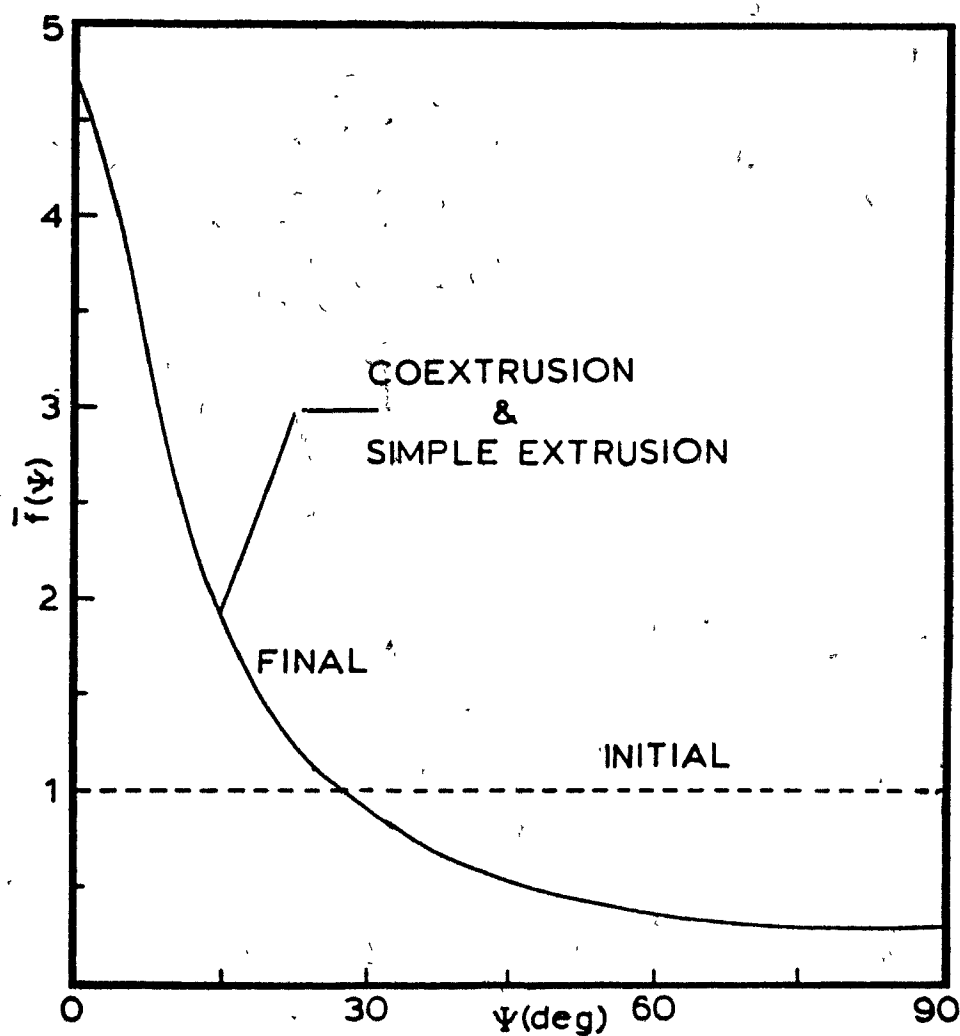


Figure 4.9a Fiber Orientation Distribution Function at $y/H = 0$ (Center Line)

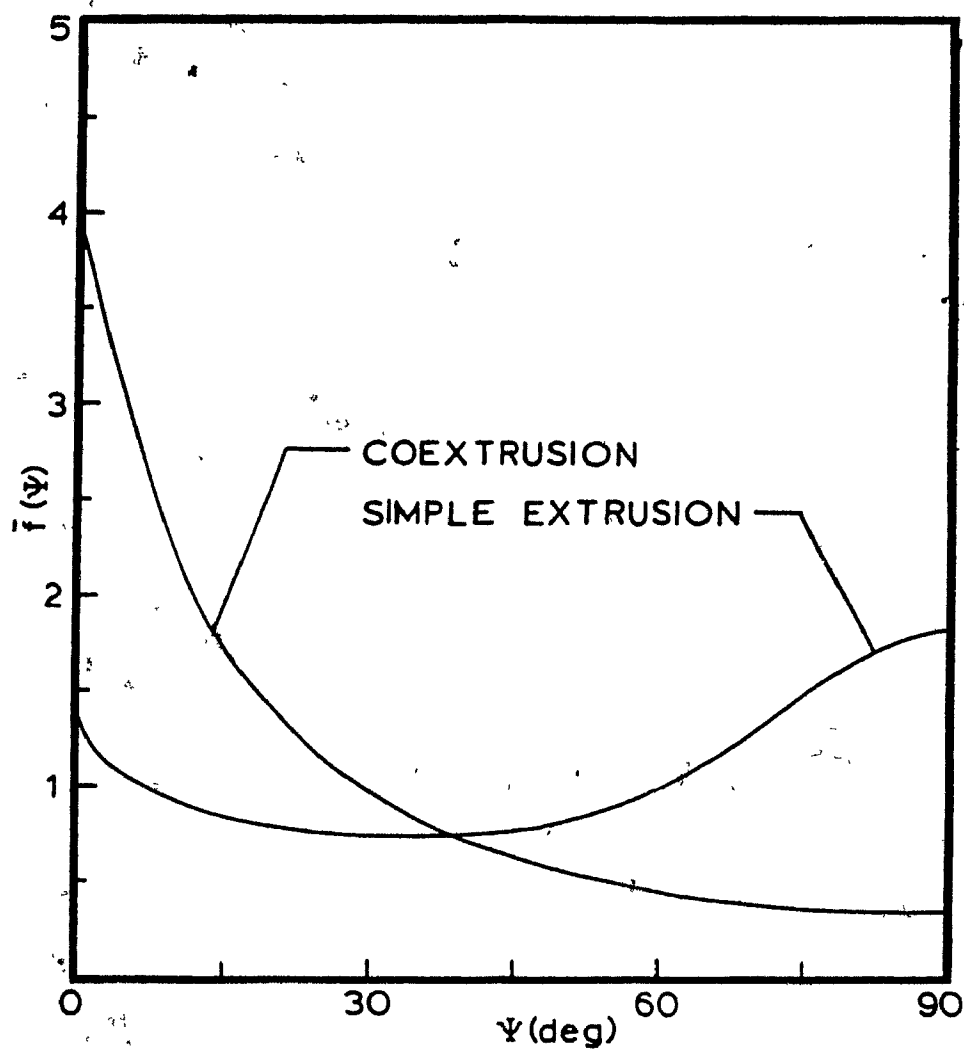


Figure 4.9b Fiber Orientation Distribution Function at $y/H = 0.25$ (Near-Center)

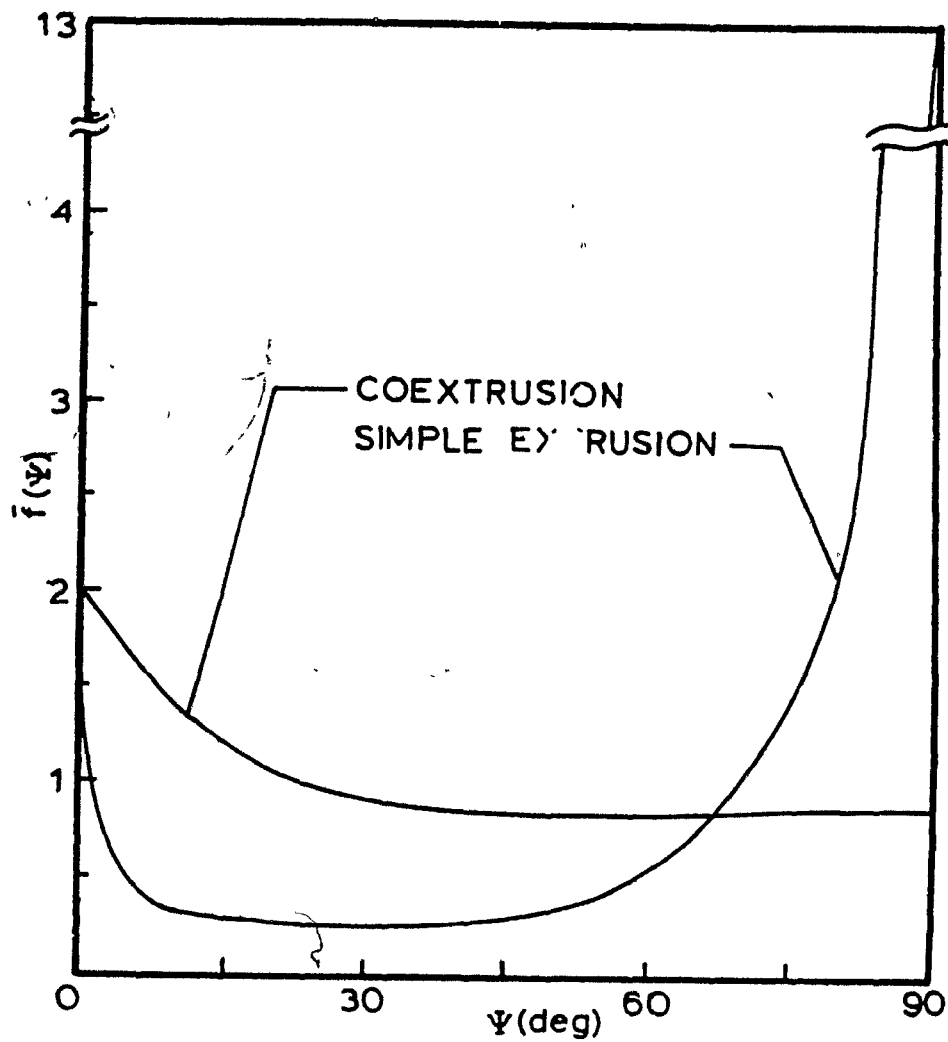


Figure 4.9c Fiber Orientation Distribution Function
at $y/H = 0.5$ (Midway)

while for the coextrusion case a preferential hoop orientation of fibers is still present.

An average angle of orientation, $\bar{\psi}$, with respect to the hoop direction was also calculated at each of the above locations using the method described in Section 3.3.3. The values of $\bar{\psi}$ are listed in Table 4.1 for coextrusion (condition I) and simple extrusion (condition II). It is seen that in the case of simple extrusion, $\bar{\psi}$ varies from 23° at the center line to 72° near the wall. In the case of coextrusion under the above conditions, it only varies from 23° to about 40° over an equivalent distance. These results clearly demonstrate the advantages offered by the proposed coextrusion process in promoting a fairly uniform hoop orientation of fibers in the middle SF RTP layer.

4.1.4.1 Effects of Rheological, Geometrical and Processing Parameters On Fiber Orientation Distribution

As in the case of simple extrusion (cf. Section 3.3.4), rheological and geometrical parameters have significant effects on the fiber orientation distribution developed in the SF RTP layer during coextrusion. In addition, in the coextrusion of melts of given power-law indices through an expanding channel die of specified geometry, the relative flow rates of the two melts govern the overall velocity profile in the channel and, in turn, the degree of lubrication of the middle SF RTP layer achieved, and hence have a strong effect on the fiber orientation distribution.

While the model developed above is not intended to provide predictions for quantitative comparison with the experimental results, it can be used to investigate qualitatively the effects of the above parameters. Such an investigation can provide general guidelines for die design and optimal coextrusion conditions for the control of fiber orientation distribution in the middle layer.

The effects of the power-law indices and the melt flow rates on the degree of lubrication are coupled. In order to illustrate this, two cases representing two types of relative shear-thinning behaviors of the SF RTP and unreinforced melts are considered in the following, and the above analysis is used to identify dimensionless melt flow rates Q_I^* and Q_{II}^* , which would improve the degree of lubrication of the SF RTP layer.

In the first case, the power law index of the unreinforced melt is higher than that of the SF RTP melt, as illustrated schematically by the flow curves in Figure 4.10. The addition of reinforcing fibers or other fillers to a polymer melt usually leads to a moderate increase in both the viscosity and the shear thinning tendency (70). Hence the case where the matrix resin of the SF RTP itself is used for the unreinforced surface layers for coextrusion would correspond to such a situation. The flow curves of the two melts are assumed to intersect and cross over at a shear rate $\dot{\gamma}^*$ given by:

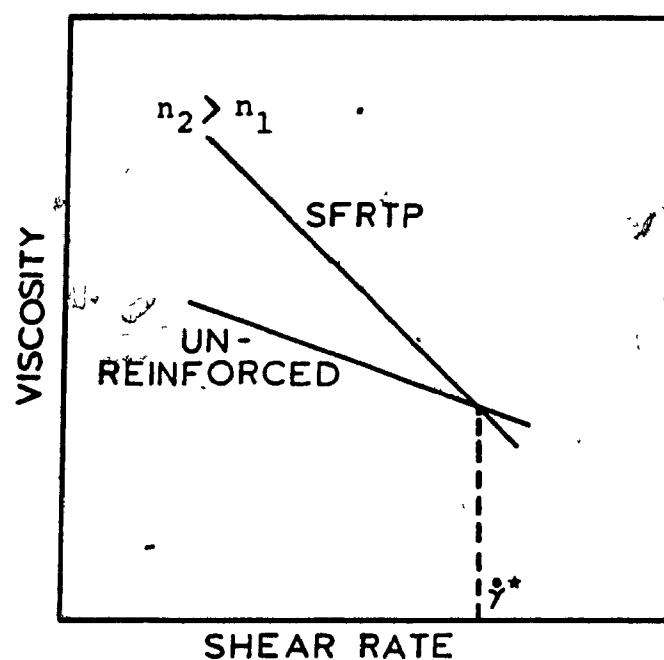


Figure 4.10 Flow Curves for the SF RTP and Unreinforced Melts (SF RTP Melt more Shear-Thinning than Unreinforced Melt).

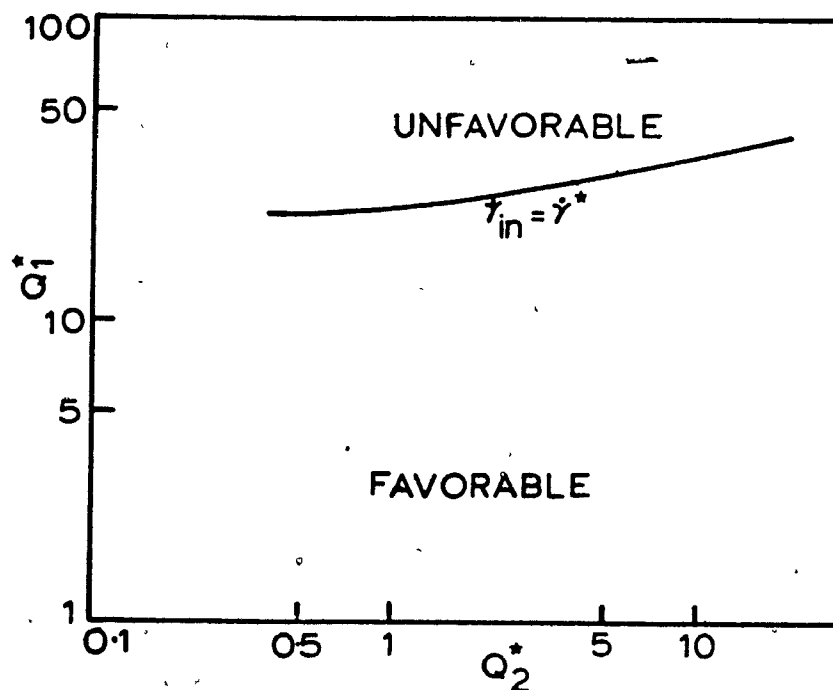


Figure 4.11 Conditions Characterizing Favorable and Unfavorable Regions for Lubrication of SF RTP Layer in Coextrusion. ($n_1 = 0.5$, $n_2 = 0.7$)

$$\dot{\gamma}^* = \left(\frac{K_2}{K_1} \right)^{1/(n_1 - n_2)} \quad (4.20)$$

In order to achieve the lubrication of the middle SF RTP layer, its viscosity at the interface must be higher than that of the unreinforced melt, and the interfacial shear rate in either layer must therefore be less than $\dot{\gamma}^*$. Using the coextrusion analysis developed above, it is possible to determine values of the flow rates Q_I^* and Q_{II}^* that satisfy this condition. For a given value of Q_I^* , the interface location y_1^* that gives rise to an interfacial shear rate of $\dot{\gamma}^*$ can be determined using Equations 4.4 and 4.6. Using Equation 4.9, the corresponding limiting value of Q_{II}^* can then be determined.

Figure 4.11 shows such a relationship between Q_I^* and Q_{II}^* for the case of coextrusion (condition I) considered in the previous section. The region under the curve, marked "favorable" represents the region in which the interfacial shear rate is less than $\dot{\gamma}^*$ so that surface lubrication of the middle layer is achieved. Moreover, the farther the conditions of coextrusion are from the curve, the greater is the degree of lubrication achieved. The area above the curve, marked "unfavorable", represents the conditions for which the interfacial shear rate is greater than $\dot{\gamma}^*$ so that a lubrication effect is not achieved.

The second case is opposite to the one considered above. In this case, the unreinforced melt has a power-law index lower than

that of the SF RTP melt, as shown schematically in Figure 4.12. Such a situation can be encountered when the surface layers are of a resin different from the SF RTP matrix resin. In Figure 4.12, the two power-law flow curves are shown to intersect at a shear rate $\dot{\gamma}^*$ given by Equation 4.21. In this case, the shear rate at the interface must be higher than $\dot{\gamma}^*$ in order to achieve the lubrication effect. In Figure 4.13 is shown the relationship between the two flow rates Q_I^* and Q_{II}^* that gives rise to an interfacial shear rate equal to $\dot{\gamma}^*$ for power-law indices of $n_1=0.5$ (SF RTP) and $n_2=0.3$ (unreinforced) (the geometrical parameters being the same as before). The area above the curve now represents the "favorable" region for which the lubrication effect is achieved.

The effects of geometrical parameters have been discussed in detail in Section 3.3 for the case of simple extrusion of SF RTP. The same general conclusions are also qualitatively valid for the case of coextrusion. A thicker channel and a larger angle of expansion can lead to a higher degree of fiber orientation in the hoop direction within a certain range. An increase in the degree of expansion (R_1/R_0) also leads to an increase in the degree of hoop fiber orientation up to a certain point, beyond which it does not have a significant influence.

The theoretical model developed above provides qualitative insight into the interaction of various rheological and geometrical parameters, and the extrusion conditions in

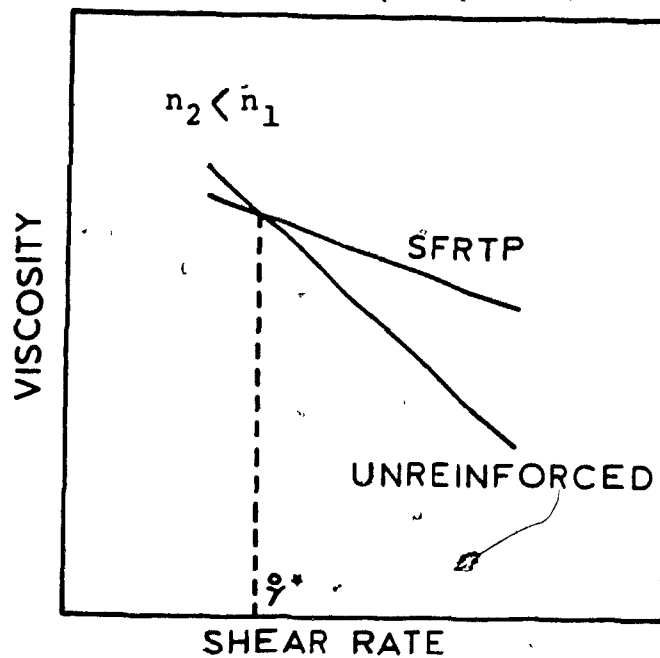


Figure 4.12 Flow Curves for the SF RTP and Unreinforced Melts (SF RTP Melt less Shear-Thinning than Unreinforced Melt).

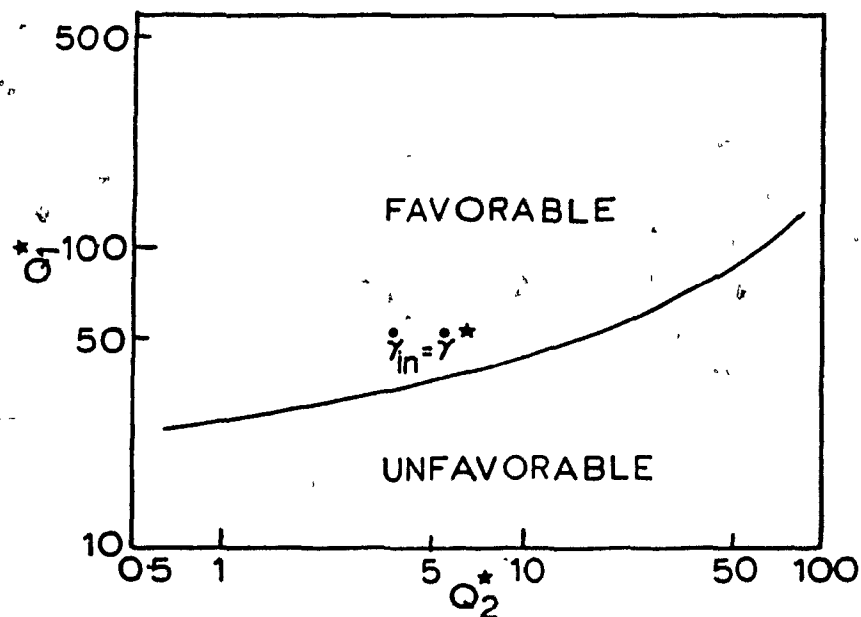


Figure 4.13 Conditions Characterizing Favorable and Unfavorable Regions for the Lubrication of SF RTP Layer in Coextrusion.
($n_1 = 0.5$, $n_2 = 0.3$)

determining the fiber orientation distribution in the coextruded pipe. It also provides guidelines for die design and extrusion conditions in order to optimize the degree of hoop orientation of fibers in the SFRTF layer. It must be remembered that entrance and exit effects have not been considered in the above analysis. Also, the fiber-fiber and fiber-wall interactions and other rheological characteristics of the resin (such as viscoelasticity) can have strong effects on the fiber orientation distribution. There is also a possibility of interfacial instability in coextrusion, which could influence the fiber orientation distribution, and even make the product unacceptable. The causes of such instabilities are not understood very well as discussed in Section 4.2.1 (74,77-80). Finally, the post extrusion operations can also influence the fiber orientation distribution as discussed in Section 4.2.6.

4.2 Experimental Study

4.2.1 Coextrusion Process

The objective of the experimental study was to produce pipe having a three-layer wall structure, the inner and outer surface layers being of an unreinforced polymer and the middle layer of a polymer containing short fibers for reinforcement. In order to produce such a pipe by a single step coextrusion process, a coextrusion die was required to bring together the melt streams for various layers, form the desired layered structure, and then

extrude a continuous tubular profile.

Several investigators have studied the coextrusion process both theoretically and experimentally. Maclean (71) and Everage (72) have presented theoretical analyses of the equilibrium interfacial configuration in a stratified flow of two fluids of different viscosities. Based on the postulation of minimum viscous dissipation at the interface, they have shown that the energetically preferred configuration is one in which the higher viscosity component is encapsulated by the lower viscosity component. White and Lee (73) have also shown that for two Newtonian fluids in stratified, two phase flow between parallel plates, the higher viscosity fluid tends to push into the lower viscosity fluid forming an interface that bulges out into the low viscosity fluid before steady state is achieved. Southern and Ballman (74), Yu and Han (75) and Manigawa and White (76) have made similar observations in their coextrusion experiments using a variety of polymeric systems. As shown by White et al. (77) for the case of viscoelastic fluids, in addition to the difference in viscosity at the interface, the second normal stress difference can also influence the interface configuration significantly.

One of the important problems associated with the coextrusion process is maintaining stability at the interface. Interfacial instability can give rise to void formation or waviness, and thus to a deterioration of product quality. The most important cause of interfacial instability has been found to

be large differences between the viscosities of the two melts (74,77). Khan and Han (78) have shown that viscoelastic effects such as normal stress differences can also contribute significantly to interfacial instability. Shetty (79) has shown experimentally that interfacial instability can occur when the shear stress at the interface exceeds a certain critical value that is specific for the system under consideration. Schrenk et al. (80) have reported similar observations of the coextrusion of ABS and high impact PS.

In addition to the interfacial instability, differences in the crystallization characteristics of the polymers used to make a coextruded product can lead to quality deterioration. Han and Rao (81) have reported that in coextrusion wire coating, a non-circular distorted coating resulted when a highly crystalline polymer was used to form an inner layer inside a layer of an amorphous polymer, due to the difference in shrinkage upon solidification.

The success of a coextrusion process is highly dependent on the design of the coextrusion die. Two distinct methodologies have been used to design sheet coextrusion dies (82). The earlier method is to incorporate multiple channels or manifolds for the various melt streams. The melt streams are allowed to spread over the entire width of the die, and are combined in the land region just prior to the exit to form the final extrudate. In the newer approach, several polymer streams are brought together, and

combined within an adapter or feed-block, which may be a separate or integral part of the die. The arrangement of proper order and relative distribution of various layers is achieved in the feed-block, and the combined multiple layer stream subsequently flows to the die. Because of this, the die can be very similar to that for simple extrusion.

4.2.2 Coextrusion Equipment

The pipe coextrusion experiments were carried out at the Industrial Materials Research Institute (National Research Council of Canada) with the help of Messrs. F. A. Hamel and C. de Grandpré. Two extruders were used to produce the three-layer pipe; one supplying each type of melt. The coextrusion arrangement is shown schematically in Figure 4.14. A main extruder with a screw diameter of 63 mm (2.5 in) was used to supply the SFRTM melt, and the coextrusion die was mounted on it. A smaller extruder with a screw diameter of 32 mm (1.25 in) was used to supply the unreinforced polymer melt for both the inner and outer surface layers via a side feed port in the die. Specifications of the two extruders are given in Table 4.2. Distribution of the melt streams and rearrangement of the layers occurred inside the die. The design and construction of the die are discussed in detail in the next section. The coextrusion line included a vacuum cooling tank with a 50 mm (2 in) external pipe calibrator, a caterpillar haul-off, and an automatic saw cutter. Specifications of the auxiliary equipment are given in Table 4.2.

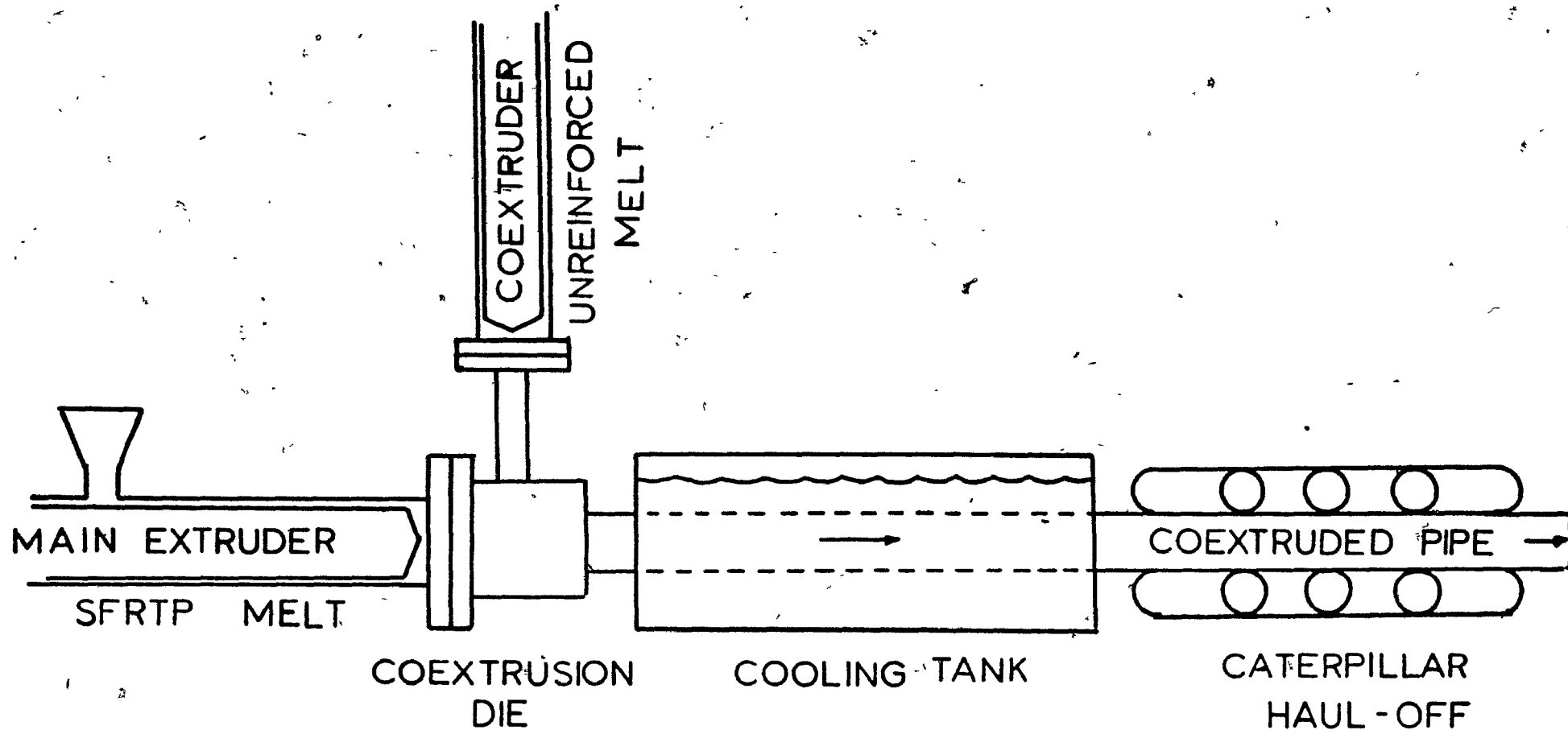


Figure 4.14 Pipe Coextrusion System (Schematic)

Table 4.2

Specifications for Pipe Extrusion System Equipment

(a) Main Extruder

Make : Danson Model 25D30V Heavy Duty Extruder
Screw Diameter : 63 mm (2.5 in)
Screw Type : Single Stage
Screw /D Ratio : 30:1
Barrel Heating/Cooling : 5 Zones, Electrical Heating,
Water Cooling
Drive Motor : 50 HP

(b) Coextruder

Make : Danson Model 1250 Heavy Duty Extruder
Screw Diameter : 32 mm (1.25 in)
Screw Type : Single Stage
Screw /D Ratio : 24:1
Barrel Heating/Cooling : 3 Zones, Electrical Heating,
Air Cooling
Drive Motor : 5 HP

(c) Cooling Tank

Make : Flag Machinery Vacuum Sizing Tank
Model MVS 3-14-4
Construction : Three Compartments of Length 3 m (10 ft)
0.6 m (2 ft) and 0.6 m (2 ft)
Calibrator : 50 mm (2 in) dia., 0.3 m (1 ft) Length

(d) Haul Off

Make : Flag Machinery Model PB-4-CL
Drive : 3 HP, Individual Adjustment for
Belt

(e) Saw Cutter

Make : Flag Machinery Model MST4
Saw : 0.3 m (1 ft) dia.
Drive : 1 HP

The complete coextrusion system is shown in Figure 4.15.

4.2.3 Coextrusion Die Design

The design of the coextrusion die was based primarily on the multi-manifold approach with some features of the feed-block approach.

Several factors had to be considered in designing the die. First of all, as indicated by the results of the theoretical analysis, it is desirable to subject all the three layers to flow through the expanding channel of the die in order to promote uniform hoop orientation of fibers throughout the thickness of the middle layer. In order to achieve this, the various melt streams forming the three layers of the pipe must be brought together prior to the expanding section. Also it may be remembered from the results of the simple extrusion study discussed in Chapter 3 that splitting and recombining a flow stream may lead to the formation of undesirable weld lines along the length of the pipe. The fiber orientation pattern in the regions of the weld lines is altered, and this can adversely affect pipe properties. The conventional method of pipe calibration requires blowing air into the extrudate through an air channel in the mandrel. Although calibration methods that do not require air blowing into the extrudate have been proposed, it was felt that the conventional procedure was appropriate due to ease of operation. It was hence necessary to provide a suitable

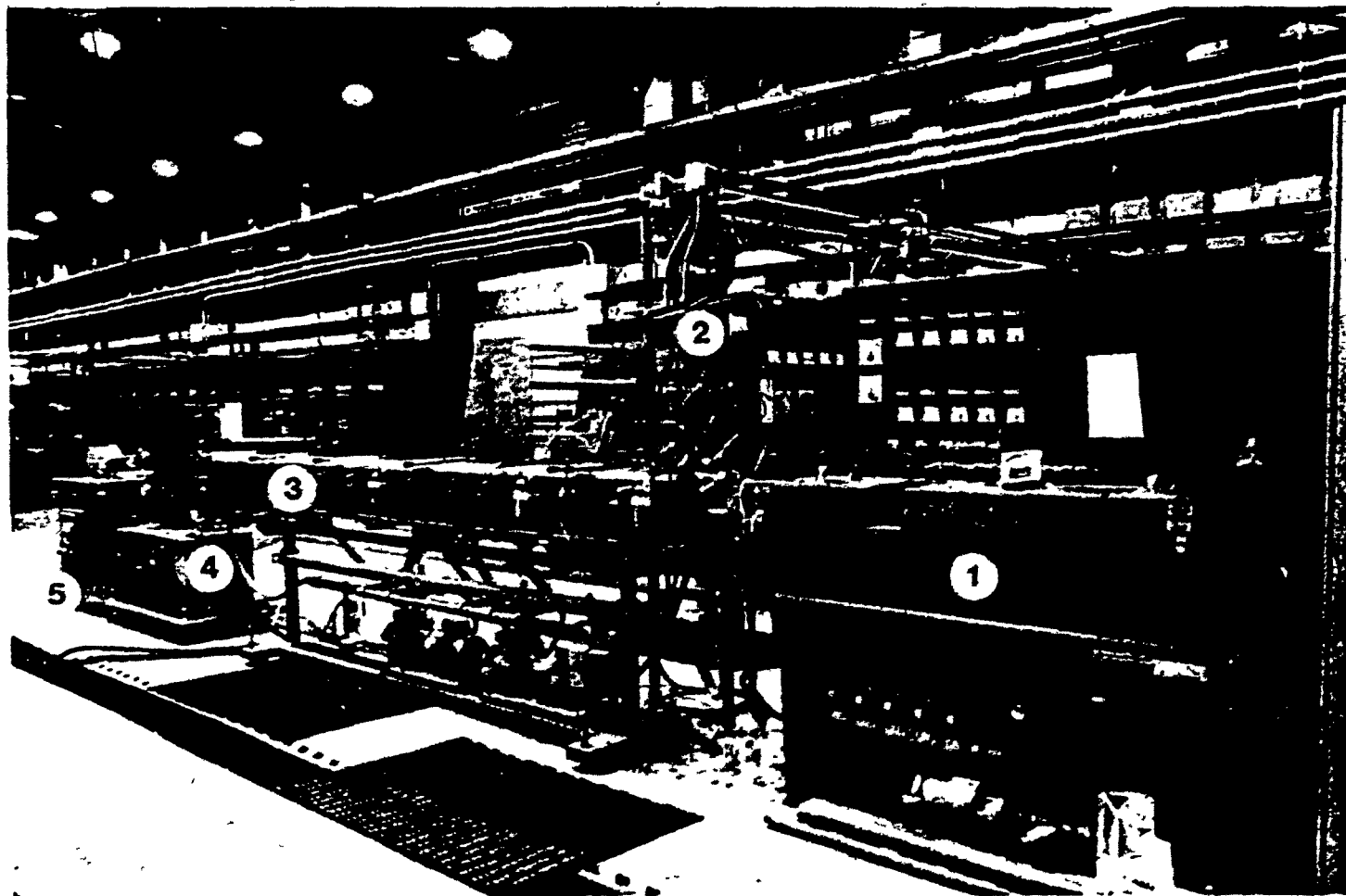


Figure 4.15 Pipe Coextrusion System

(1) Main Extruder	(2) Coextruder	(3) Vacuum Sizing/Cooling Tank
(4) Haul-Off	(5) Saw Cutter	

air channel in the die that could continue into the mandrel. In addition, the die had to be provided with a suitable adapter for its direct mounting on the main extruder supplying the SF RTP melt. Also, for ease of machining, assembly and operation, it was decided to provide a single feed port for the entry of the unreinforced melt into the die and appropriate flow channels for its subsequent division to form the inner and outer layers of the coextruded pipe.

The coextrusion die designed on the basis of the above strategy is shown schematically in Figure 4.16, while engineering drawings showing various sectional projections are included in Appendix 4.4. The die consists of six matching plates, a spider construction, an expanding section and an expanding mandrel. The multi-plate construction allowed flow channels normal to the die axis to be located at the interfaces of the adjacent plates and simplified machining, assembly and cleaning. Plate 1 is an adapter for the main extruder providing the SF RTP melt; it features a flanged end, and can be mounted on the extruder using a C-clamp, as shown schematically in Figure 4.17.

The SF RTP melt enters the die through the feed channel provided in the center of the plate. At the interface between plates 1 and 2, the SF RTP melt is distributed into three symmetrically located axial channels that carry the melt along the die. Plate 2 provides a feed channel for the unreinforced

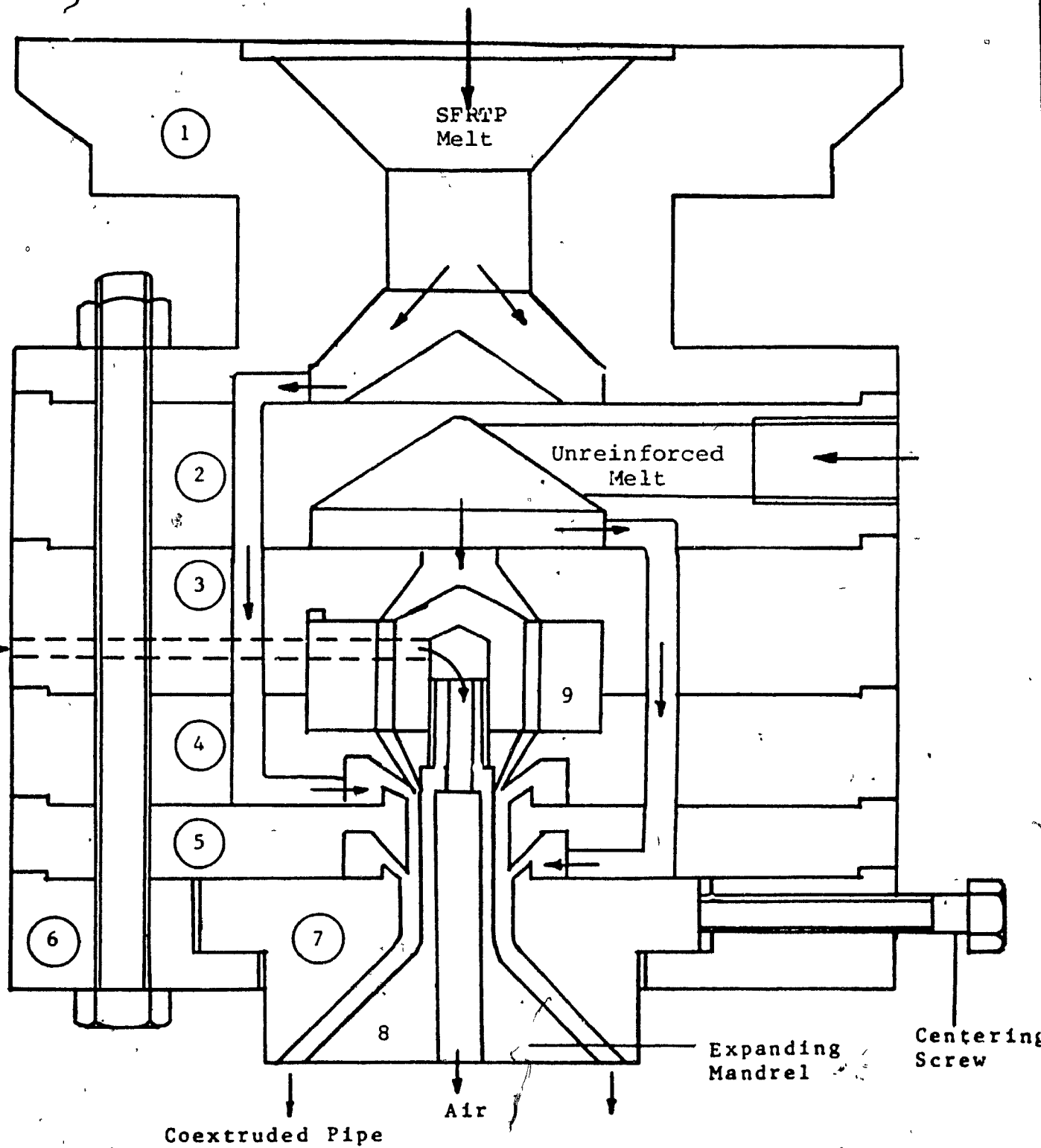


Figure 4.16 Coextrusion Pipe Die (Schematic).

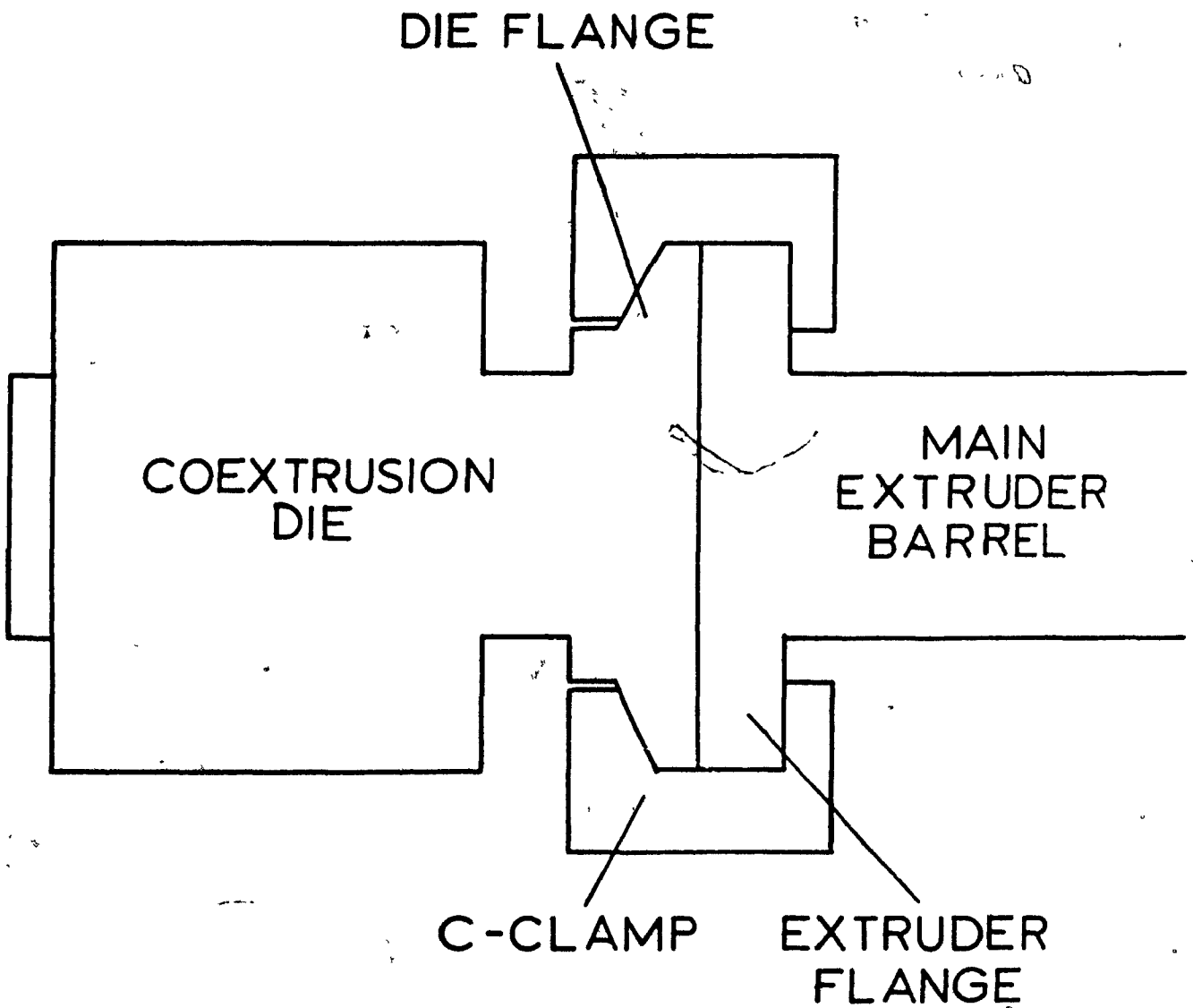


Figure 4.17 Coextrusion Die Mounted on the Main Extruder Using a C-Clamp.

melt from the coextruder. This melt is conveyed to the die by a flanged length of a steel tube fitted with heating elements (500 W, 240 V) and a thermocouple port, as shown schematically in Figure 4.18. For convenience of assembly, this melt conveying pipe is straight, and the coextruder is oriented perpendicular to the main extruder, as shown in Figure 4.14. The actual arrangement of the die and the two extruders is shown in Figure 4.19.

At the interface between plates 2 and 3, the unreinforced melt is distributed into a larger central channel and three smaller external channels located symmetrically with respect to the die axis. The central channel carries the melt for the inner unreinforced layer of the coextruded pipe, while the three external channels carry the melt for the outer unreinforced layer. Various channels carrying the SF RTP and the unreinforced melt for the three layers of the pipe continue through plates 3 and 4.

At the interface between plates 3 and 4, a cavity is provided for housing a spider construction 9. The spider construction, shown in Figure 4.20, provides support for the mandrel. It has three legs supporting the central hub. The unreinforced melt in the central channel flows around the legs, and the split streams subsequently recombine to form a continuous annular stream around the mandrel constituting the inner unreinforced layer of the pipe. An air channel is also provided

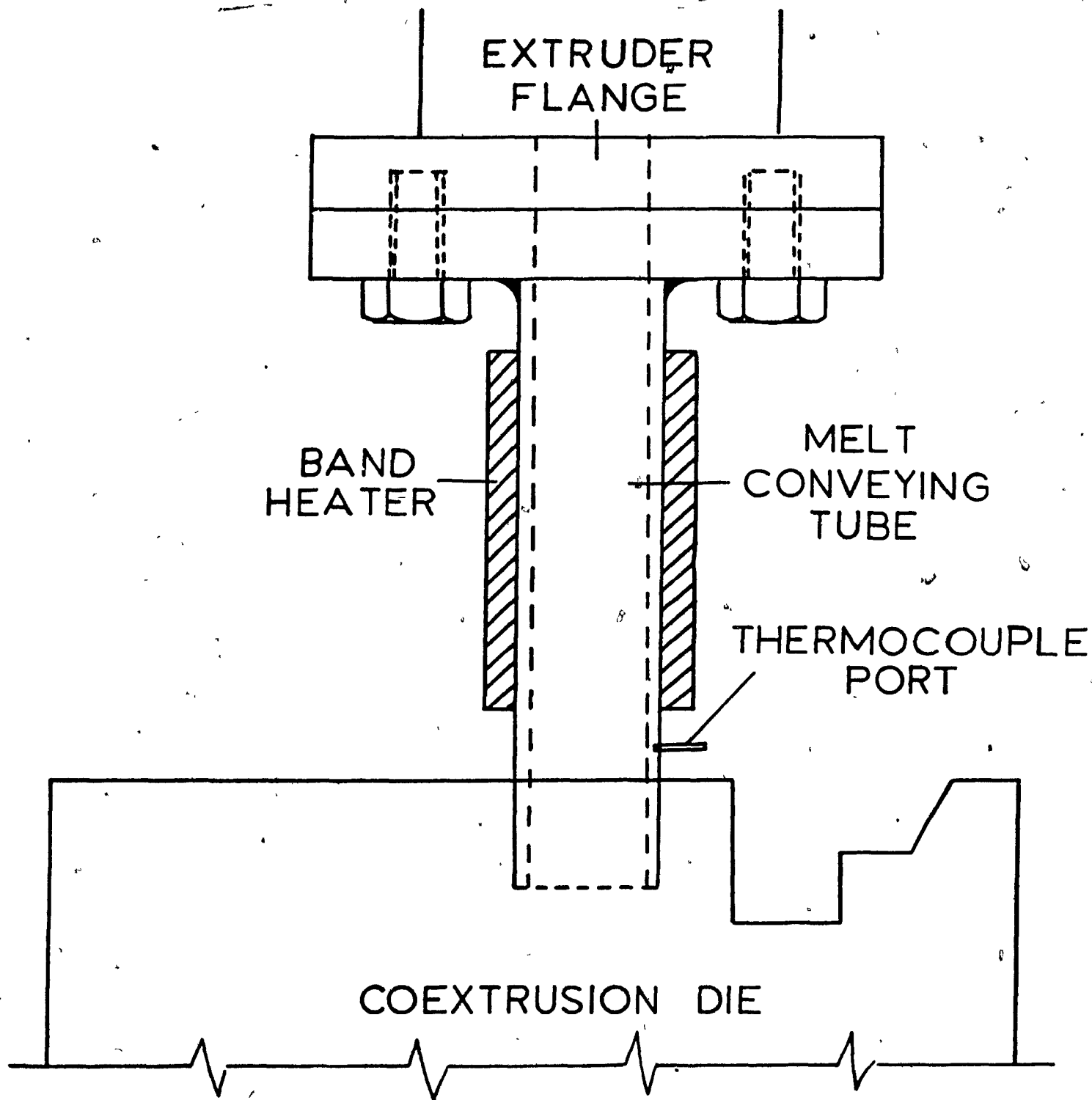


Figure 4.18 Tube for Conveying Unreinforced Melt from the Coextruder to the Die (Schematic).

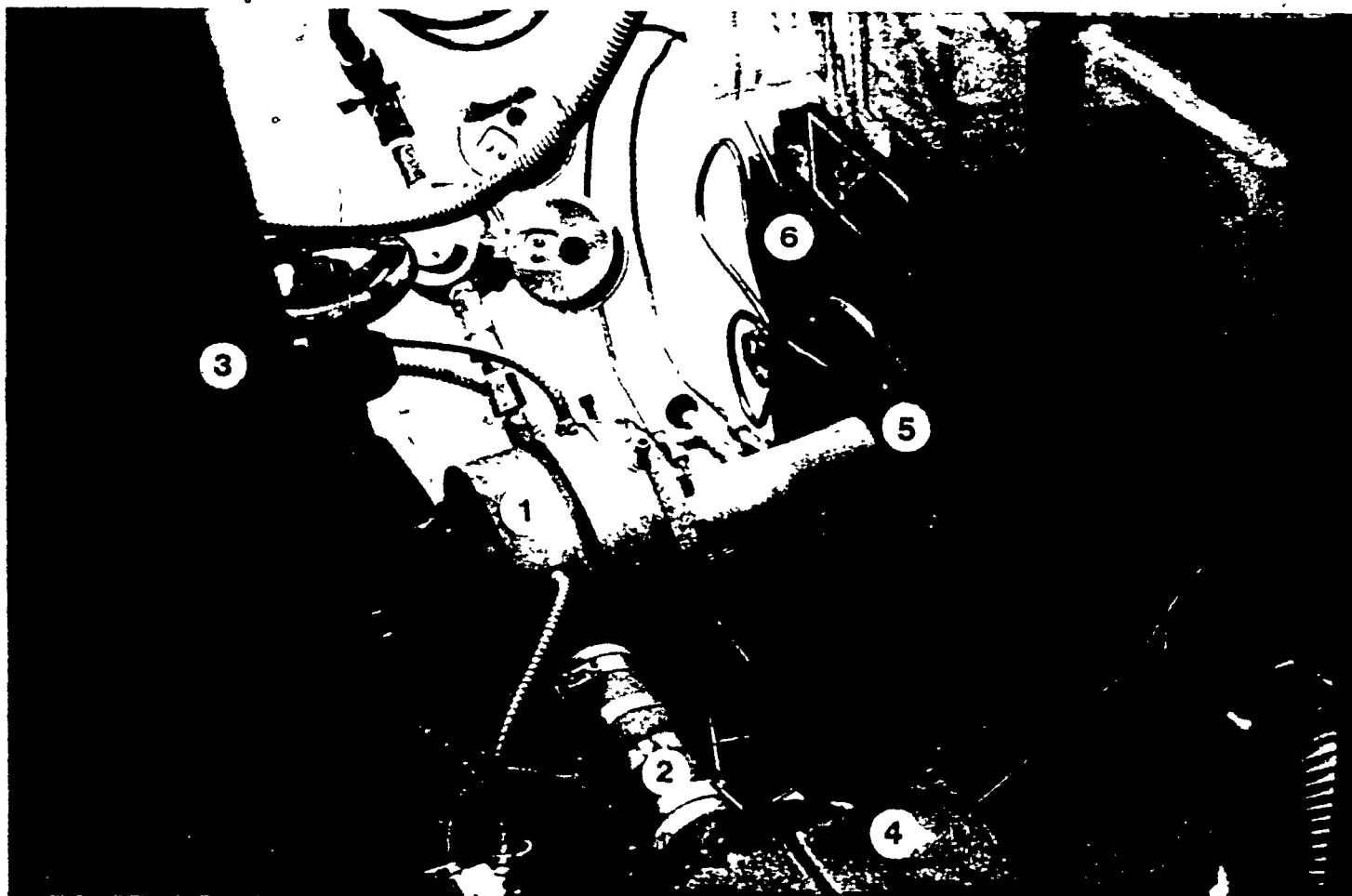


Figure 4.19 Assembly of Coextrusion Die and Two Extruders.

- (1) Coextrusion Die (2) Tube Conveying Melt from Coextruder to Die
(3) Main Extruder (4) Coextruder (5) Calibrator (6) Cooling Tank

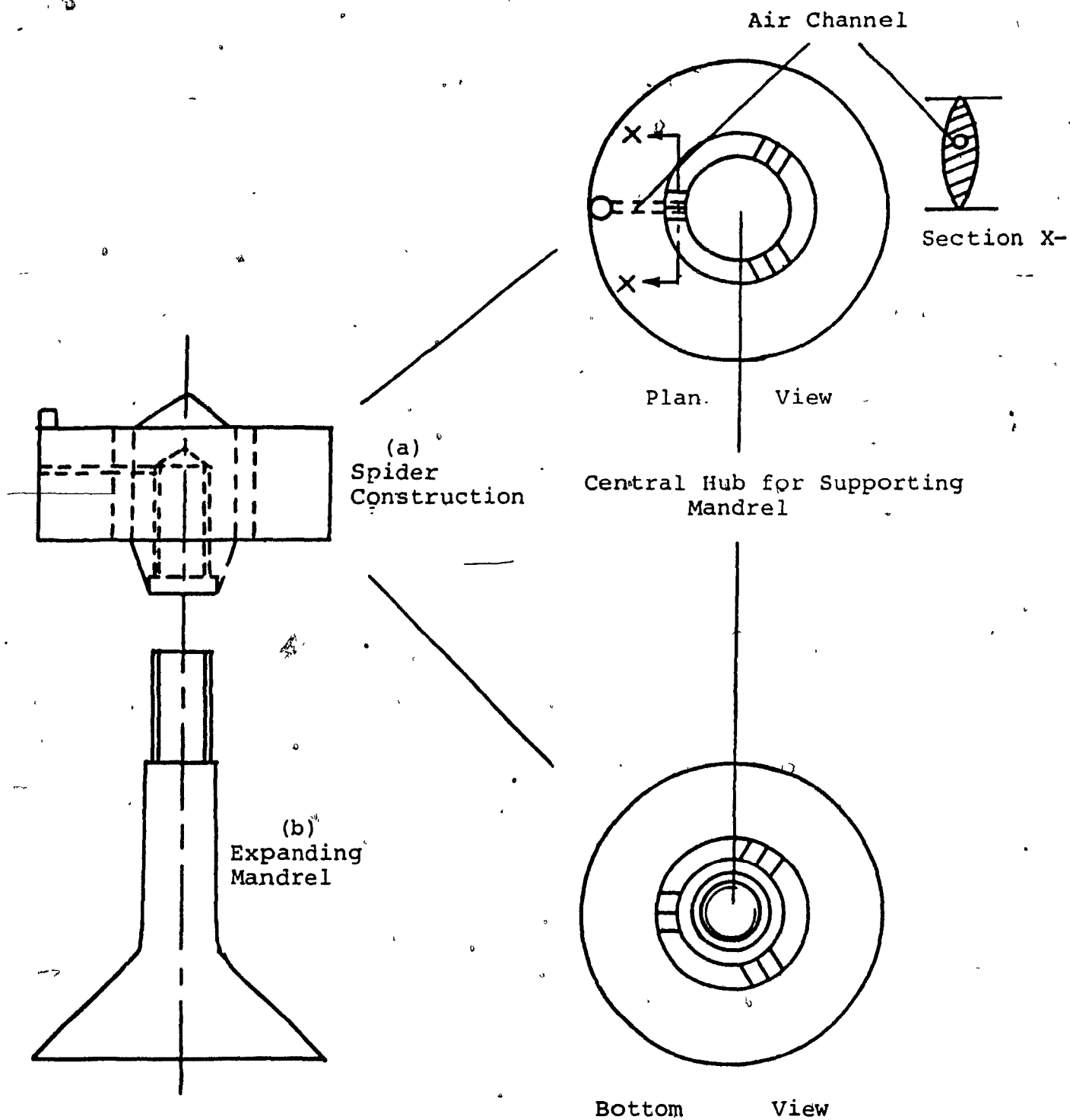


Figure 4.20 Spider Construction and Expanding Mandrel.

in plate 3 from the side of the die as shown in Figure 4.16. The air channel continues through one of the legs of the spider construction, and leads into an axial channel along the mandrel.

At the interface of plates 4 and 5, channels are provided to recombine the SF RTP melt streams from the three symmetrically placed external channels to form a continuous annular ring. This is shown in greater detail in the bottom view of plate 4, shown in Figure 4.21. The SF RTP melt is subsequently introduced over the inner unreinforced layer to form the middle SF RTP layer. A raised portion in the channel upstream of the mandrel allows some pressure build-up in the annular ring to promote uniformity of flow over the circumference.

Similarly, at the interface between plate 5 and the expanding section 7, channels are provided to bring the unreinforced melt streams from the three external channels, and recombine them to form a continuous annular ring. This melt is subsequently introduced over the middle SF RTP layer so as to form the outer unreinforced layer of the pipe. The three-layer melt thus formed is then subjected to flow through the expanding channel formed between the expanding section 7 and the expanding mandrel 8 before coming out of the die.

Since the three-layer melt is formed before entry into the expanding channel, the expanding channel itself plays the role of a die land. Thus, no cylindrical land region following the

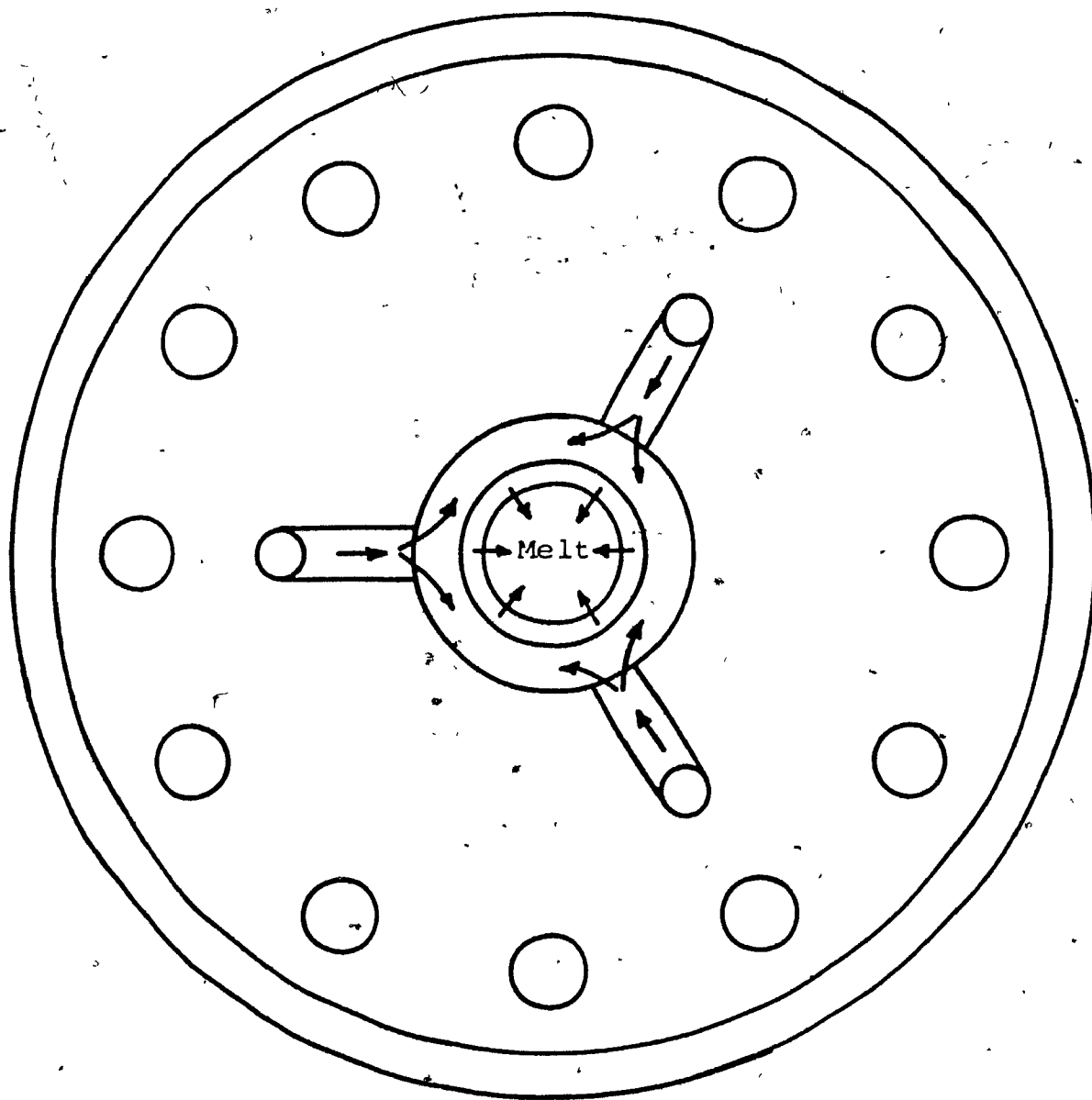


Figure 4.21 Bottom View of Plate 4 of the Coextrusion Die.

expanding channel is provided in this design.

The mandrel 8 is attached at its top to the spider construction 9 located between plates 3 and 4. It is provided with a central channel that prolongs the air channels in plate 3 and spider construction 9.

The expanding section 7 is supported by plate 6, as shown in Figure 4.16. Plate 6 is provided with six symmetrically placed centering screws to allow radial adjustment of the expanding section with respect to the mandrel without dismantling the entire die.

Plates 1 to 6 are held together by 11 symmetrically placed bolts on their outer periphery. The die is provided with a pressure transducer port in the neck region of plate 1 and two band heaters (240 V, 1200 W each). Two thermocouple ports are provided close to the melt flow channels, one in the neck region of plate 1 and the other near the die exit in the expanding section 7 for temperature control.

Clearly, splitting and recombining the various flow streams is unavoidable in this design. However, all the splitting and recombination zones are located away from the die exit so as to allow some flow length for the healing of the weld lines. Furthermore, the flow geometry is streamlined where possible, and flow restrictions are provided to allow pressure build-up in the

recombination zones, and hence promote flow uniformity. Coextruded pipe produced using this die showed no visible evidence of weld lines.

Initially it was thought that due to the complexity of the flow geometry and the differences in the pressure drops along the flow paths of the unreinforced melt for the inner and outer layers of pipe, unequal distribution might occur at the interface of plates 2 and 3 leading to unequal inner and outer layer thicknesses. However, it was subsequently found that the thicknesses of the inner and outer unreinforced layers were about the same under all the conditions of coextrusion used in this study. This is attributed to the fact that the major contribution to the pressure drops along the flow paths of the melt streams for both of these layers comes from the expanding portion of the channel. Because this contribution is about the same for both flow paths, the flow rates for the inner and outer unreinforced layers are nearly equal.

The extrudate from the die enters the calibrator located at the front end of the cooling tank, as shown in Figures 4.19 and 4.22. The pipe leaves the cooling tank through a rubber gasket at the rear end that provides a seal for the vacuum in the tank. The take-off system consisting of a haul-off and an automatic saw cutter is shown in Figure 4.23.

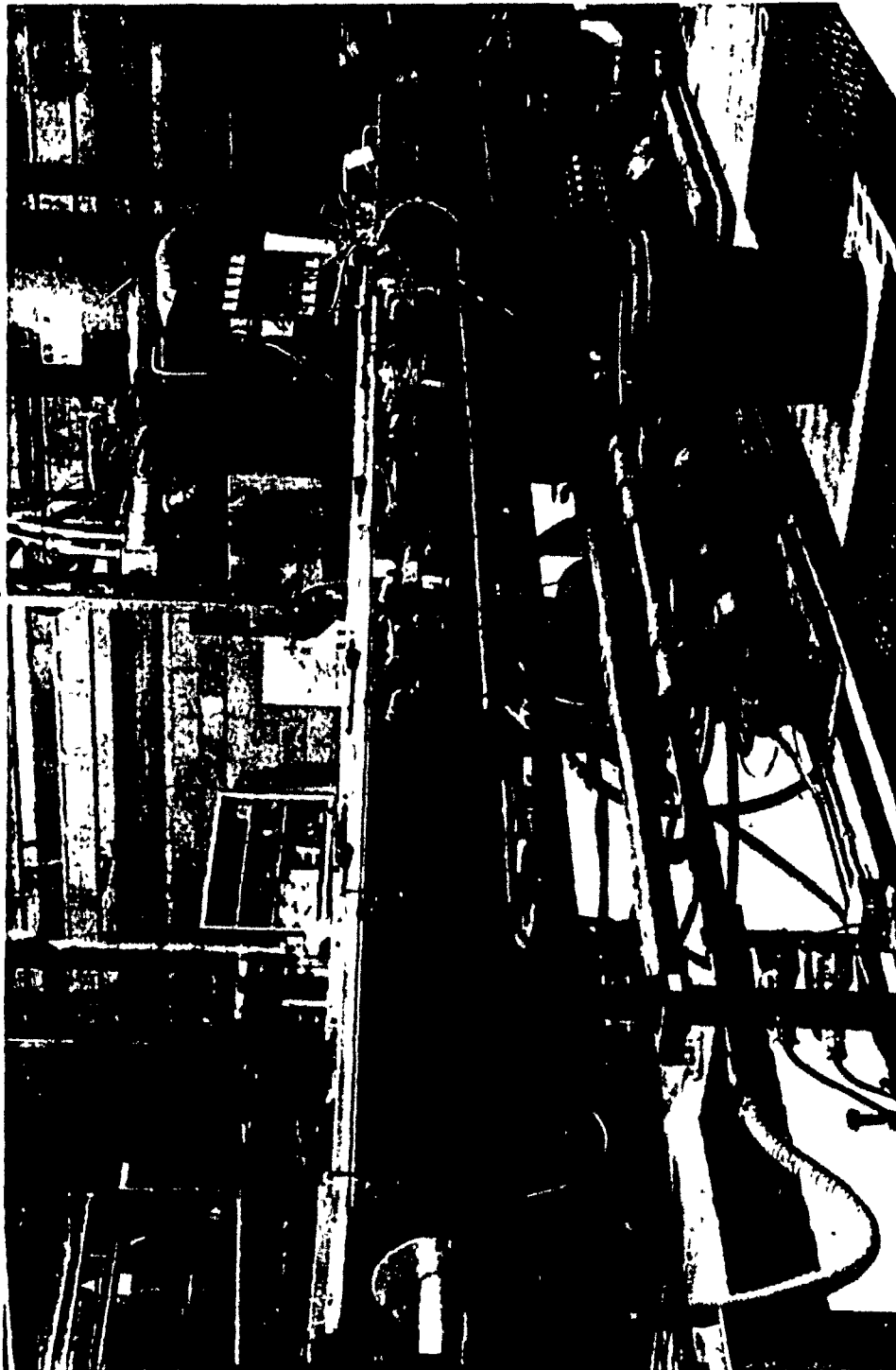


Figure 4.22 Pipe Sizing and Vacuum Cooling Tank.

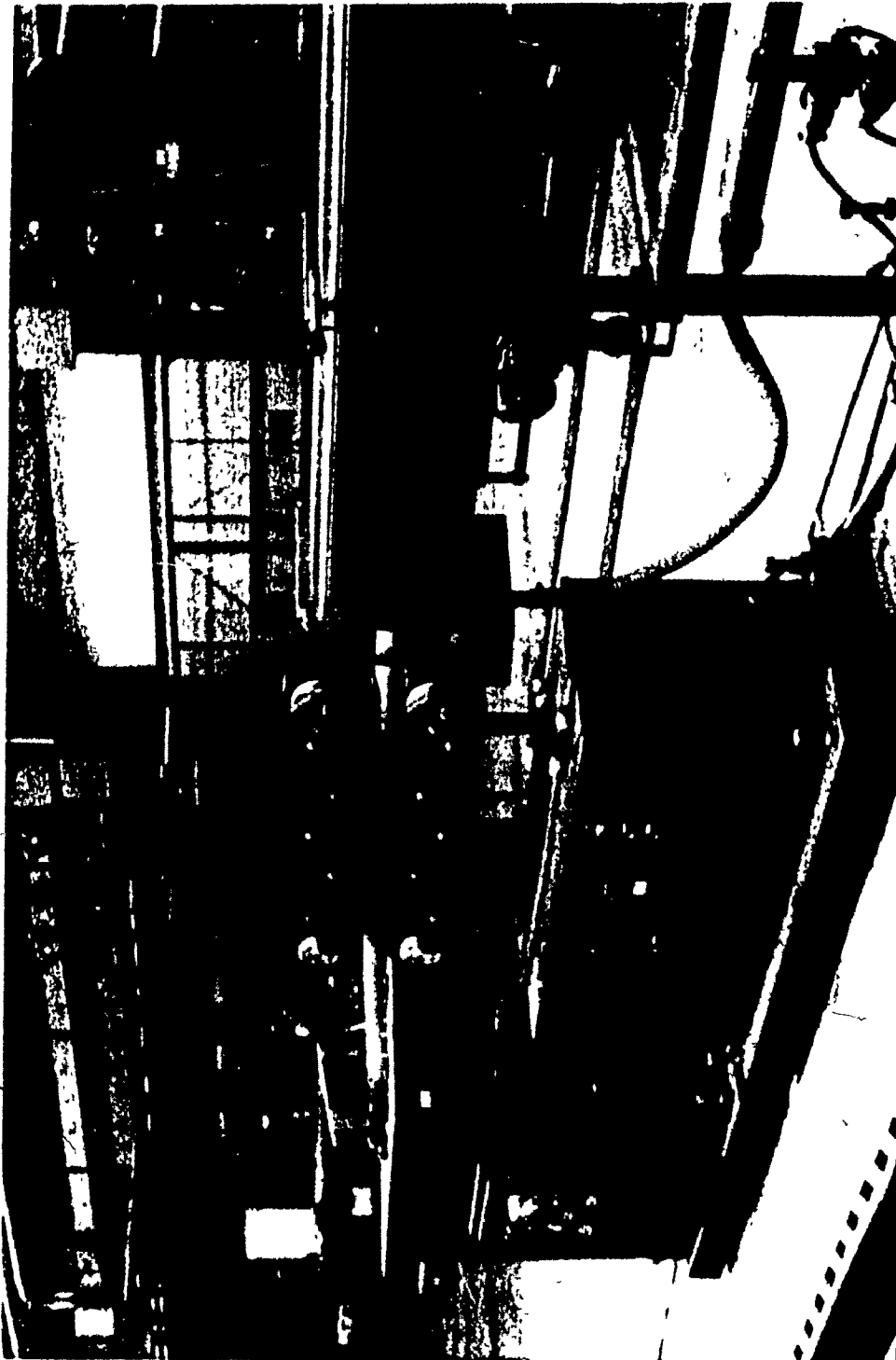


Figure 4.23 Caterpillar Haul - Off and Automatic Saw Cutter.

4.2.4 Coextrusion Experiments

Coextruded pipe was produced using both the low density (PE 74 A) and high density (PE 2906 M6) unreinforced resins for the surface layers, while a single SF RTP resin (PE 2906 M6 + 30 % glass fibers) was used for the middle layer in all cases. The coextrusion experiments were carried out at a melt temperature of about 200°C, with some variation of temperature along the extruder barrels and the die as required for a proper operation.

A brief account of the procedure used for start-up and for maintaining steady state conditions for the continuous manufacture of plastic pipe is presented in Appendix 4.3. It is very difficult to establish suitable conditions for continuous coextrusion, cooling and take-up. Instead, both the extruders were initially fed with the low density, unreinforced resin (PE 74 A). Since this resin is an extrusion grade, it was suitable for start-up and for establishing a synchronization of the operations necessary to produce the pipe continuously. The take-off speed was somewhat higher than the speed at which the extrudate emerged from the die in order to overcome friction at the calibrator, and this caused some axial stretching of the extrudate. Slightly warm water ($\approx 40^{\circ}\text{C}$) was used in the cooling tank rather than the usual cold water ($\approx 10^{\circ}\text{C}$), to prevent sudden chilling of the extrudate as it entered the tank. Reasonably good pipe of the low density unreinforced polymer (PE 74 A) was thus produced in a continuous manner. After establishing steady-state

conditions, the resin feed to the main extruder was switched to SF RTP (PE 2906 M6 + 30 % glass fibers) so as to produce coextruded pipe having inner and outer surface layers of the low density unreinforced resin (PE 74 A) and a middle layer of SF RTP. Only minor adjustments in the take-up speed and other process variables were then necessary to maintain steady state extrusion conditions.

Coextruded pipe was produced at two different SF RTP extrusion rates. It was found necessary to operate the coextruder at close to its maximum speed in order to supply the unreinforced melt against the pressure prevailing in the die. Hence there was little scope for varying its rate of extrusion. The overall coextrusion rate was determined by measuring the length of pipe extruded in a given interval of time at steady state. The conditions of extrusion for these experiments, referred to as conditions A and B for the two different SF RTP extrusion rates, are given in Table 4.3.

Once a reasonable length of pipe had been produced for both main extruder speeds, the low density unreinforced resin in the coextruder was replaced by the high density unreinforced resin (PE 2906 M6). Again only minor adjustments of parameters such as take-off speed and rate of air blowing allowed steady state conditions for pipe coextrusion to be maintained. Coextruded pipe with inner and outer surface layers of the unreinforced high density resin (PE 2906 M6) and the middle layer of SF RTP was

Table 4.3

Conditions of Coextrusion

(a) Pipe Made Under Condition A

Main Extruder		Coextruder		Die	
(SF RTP)		(PE 74 A)			
Barrel Zone	Temp. (°C)	Barrel Zone	Temp. (°C)	Zone	Temp. (°C)
I	188	I	185	I	200
II	192	II	195	II	200
III	198	III	200	III	200
IV	203				
V	205				

Screw RPM = 13.5

Screw RPM = 87.5

Head Pressure =

Head Pressure =

16.2 MPa (2350 psi)

20.7 MPa (3000 psi)

Melt Temperature = 198°C

Linear Rate of Extrusion = 1.96 m/min

(b) Pipe Made Under Condition B

Main Extruder		Coextruder		Die	
(SF RTP)		(PE 74 A)			
Barrel Zone	Temp. (°C)	Barrel Zone	Temp. (°C)	Zone	Temp. (°C)
I	192	I	168	I	200
II	196	II	195	II	200
III	198	III	198	III	200
IV	203				
V	205				

Screw RPM = 27

Screw RPM = 87.5

Head Pressure =

Head Pressure =

19.3 MPa (2800 psi)

20.7 MPa (3000 psi)

Melt Temperature = 198°C

Linear Rate of Extrusion = 2.97 m/min

(c) Pipe Made Under Condition C

Main Extruder		Coextruder		Die	
(SF RTP)		(PE 2906 M6)			
Barrel Zone	Temp. (°C)	Barrel Zone	Temp. (°C)	Zone	Temp. (°C)
I	195	I	185	I	200
II	200	II	190	II	200
III	203	III	195	III	200
IV	206				
V	210				

Screw RPM = 7.5

Screw RPM = 75

Head Pressure =

Head Pressure =

13.8 MPa (2000 psi)

19.3 MPa (2800 psi)

Melt Temperature = 198°C

Linear Rate of Extrusion = 1.59 m/min

(d) Pipe Made Under Condition D

Main Extruder		Coextruder		Die	
(SF RTP)		(PE 2906 M6)			
Barrel Zone	Temp. (°C)	Barrel Zone	Temp. (°C)	Zone	Temp. (°C)
I	195	I	168	I	200
II	200	II	195	II	200
III	203	III	198	III	200
IV	206				
V	210				

Screw RPM = 30

Screw RPM = 87.5

Head Pressure =

Head Pressure =

20.7 MPa (3000 psi)

20.7 MPa (3000 psi)

Melt Temperature = 197°C

Linear Rate of Extrusion = 3.28 m/min

(e) •SF RTP Pipe Made by Simple Extrusion

Extruder			Die	
(SF RTP)				
Barrel	Zone	Temp. (°C)	Zone	Temp. (°C)
	I	187	I	200
	II	192	II	200
	III	198	III	200
	IV	203	IV	200
	V	205		

Screw RPM = 15

Head Pressure = 12.5 Mpa (1800 psi)

Melt Temperature = 200°C

Linear Rate of Extrusion = 1.35 m/min

produced at two different SFRTTP extrusion rates using the same rate of extrusion for the unreinforced polymer. These conditions of extrusion, referred as conditions C and D, are given in Table 4.3.

The surface finish of the coextruded pipe produced using low density unreinforced resin (PE 74 A) for the surface layers was superior to that produced using the high density unreinforced resin (PE 2906 M6) for these layers. This is attributed to a significantly higher degree of crystallinity of the latter resin in the solid state, and thus a much higher degree of contraction upon solidification. The surface finish of the pipe made by simple extrusion of the high density unreinforced resin (PE 2906 M6) in some trial runs was also inferior to that made by simple extrusion of the low density unreinforced resin (PE 74 A) for the same reason. Under no conditions of coextrusion studied was any form of interfacial flow instability detected. The surface finish, however, did seem to improve with an increase in the overall rate of extrusion. This may be due to the fact that at higher extrusion rates, the interfacial shear rate is higher, and the difference in the viscosities of the adjacent layers is thus smaller, leading to a more uniform interface.

For comparison, SFRTTP pipe was also produced using the conventional simple extrusion pipe die shown schematically in Figure 4.24. The die gap and the outer diameter at the exit are about the same as for the expanding channel coextrusion die. This

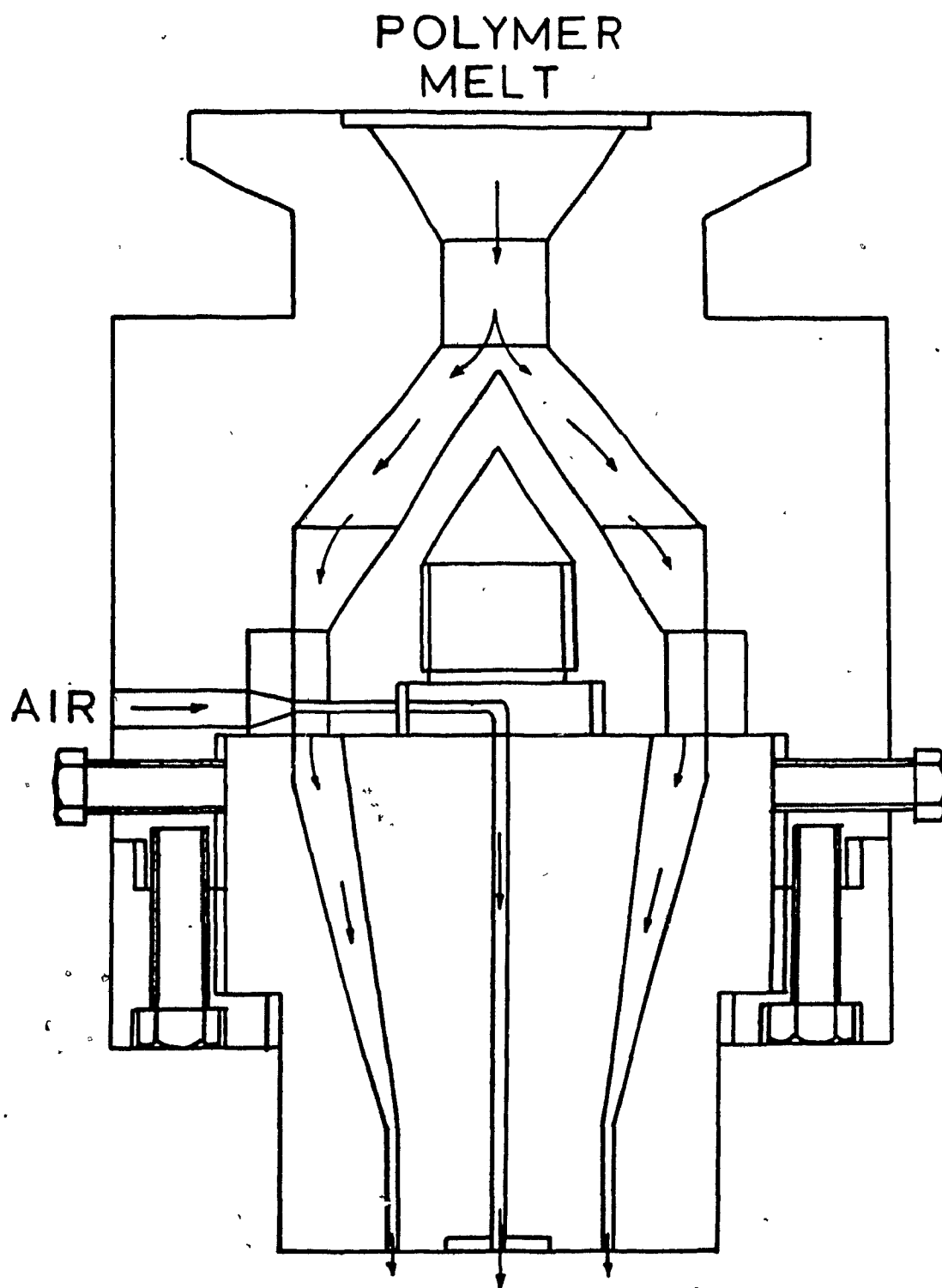


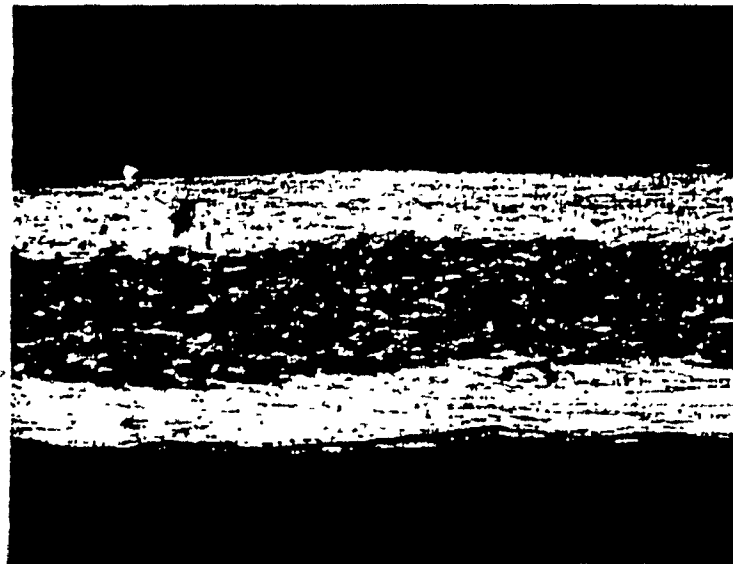
Figure 4.24 Conventional Pipe Extrusion Die Used for the Simple Extrusion of SFRTF Pipe (Schematic).

die was mounted on the larger extruder (screw diameter 63 mm), and the above-described calibration-cooling system was used to produce SF RTP pipe. The conditions of extrusion are given in Table 4.3. The extrusion rate had to be kept somewhat lower than the highest rate of extrusion used for coextrusion due to the lower melt strength of the SF RTP resin which tended to cause rupture of the extrudate.

4.2.5 Analysis of Flow Conditions for Coextrusion

The layer thickness distribution of coextruded pipe produced under various conditions was determined as follows. About 30 μ m thick slices were microtomed at various locations around the circumference in a plane normal to the pipe axis. The thicknesses of the individual layers in these slices were measured, using a low magnification transmission light microscope. Figure 4.25 shows a typical microtomed slice. It can be seen that the layers are separated by clearly defined interfaces, and that the thicknesses of the inner and outer unreinforced layers are about the same, indicating a fairly balanced distribution of the unreinforced melt between the central and external channels of the coextrusion die.

The overall thickness of the pipe, however, was somewhat nonuniform around the circumference, as shown schematically in Figure 4.26. This may be due to a slight misalignment of the mandrel with respect to the outer expanding section, or to a



- Outer Unreinforced Layer
- Middle SF RTP Layer
- Inner Unreinforced Layer

Figure 4.25 Optical Micrograph Showing Three Layers of Coextruded Pipe Wall (25x).

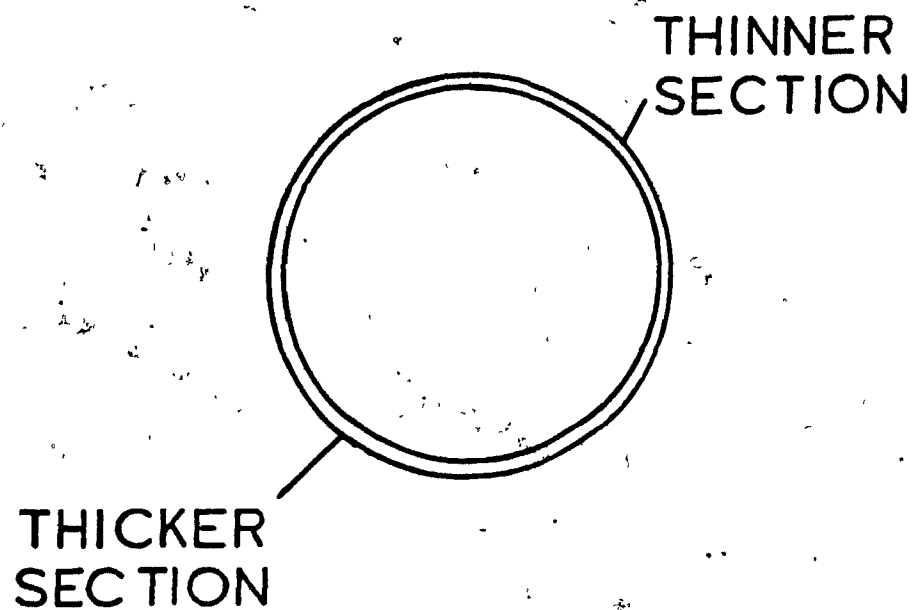


Figure 4.26 Non-Uniform Wall Thickness around the Circumference of Coextruded Pipe (Schematic).

pressure imbalance around the circumference leading to a higher flow rate in some regions. The pipe produced, however, was found to be satisfactory in other respects, viz., continuity of layers and uniformity of interfaces, and was therefore deemed suitable for further analysis.

Several measurements of the thicknesses of the individual layers were made around the circumference, and average values were calculated. The layer thickness distributions for the pipe produced under different conditions of coextrusion are given in Table 4.4a. Since the linear velocity of extrusion was also determined during coextrusion, the flow rate of individual layers could be determined as follows:

$$Q_i = \pi D t_i V \quad (4.21)$$

where D and V are the pipe diameter and extrusion velocity, and Q_i and t_i are the volumetric flow rate and thickness of a given layer.

The flow rates of the unreinforced and SF RTP melts for pipe made under different conditions of coextrusion are given in Table 4.4 a. Knowing the flow rates of the individual layers and the rheological characteristics of the melts, the previously developed theoretical analysis can be used to describe the velocity profile in the expanding channel of the die and to predict the fiber orientation distribution.

The power-law parameters for the SF RTP and the unreinforced

Table 4.4a

Flow Rates and Layer Thickness Distribution in Coextrusion

Coextrusion Condition	Layer Resin	Layer Thickness (mm)			Melt Flow Rate (cm ³ /s)	
		Inner	Middle	Outer	Unreinforced	SF RTP
A	PE 74 A/ SF RTP/ PE 74 A	0.35	0.95	0.29	3.16	4.69
B	PE 74 A/ SF RTP/ PE 74 A	0.19	1.0	0.21	3.05	7.56
C	PE 2906 M6/ SF RTP/ PE 2906 M6	0.38	0.56	0.30	2.75	2.27
D	PE 2906 M6/ SF RTP/ PE 2906 M6	0.18	1.14	0.2	3.14	9.47

Table 4.4b

Theoretically Predicted Interface Location, Interfacial Velocity and Viscosity Ratio at the Interface for Coextrusion

Coextrusion Condition	Interface Location (Y ₁ /H)		Fractional Interfacial Velocity (u _{zyl} /u _{zm})		Viscosity Ratio at Interface $\eta_{\text{SF RTP}}/\eta_{\text{Unrein.}}$	
	z=z ₀	z=z ₁	z=z ₀	z=z ₁	z=z ₀	z=z ₁
A	0.472	0.475	0.943	0.959	1.225	1.790
B	0.571	0.578	0.866	0.899	0.979	1.418
C	0.359	0.360	0.992	0.993	2.730	3.310
D	0.625	0.627	0.904	0.919	1.890	2.290

melts, determined by fitting the power-law equation to their viscosity-shear rate data at 200°C (Section 2.2), are given in Table 4.5. The velocity and shear rate profiles at the entrance to the expanding channel of the die, predicted according to the above analysis, are shown in Figure 4.27 for condition A.

It can be seen that reasonably good lubrication of the middle SFRTTP layer is achieved due to the highly shear thinning characteristics of the resins and the presence of the unfilled surface layers. The predicted interface locations at the entrance and exit for all the conditions of coextrusion are given in Table 4.4 b. The interface moves slightly outward towards the wall along the expanding channel in all cases. The interfacial velocities as fractions of the center line velocities (u_{zyl}/u_{zm}) are given in Table 4.4b. As mentioned earlier, these fractions indicate the degree of lubrication achieved as a result of coextrusion. As can be seen from their values at the entrance and exit, the degree of lubrication increases somewhat along the expanding channel.

The conditions of coextrusion A, B, C and D at the entrance to the expanding channel ($z=z_0$) are also indicated on the flow curves of the three resins in Figure 4.28. The ratios of viscosities of the two adjacent layers at the interface are given in Table 4.4 b. The viscosities were calculated using the interfacial shear rate and the power-law parameters of the resins. As can be seen, all the viscosity ratios, except that for

Table 4.5

Power-Law Parameters for the Resins Used

Temperature = 200°C

Resin	Consistency Index K (Pa-s ⁿ)	Power-Law Index n
SFRT (PE 2906 M6 + 30% Fibers)	2.37x10 ⁴	0.33
High Density Unreinforced (PE 2906 M6)	1.35x10 ⁴	0.38
Low Density Unreinforced (PE 74 A)	1.23x10 ⁴	0.43

Table 4.6

**Theoretically Predicted Average Angle of Orientation
w.r.t. Hoop Direction ($\bar{\psi}$)**

Coextruded Pipe Made Under Condition A

Location Across Channel (y/H)	$\bar{\psi}$
0 (Center Line)	19°
0.25 (Near-Center)	21°
y ₁ /H (Interface)	38°

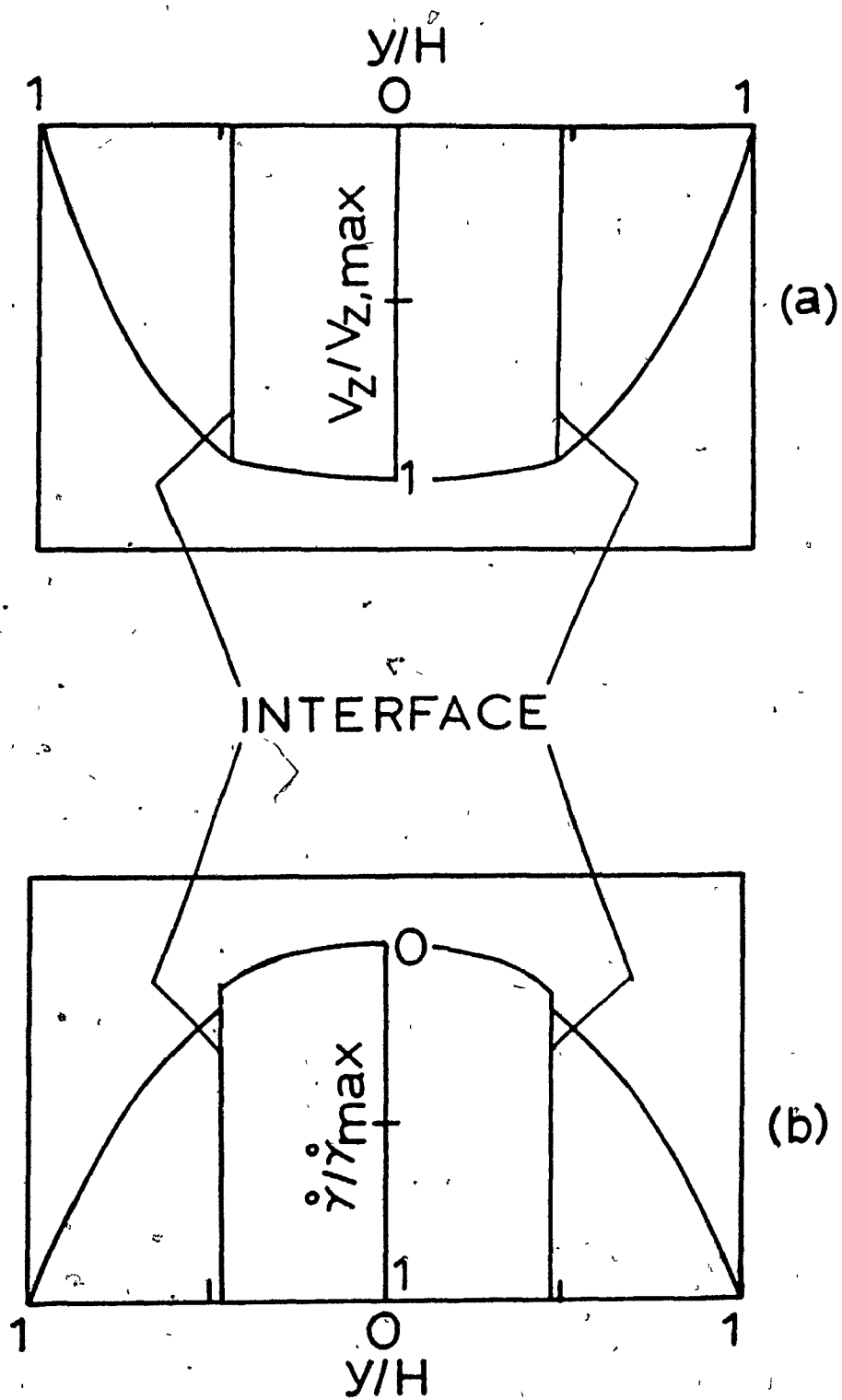


Figure 4.27 (a) Velocity Profile and (b) Shear Rate Profile at the Entrance to the Expanding Channel of the Coextrusion Die for Pipe Made Under Condition A.

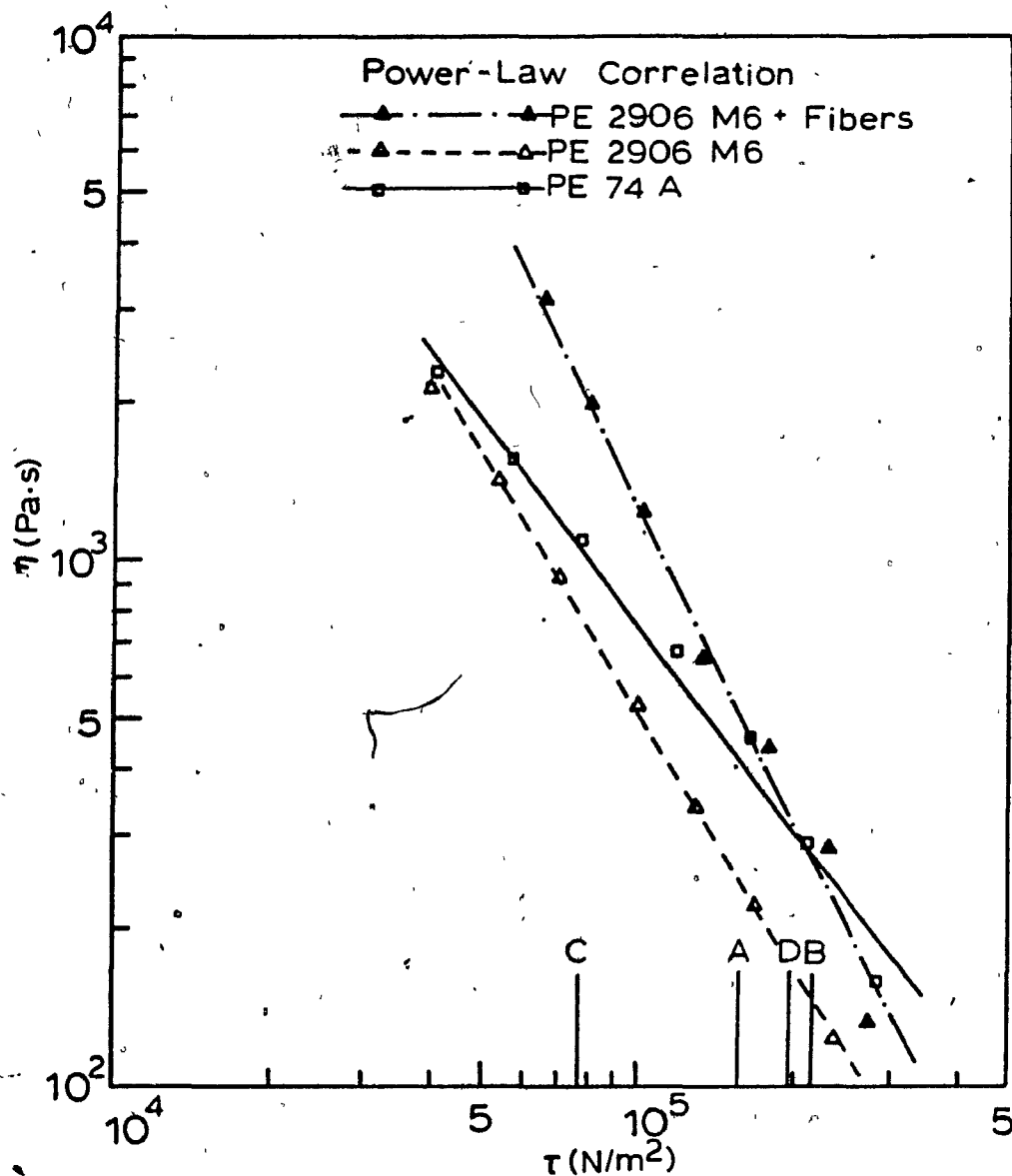


Figure 4.28 Conditions of Coextrusion at the Entrance to the Expanding Channel of the Die Predicted According to the Theoretical Analysis.

coextrusion condition B, are greater than unity implying lubrication of the middle SF RTP layer by the unreinforced surface layers. Also, the viscosity ratio increases toward the die exit in each case, indicating an increase in the degree of lubrication along the channel.

The theoretical analysis can also be used to predict the fiber orientation distribution at the exit for the above conditions of coextrusion. This was done for the coextruded pipe produced under condition A for the purpose of illustration. The fiber orientation distributions predicted at the center line ($y/H = 0$), near-center ($y/H = 0.25$) and at the interface ($y/H = y_1/H$) are presented in Figure 4.29. The average angle of fiber orientation with respect to the hoop direction was also calculated theoretically using the method described in Section 3.3.3. The results are presented in Table 4.6. It is seen that the degree of hoop orientation decreases from the center line towards the interface, the average angle of orientation with respect to the hoop direction changing from about 20° at the center line to about 40° near the interface.

4.2.6 Fiber Orientation in Coextruded Pipe

The fiber orientation pattern in the pipe produced by coextrusion through the expanding channel die, as well as by simple extrusion of SF RTP through the conventional die, was assessed by measuring the mechanical properties of test pieces

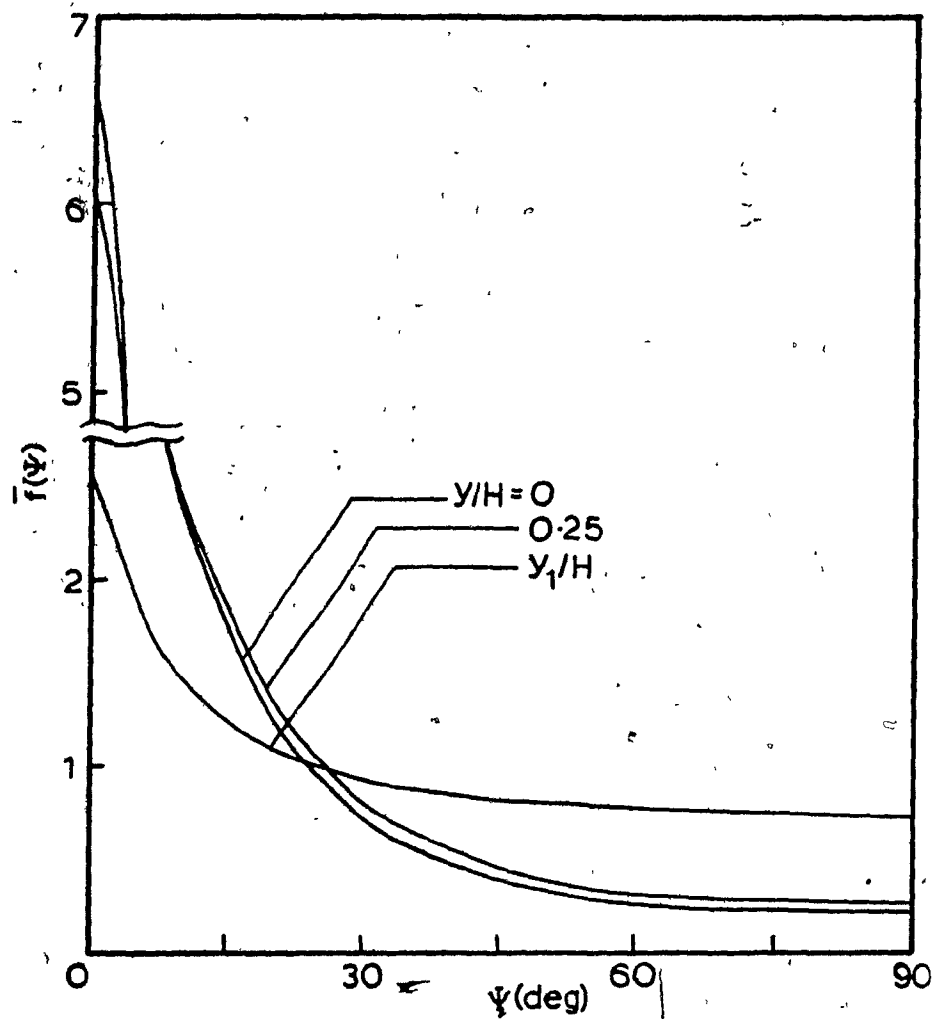


Figure 4.29 Predicted Fiber Orientation Distribution Function across the SF RTP Layer of Coextruded Pipe Produced under Condition A.

cut from the pipe samples in the axial and hoop directions. Measurement of the flexural stiffness of coextruded pipe in the two directions was not as relevant as in the case of simple extrusion extrudates, because the region of maximum strain in a flexural test is at the surface, which in the case of coextruded pipe consists of unreinforced layers. It was thus decided to measure the tensile strength in the two directions. Because of the absence of weld lines in the coextruded pipe, no problems were encountered in obtaining test pieces in either the axial or hoop directions. Strips were cut from the pipe in the two directions and were flattened at moderate temperature and under low force as described earlier (cf. Section 3.4.1). Test pieces were then cut from the flattened strips. The test piece geometry is shown schematically in Figure 4.30. Tensile tests were carried out on an Instron Universal Tester at a crosshead speed of 0.5 mm/min.

Tensile strength results are given in Table 4.7a for coextruded pipe produced under different conditions of coextrusion and in Table 4.7b for SF RTP pipe produced by simple extrusion. It can be seen that the anisotropy ratio (ratio of the tensile strength in the axial direction to that in the hoop direction) is close to 1 for all coextrusion conditions, and there does not seem to be any systematic effect of the relative flow rates of the SF RTP and unreinforced melts on these results in the range studied. On the other hand, for the SF RTP pipe produced by simple extrusion using a conventional die, the ratio

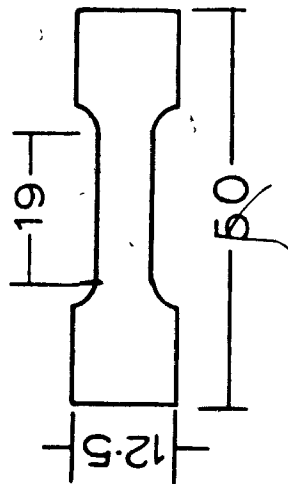


Figure 4.30 Tensile Test Piece Geometry
(Dimensions are in mm).

Table 4.7

Tensile Strength of Coextruded and SF RTP Pipe

Extrusion Condition	Layer Resin	Tensile Strength (MPa)		Anisotropy (A/H)
		Axial	Hoop	
(a) Coextruded Pipe				
A	PE 74 A/ SF RTP/ PE 74 A	24.7+2.7	21.5+0.7	1.2
B	PE 74 A/ SF RTP/ PE 74 A	26.9+1.3	24.5+0.8	1.1
C	PE 2906 M6/ SF RTP/ PE 2906 M6	33.1+1.5	25.9+0.7	1.3
D	PE 2906 M6/ SF RTP/ PE 2906 M6	26.4+1.9	27.7+1.5	0.9
(b) SF RTP Pipe made by Simple Extrusion				
E	SF RTP	38.3+1.5	16.5+0.7	2.3

is about 2.3. These results show clearly that while a strong axial orientation of fibers is present in pipe produced by simple extrusion, fiber orientation can be balanced between the axial and hoop directions in coextruded pipe.

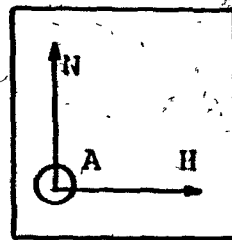
The fiber orientation pattern was also assessed by the previously described method of microtoming and transmission light microscopy (cf. Section 3.5.3). Figure 4.31 shows a typical fiber orientation pattern in the coextruded pipe. The axial and hoop directions with respect to the plane of the figure are also indicated in each case. These photographs show that there is only a small change in the orientation pattern from the interface to the center of the middle SF RTP layer. The overall orientation appears to be balanced with respect to the axial and hoop directions. Figure 4.32 shows the orientation pattern in the SF RTP pipe produced by simple extrusion through the conventional die. Clearly, there is a predominantly axial orientation of fibers in the pipe from the surface to the core region.

The above results demonstrate the ability of the expanding channel coextrusion die to promote hoop orientation of fibers throughout the thickness of the SF RTP layer. The degree of hoop orientation of fibers is significantly higher in the coextruded pipe than in the SF RTP pipe produced by simple extrusion through a conventional die. Several factors can inhibit or reduce the degree of hoop orientation of fibers in the coextruded pipe. One such factor is the interaction of a fiber with the

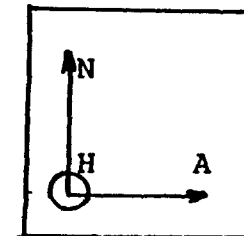
Inner Wall



Outer Wall

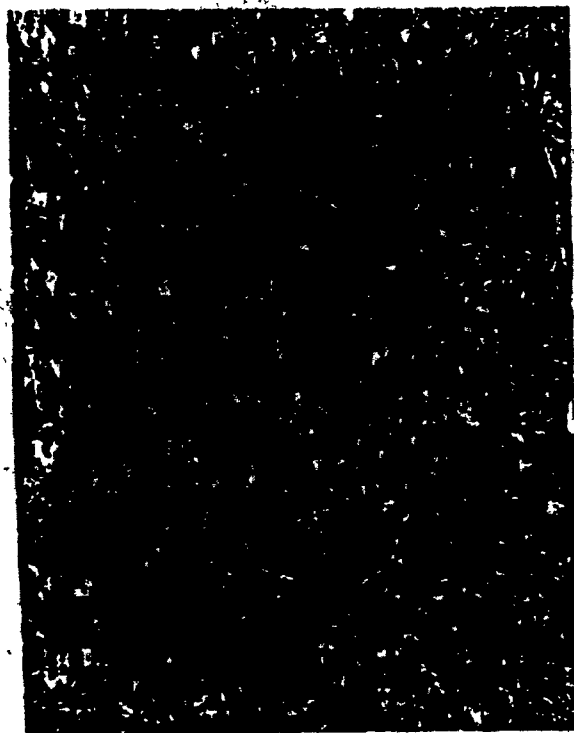


(a)



(b)

Figure 4.31a and b Optical Micrographs Showing Fiber Orientation in the SFRTTP Layer of Coextruded Pipe (40x).



Near Outer Interface



Core



Near Inner Interface

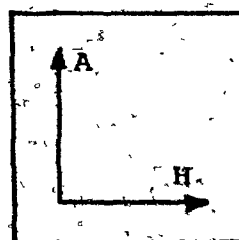


Figure 4.31c Optical Micrographs Showing Fiber Orientation in the SFRTTP Layer of Coextruded Pipe (40x).

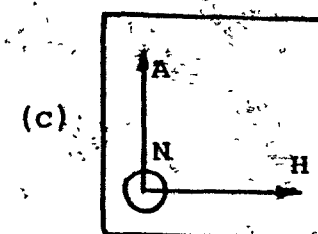
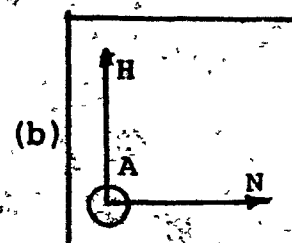
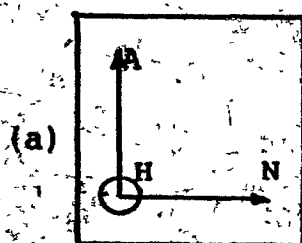
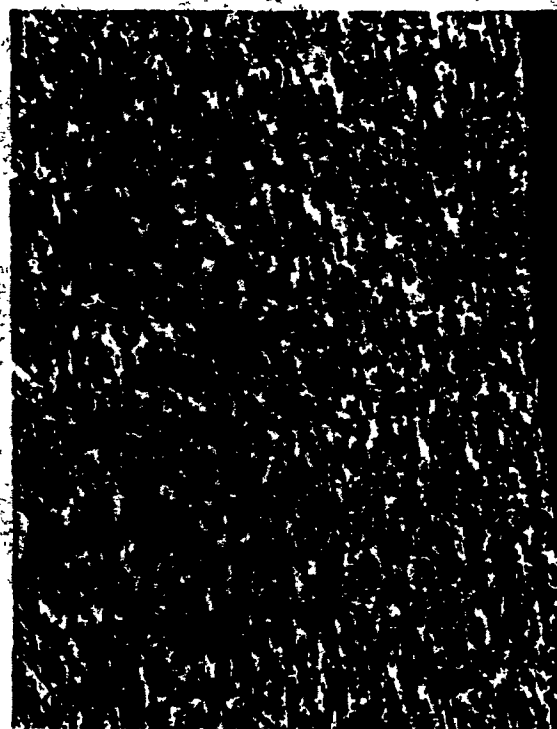
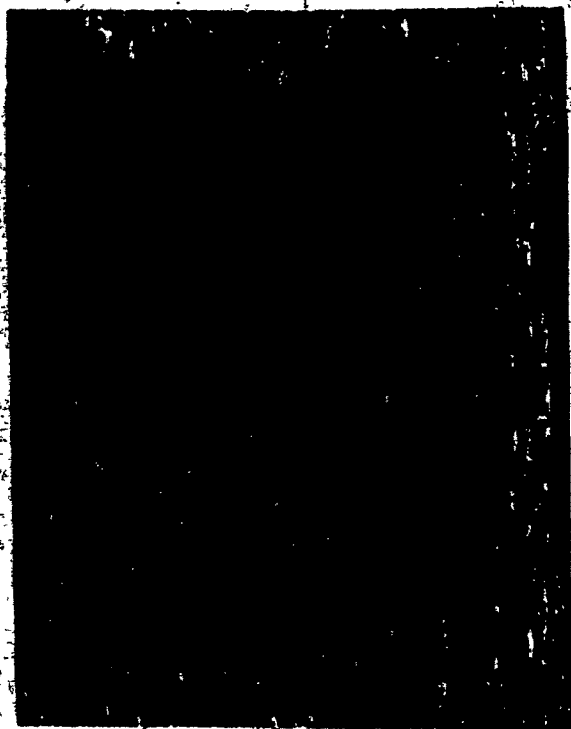


Figure 4.32 Optical Micrographs Showing Fiber Orientation in SFRTP Pipe Produced by Simple Extrusion Using a Conventional Pipe Die (40x).

surrounding fibers in a concentrated SFRTTP suspension. These interactions hinder the motion of the fiber in response to the flow field, and can in turn, reduce the degree of hoop orientation. Another important factor is the post extrusion stretching of the extrudate by the take-up system, as it leaves the die. Since the extrudate remains in the molten state for a certain length of travel through the cooling tank, it undergoes an axial extensional flow under the pulling force. This can cause a reorientation of fibers towards the axial direction destroying the degree of hoop orientation achieved in the die. All these factors can make it very difficult to achieve a predominantly hoop orientation of fibers in the pipe. However, the extent of hoop orientation can be controlled to a certain degree by the selection of resins with suitable rheological properties, of die geometry and of operating conditions, as discussed in Section 4.1.4. Moreover, as discussed in Chapter 5, considerations of the mechanical characteristics of coextruded pipe suggest that an orientation pattern giving rise to balanced properties in the axial and hoop directions may, in fact, be more desirable than preferential orientation in any one direction from the performance point of view.

Chapter 5

MECHANICAL PROPERTIES OF COEXTRUDED PIPE

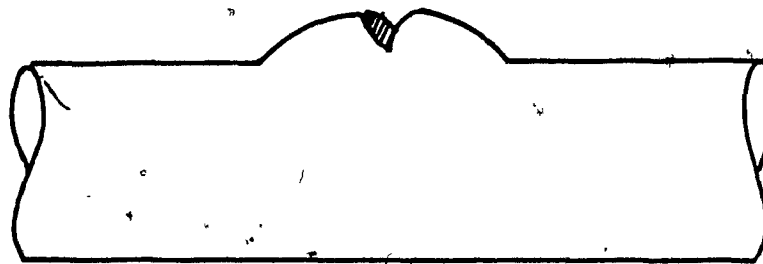
The use of plastic pipe for underground water and gas distribution lines is a long term application requiring safety and reliability. Considerable research effort has hence been devoted to the study of failure mechanisms in plastic pressure pipe (83-97). Metal piping systems have been traditionally designed for 50 years of service life, and equivalent service performance is normally expected of plastic piping systems (98). Key mechanical properties that affect pipe performance include stiffness, strength, toughness and ductility. While stiffness and strength are important for dimensional stability and ability to sustain imposed stresses, ductility and toughness are important for resistance to crack growth and impact. In order to establish standard design procedures and to ensure reliability and safety, the American Society for Testing and Materials (ASTM) provides several standard test methods (99). Some of these tests are described briefly in Appendix 5.1.

As suggested by some of these test methods, the design of a piping system and the estimation of its performance are often based on short term internal pressure testing. Although, plastic piping systems have shown a service performance that is comparable or superior to that of metallic piping systems (83), service failures have occurred, and the causes of some of these

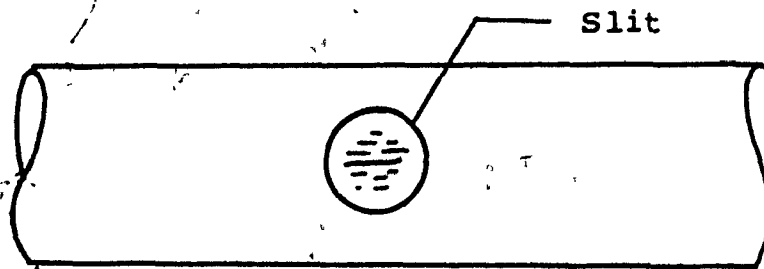
failures are peculiar to plastic material. Furthermore, the modes of a majority of field failures are distinctively different from those observed in the case of short-term laboratory tests.

5.1 Failure Modes in Plastic Pipe

Failures in plastic pipe (particularly polyethylene pipe used for gas transportation) have been found to occur in several distinct modes. Short-term laboratory tests involving internal pressurization invariably lead to a ductile failure involving localized macroscopic yielding, as illustrated in Figure 5.1.a. Such a failure, described as ballooning (84), usually occurs when stresses due to internal pressure exceed the yield strength. This type of failure, however, has rarely been observed in service. A service failure usually results from a combination of several factors such as defects, third party damage, poor installation, and imposed long term internal or external stresses. A common mode of failure features a localized crack, with little or no macroscopic deformation in the vicinity of the fracture, leading to the formation of a slit, as shown in Figure 5.1b. Such a failure occurs at an overall stress far below the yield strength of the material, and exhibits the characteristics of a brittle fracture. Failure can also occur in a brittle mode due to a crack running at high speed along the length of the pipe over a considerable distance (Figure 5.1c) (87, 100). Such a mode of failure, which is due to rapid crack propagation (RCP), has been found to occur in metal and rigid PVC pipe, but is not common in



(a) Ballooning in Plastic Pipe
(Ductile Failure)



(b) Slit Failure in Plastic Pipe
(Brittle Failure)



(c) Rapid Crack Propagation (RCP) in Plastic Pipe
(Brittle Failure)

Figure 5.1 Failure Modes in Plastic Pipe.

polyethylene piping systems (100).

Microscopic examination of several slit-type field failures and analysis of the conditions leading to these failures indicate that they result from the initiation of a crack at a defect or flaw and its subsequent propagation through the pipe wall under sustained stresses over a long period of time. The defect may originate during pipe extrusion (void or inclusion) or it may result from external factors such as rock impingement, excessive bending due to poor installation, mishandling during transportation, installation or repair and earth movement. Poorly made pipe joints are also potential sites for crack initiation (85,86). While defects or flaws such as voids and inclusions may be located anywhere across the pipe wall thickness, those due to third party damage are usually located at the surface where the bending stresses, for example, may also be highest. Fracture mechanics concepts suggest that such flaws are obvious sites for stress concentration leading to very high stresses in the vicinity of defects even at relatively low overall stresses. This localized stress intensification has been suggested as the cause of brittle failure in plastic pipe (85,86). Consequently, significant research effort has been directed towards the application of fracture mechanics principles to the brittle failure of plastic pipe (84,90-97). Several tests have also been proposed to characterize the resistance of piping materials to crack initiation and propagation, and to correlate test results with the occurrence of field failures (100-105). Some of these

tests are briefly described in Appendix 5.2.

5.2 Brittle Failure in Plastic Pipe and the Coextrusion Process

5.2.1 Resin Selection for Improved Performance of Plastic Pipe

As discussed above, brittle failure in plastic pipe due to crack initiation and slow crack growth has been identified as the most important mode of field failure. Consequently, resins used for making plastic pipe should be selected not only for their stiffness and strength, but also for their fracture toughness or resistance to crack initiation and propagation. In general, the properties of PE resins depend on molecular weight distribution and molecular structure which, in turn, determine crystallinity, density and mechanical properties. While high density grades ($\rho \geq 0.96 \text{ gm/cm}^3$) are usually stiffer and stronger, they tend to have poor fracture toughness. Low density resins ($\rho \leq 0.92 \text{ gm/cm}^3$), on the other hand, offer excellent resistance to crack growth, but lack the stiffness and strength needed for pipe. A compromise is therefore unavoidable, and most pipe grade resins usually fall in the medium density range ($0.92 \text{ gm/cm}^3 \leq \rho \leq 0.96 \text{ gm/cm}^3$) (97,106,107). Attempts have been made to improve the resistance to brittle failure by raising the molecular weight, adjusting the molecular weight distribution, and incorporating comonomers (108-111). The scope for such adjustments is, however, limited as they tend to adversely affect the flow properties of the resin (108).

5.2.2 Possible Advantages Offered by the Coextrusion Process

In the proposed coextruded SFRTTP pipe, the wall has a sandwich structure, the inner and outer surface layers are of unreinforced polymer and the middle layer contains reinforcing fibers, as shown in Figure 1.2. Overall pipe properties are obviously determined by the properties of the individual layers. The presence of reinforcing fibers in the middle layer, and the control of their orientation allows significant improvements in pipe stiffness and strength to be achieved. Consequently, the inner and outer surface layers can be chosen to be of a resin that need not possess very good stiffness and strength, but has excellent fracture toughness. In this way, the surface layers can provide a superior resistance to the propagation of cracks originating at the surface, in addition to providing a smoother surface and assisting in the control of fiber orientation during extrusion as discussed in Chapters 3 and 4. The three-layer configuration can thus provide a distribution of properties across the wall thickness with a concentration of desired properties at appropriate locations. The resin for the inner and outer unreinforced surface layers can also be selected for several other desirable characteristics such as abrasion resistance, resistance to the soil components, lower permeability etc. The coextruded SFRTTP pipe thus allows a novel approach to the optimization of pipe properties.

5.3 Mechanical Properties of the Coextruded SFRTTP Pipe

As discussed above, the coextrusion process offers an opportunity to improve the overall stiffness, strength, and resistance to crack growth through the proper choice of materials. The following exploratory experimental study was carried out to demonstrate the principle of achieving such improvements in properties.

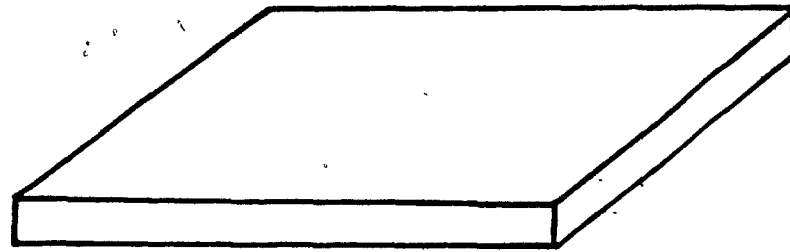
The actual pipe made by coextrusion during the course of this study (cf. Chapter 4) was not suitable for the intended mechanical testing due to its thin wall and the curvature of test pieces cut from it. Hence, a special technique was developed to make suitable flat, single-layer and multi-layer plaques. Test pieces cut from these plaques were used to measure the mechanical properties of interest. The measured properties cannot be considered to be the actual properties of coextruded pipe, but are thought to be qualitatively similar. Also, the materials used in this study are not pipe grade resins formulated expressly for pipe applications. Consequently, a direct comparison of the measured properties with those of commercial pipe resins would not be appropriate. The results reported in this chapter are intended primarily to illustrate the principle of achieving improvement in properties via a layered structure, and thus serve the purpose of highlighting the property advantages associated with the coextrusion process.

It should be stressed that the macroscopic inhomogeneous nature of the multi-layered structure of the test pieces often makes it impossible to follow standard test procedures, and to apply the associated test validity criteria. Hence the scope of this study is primarily limited to a relative comparison of the results among themselves.

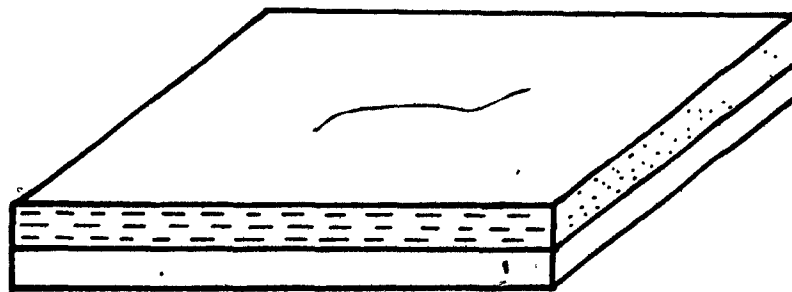
5.3.1 Objectives of the Study

This study concentrated on the three important properties, stiffness, strength and resistance to crack growth (initiation and propagation) in single and multi-layer test pieces. The specific objectives were as follows.

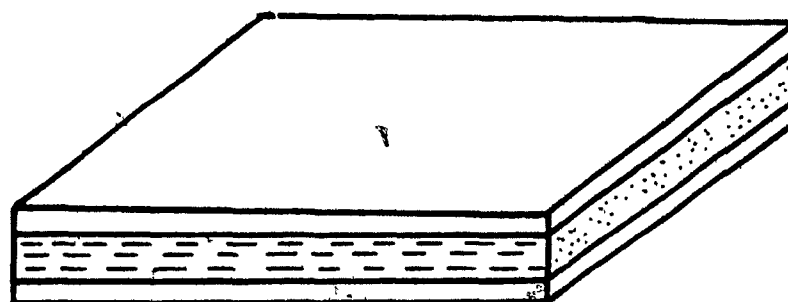
- 1) To prepare either single-layer or multi-layer (two or three layers) flat plaques, as shown schematically in Figure 5.2. In the plaques containing a SF RTP layer, it was also desirable to have a preferential orientation of the fibers in a given direction.
- 2) To measure the flexural stiffness and tensile strength of the test pieces obtained from these plaques as functions of the layer thickness distribution and fiber orientation in the SF RTP layer.
- 3) To characterize the crack initiation resistance of the individual materials according to post-yield fracture mechanics concepts (the J integral). Also to study the



(a) Single Layer Plaque



(b) Two Layer Plaque



(c) Three Layer Plaque

Figure 5.2 Single and Multi-Layer Plaques for Mechanical Testing.

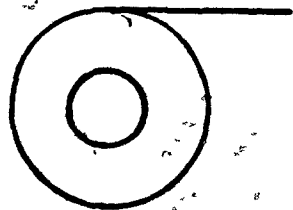
effect of the presence and orientation of fibers on the crack initiation resistance of SF RTP.

- 4) To characterize the crack propagation resistance of the individual materials according to the so-called R curve method, and to study the effect of the presence and orientation of fibers on the crack propagation resistance of SF RTP.
- 5) To carry out crack propagation studies in two-layer test pieces under conditions such that the crack extension started at the initiating notch in one layer, advanced to the interface, and subsequently into the next layer. To study the effect of the relative layer thicknesses and the elastic properties of the second layer on the crack propagation resistance of the first layer.

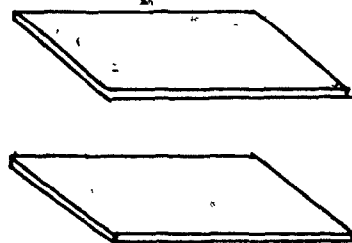
5.3.2 Method of Plaque Preparation

The flat single-layer or multi-layer plaques were prepared in two stages. First, thin strips of the individual materials were produced by extrusion. Pieces were then cut from these strips, stacked and compression molded to obtain the desired plaques, as shown schematically in Figure 5.3.

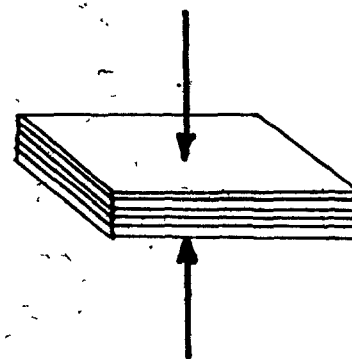
About 75 mm (3 in) wide and 1 mm thick strips of the individual materials were extruded using a Brabender Plasticorder extruder equipped with a laboratory-scale sheet die. The extruder specifications and the design of the die are given in Appendix



Roll of Extruded Sheet



Pieces Cut from the Roll



Compression Molding of Stacked Pieces



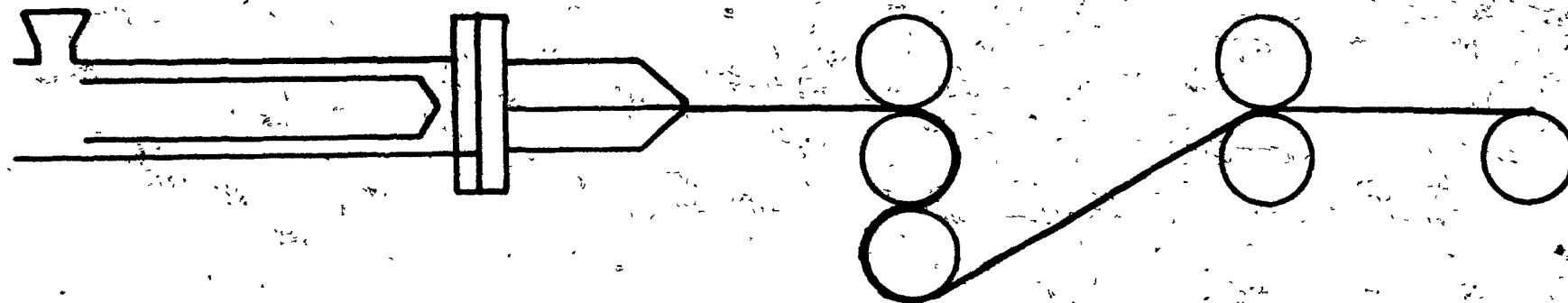
Molded Plaque

Figure 5.3 Compression Molding Procedure

5.3. The extrusion setup included chilling and take-up rolls as shown in Figure 5.4. The extrusion conditions for different materials are listed in Table 5.1. The die produced a preferential orientation of fibers in the extrusion direction of the SF RTP sheet.

A special compression mold was constructed to prepare plaques from the pieces cut from the extruded strips. This mold is shown in Figure 5.5. It is made of aluminum for good heat transfer, and is provided with two, 300-Watt cartridge heaters. The rectangular molding cavity has dimensions of 100 mm x 75 mm (4 in x 3 in), and is provided with a tight fitting piston for applying positive pressure to the material in the cavity. Plaques up to about 10 mm (0.4 in) in thickness can be prepared using the mold.

A typical molding operation consisted of cutting the appropriate number of pieces from extruded strips to dimensions equal to those of the molding cavity, and stacking them in it. While for a single-layer plaque all the pieces were of the same material, for a multi-layer plaque they were arranged in accordance with the desired order and layer thickness distribution. The mold was closed and mounted in a laboratory press with plates equipped for heating and cooling. The mold was heated to a temperature of $160 \pm 5^\circ\text{C}$, and a molding pressure of about 12 MPa was applied. Due to the tight fitting piston, little leakage flow occurred during compression. The mold was



184
Extruder

Sheet Die

Chilling
Rolls

Pulling
Rolls

Take-up
Spool

Figure 5.4 Strip Extrusion Set-Up.

Table 5.1
Conditions for Strip Extrusion

	High Density Unreinforced (PE 2906 M6)	SFRTF (PE 2906 M6 + 30% Fibers)	Low Density Unreinforced (PE 74 A)
Barrel Temp. (°C)			
Zone I	120	170	120
Zone II	130	180	130
Zone III	140	190	140
Die Temp. (°C)	150	200	150
Screw RPM	90	28	90
Die Head Pressure (psi)	700	900	750
Driving Torque (N.m)	65	25	70

25 mm

Mold
Cavity

Piston

Heater

Mold Cavity

Figure 5.5 Compression Mold for Plaques.

subsequently cooled under pressure to obtain the plaque. Because of the negligible flow involved in the process, the pre-existing fiber orientation in the extrusion direction of the strips of SF RTP was retained in the corresponding layer of the plaque. Also, the individual layers in a multi-layer plaque were separated by well defined, uniform interfaces throughout the plaque except very close to the edges.

5.3.3 Flexural Stiffness and Tensile Strength of Multi-Layer Test Pieces

5.3.3.1 Experimental Procedure

Three-layer plaques simulating the wall of coextruded pipe were prepared with two surface layers of unreinforced high density PE resin (PE 2906 M6) and a middle layer of SF RTP (PE 2906 M6 + 30% fibers) with a preferential fiber orientation along the length of the plaque. The plaques were prepared with several layer thickness distributions such that the fractional thickness (f) of the SF RTP layer varied from 0 to 1, $f = 0$ corresponding to a single-layer plaque of unreinforced PE and $f = 1$ corresponding to a single-layer plaque of SF RTP. The total plaque thickness was in the range of 2.4 to 4.2 mm depending on the layer thickness distribution.

Rectangular test pieces of dimensions 75 x 12.5 mm (3 x 0.5 in) were machined from the molded plaques either in the direction

of preferential fiber orientation (referred to as SF RTP-L) or in the direction normal to it (referred to as SF RTP-T), as shown schematically in Figure 5.6. The flexural stiffness was determined by the conventional three point bending method (112). Tests were carried out on the Instron Universal Tester (Model 1123) using a span of 50 mm (2 in) and a crosshead speed of 0.5 mm/min.

Because of the non-destructive nature of the three point bending test, the test pieces could be reused to measure the tensile strength. For this purpose, test pieces were machined to the dumbbell-like shape shown in Figure 5.7 which is similar to that described in the standard ASTM D638 procedure (113). The tensile strength was determined using the Instron Universal Tester (Model 1123) at a crosshead speed of 0.5 mm/min. Typical force-displacement records are shown schematically in Figure 5.8.

The tests were carried out for the test pieces taken in both L and T directions for each layer thickness distribution considered, and at least four test pieces were used for each case.

5.3.3.2 Results and Discussion

The flexural stiffness as a function of the fractional thickness f of the SF RTP layer is shown in Figures 5.9a and b for

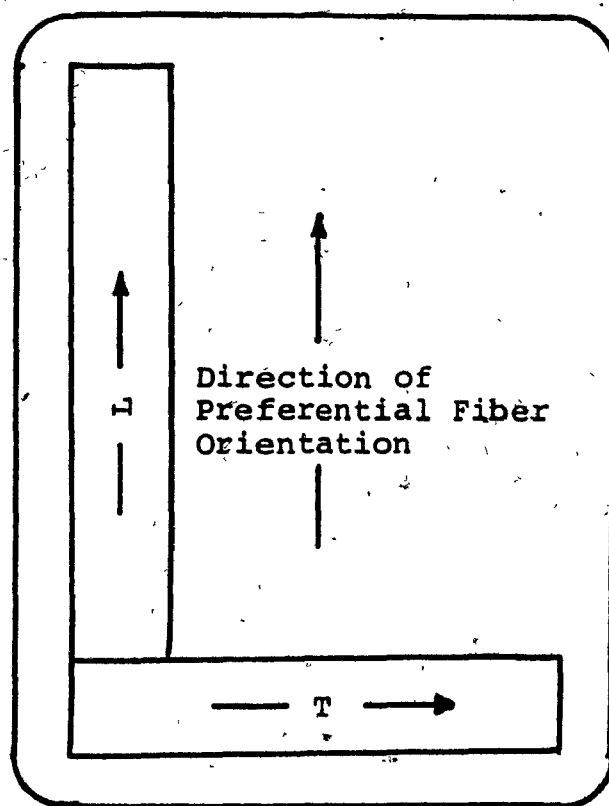


Figure 5.6 L and T Directions of the Compression Molded Plaque

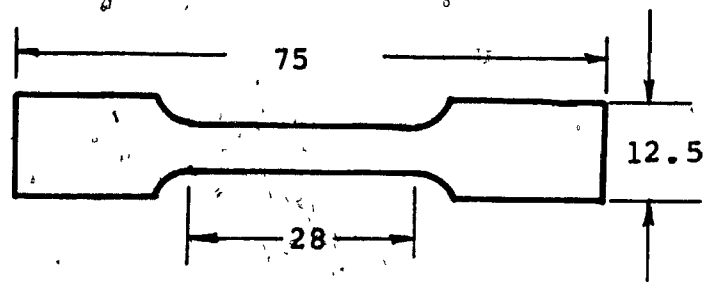


Figure 5.7 Tensile Test Piece Geometry.

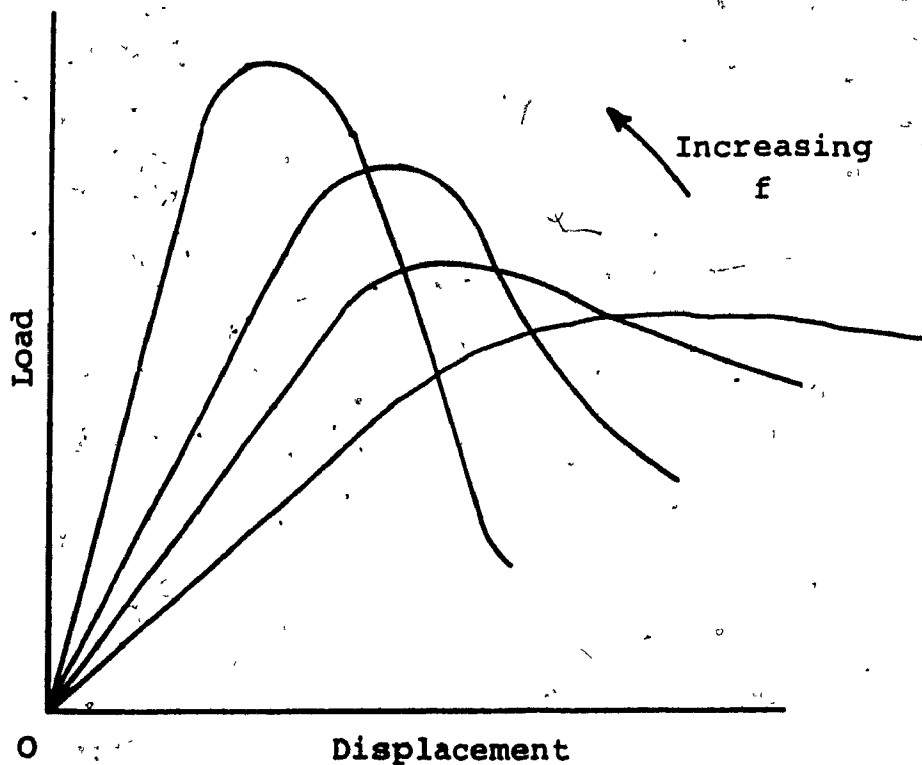
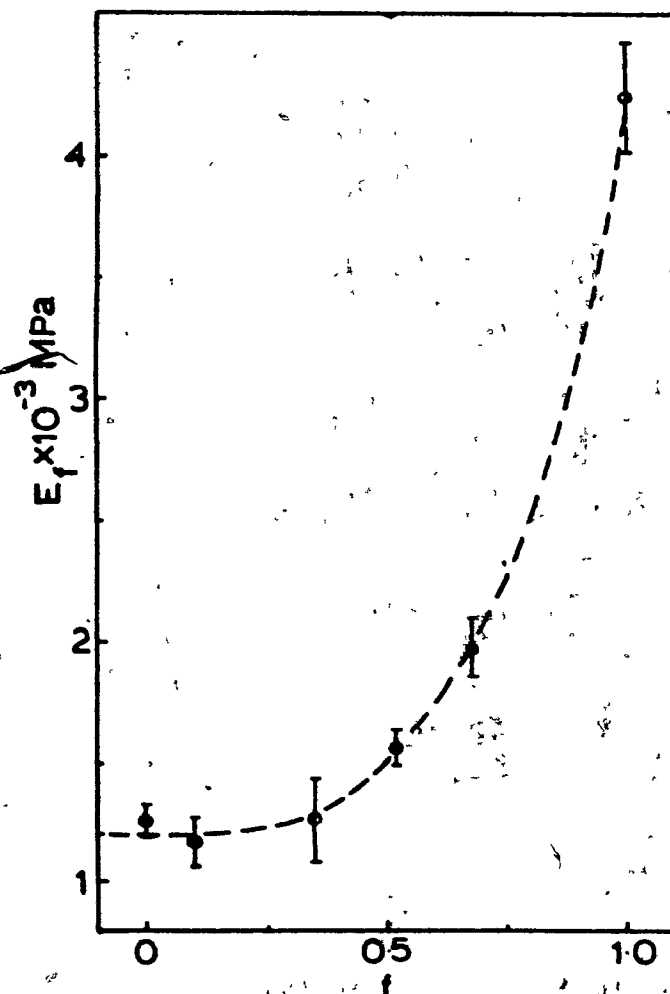
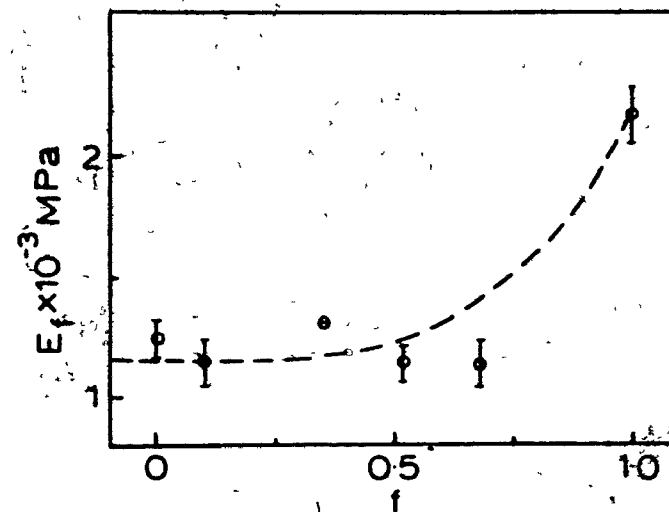


Figure 5.8 Typical Load-Displacement Records for Three Layer Tensile Test Pieces (Schematic). (f is the fractional Thickness of the SF RTP Layer)



(a)



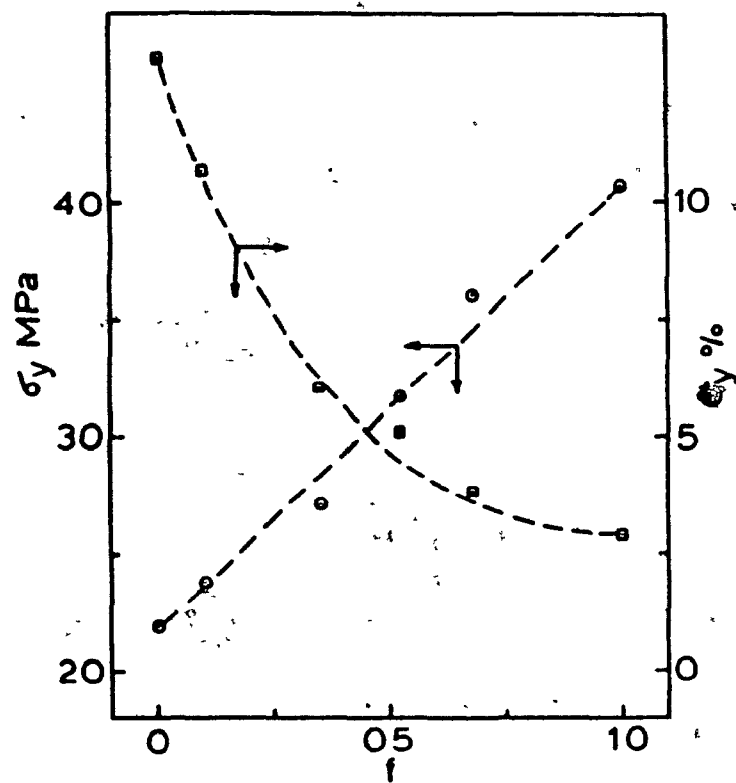
(b)

Figure 5.9 Flexural Modulus (E_f) as a Function of the Fractional Thickness (f) of the SFRTF Layer.
 (a) Test Pieces in L Direction (b) Test Pieces in T Direction.

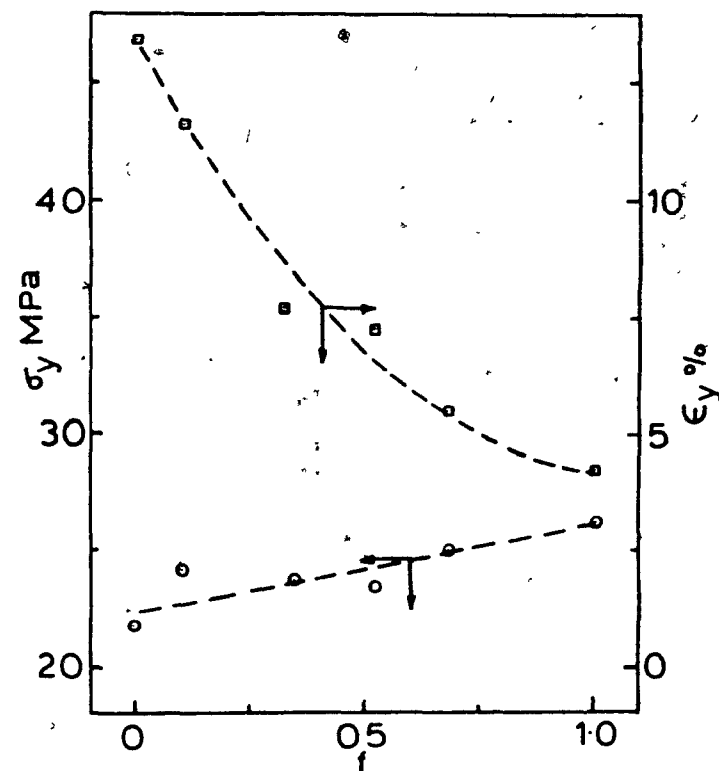
the L and T directions respectively. The actual values are tabulated in Appendix 5.4. As expected, the overall stiffness in the L direction increases with an increase in the fraction f . As discussed earlier in Section 3.5.2, the flexural bending mode imposes a linearly varying strain across the test piece thickness, the strain being maximum at the surface and zero at the neutral axis. Consequently, the effect of the middle SF RTP layer on the measured overall stiffness becomes significant only at relatively large values of f . A small reduction in the overall stiffness of the three-layer test pieces in the T direction for smaller values of f (Figure 5.9b) may be due to the residual stresses present in the multi-layer plaques.

The tensile yield stress as a function of the fractional thickness f of the SF RTP layer is shown in Figures 5.10a and b for the L and T directions respectively, while the individual values are tabulated in Appendix 5.5.1. The tensile yield stress in the L direction increases with fraction f . In the T direction, the tensile yield stress of the SF RTP used in this study is not very different from that of the matrix resin. Consequently, the increase in the tensile yield stress with fraction f is not very pronounced in that direction.

The mode of tensile failure in these test pieces exhibits interesting features. The unreinforced material tends to undergo extensive deformation upon yielding without a brittle failure, but has a low yield stress. SF RTP, on the other hand, has a high



(a)



(b)

Figure 5.10 Yield Strength (σ_y) and Yield Strain (ϵ_y) as Functions of the Fractional Thickness (f) of the SFRTF Layer.

(a) Test Pieces in L Direction (b) Test Pieces in T Direction

yield stress, particularly in the direction of preferential fiber orientation (L direction), but is not capable of undergoing a large deformation upon yielding. In the test piece with a symmetrical three-layer structure used in this section of the study, the two unreinforced surface layers seem to delay the yielding process until a greater extension is reached and, thus, contribute significantly toward the overall ductility. The SF RTP layer makes a major contribution to the overall yield strength. This is demonstrated in plots of the approximate yield strain (ϵ_y) vs. the fractional thickness f of the SF RTP layer, as shown in Figures 5.10a and b. These yield strains were calculated using the crosshead displacement at yield and the test piece gauge length as follows.

$$\epsilon_y = \frac{\Delta l}{l_0} \quad (5.1)$$

where Δl is the crosshead displacement at yield

and l_0 is the test piece gauge length taken equal to the length of its narrow section (28 mm).

During tensile testing, as the yield point is approached, the failure process begins in the middle SF RTP layer, as is indicated by the appearance of several whitened zones in the gauge section. These are associated with fiber-matrix debonding and void formation. The failure continues at the weakest section, and grows across the entire thickness of the SF RTP layer, but is stopped at the interface as the external unreinforced layers continue to deform. In some test pieces having very thick surface

layers, interfacial debonding was seen to occur after yielding due to the shear stress acting along the interfaces.

The above results indicate that in the three-layer structure of coextruded pipe, the overall stiffness, yield stress and yield strain can be varied by varying the layer thickness distribution and the fiber orientation pattern. It should be noted that the characteristics of the interface present in the compression molded plaques may be different from those of the interface formed in coextruded pipe. Moreover, the differential shrinkage of the individual layers during cooling can give rise to residual stresses in the plaques (Appendix 5.13), and these stresses may be of a different nature than those encountered in pipe. However, the results clearly indicate considerable scope for property optimization not only through the proper choice of materials for the individual layers, but also by the control of parameters such as layer thickness distribution and fiber orientation. The coextrusion process thus provides an opportunity to tailor pipe properties to service requirements.

Although, the above study was carried out using plaques in which the unreinforced layers were of the high density resin (PE 2906 M6), the general conclusions are expected to apply for plaques in which the unreinforced layers are of the low density resin (PE 74 A).

5.3.4 Crack Growth Studies

In the following, some relevant concepts of fracture mechanics are briefly discussed, and are used to characterize crack initiation and propagation resistance of the materials used in the study. Certain modifications in the conventional test method are subsequently introduced in order to test multi-layer test pieces and the corresponding results are discussed.

5.3.4.1 Relevant Concepts of Fracture Mechanics

The first theory that postulated that the brittle failure of elastic materials is controlled by the inherent defects present in the components was put forward by Griffith (114). He stated that new surfaces resulting from the extension of a pre-existing crack will form if the release of the stored elastic energy as a result of the crack extension is sufficient to provide the surface energy for the newly formed surfaces. Using this energy balance approach for a slit-like flaw of length $2a$ in an infinite plate remotely loaded under tension normal to the flaw (Figure 5.11), he derived the following expression for the fracture strength, σ_c , under plane stress conditions.

$$\sigma_c = \sqrt{\frac{2 E \gamma_0}{\pi a}} \quad (5.2)$$

where E is the Young's modulus

and γ_0 is the characteristic surface energy of the material.

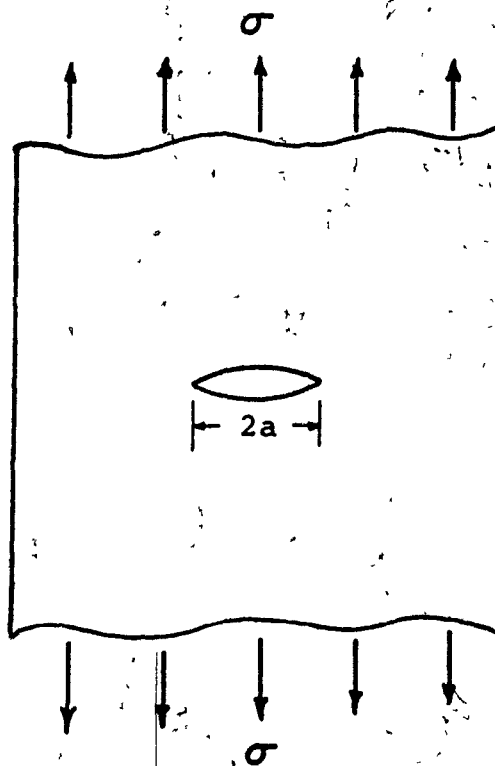
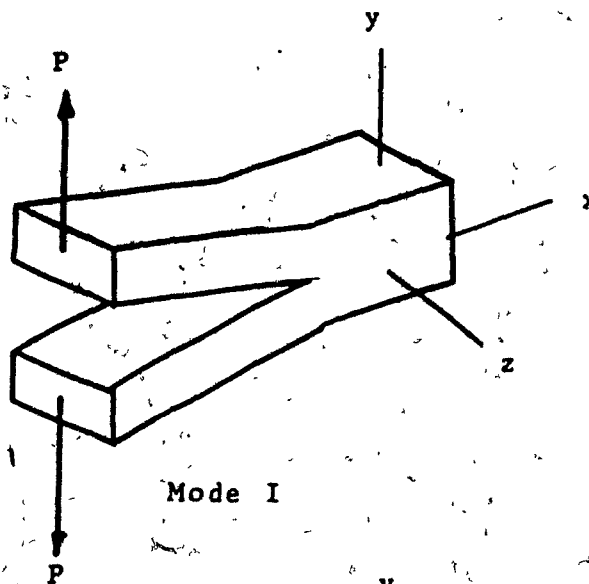


Figure 5.11 Elliptical Flaw in an Infinite Plate.

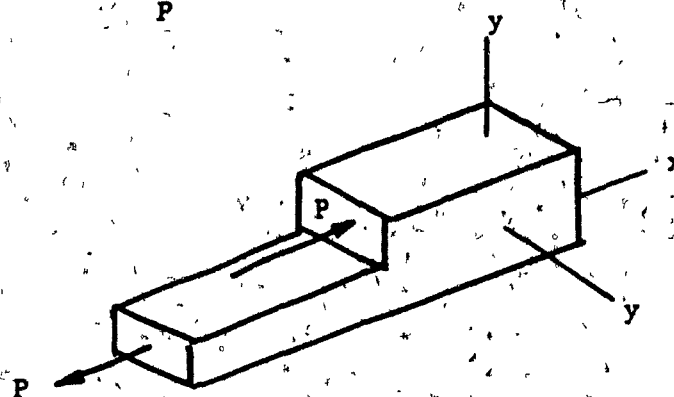
In the case of polymers, γ_0 may reflect the energy necessary to break primary (intramolecular) or secondary (intermolecular) bonds, crosslinks or simply pull out the entangled molecules during crack extension (115). A geometrical model of brittle failure in crystalline polymers in terms of "tie molecules" has been described by Lustiger (116). In composites such as SF RTP materials, the failure may involve additional energy necessary for the fiber-matrix debonding, fiber pullout, post debonding friction at the fiber-matrix interface, surface energy associated with the fibers etc. (32,117-120). In fact, as is shown below, the presence of the fibers can actually change the mode of failure in the matrix due to a complex stress redistribution and intensification, and thus significantly affect the fracture strength.

5.3.4.1.1 Linear Elastic Fracture Mechanics (LEFM)

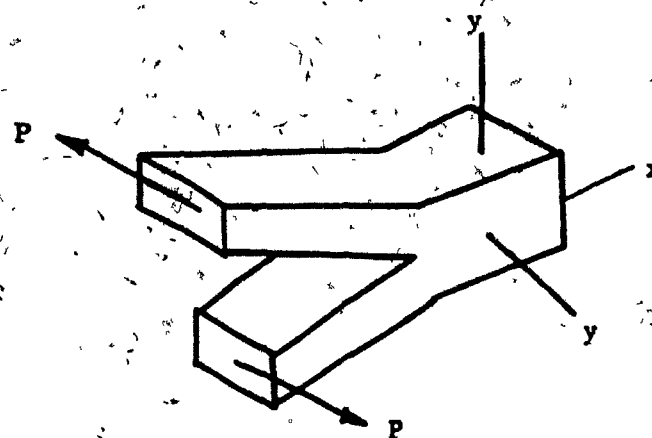
The fracture mechanics of a flawed component of a linearly elastic material (referred to as LEFM) has been rigorously analyzed in terms of the distribution of stresses around the crack tip. The stress field around a crack tip can be classified in terms of three modes of loading that involve different forms of crack surface displacement, as shown in Figure 5.12 (121). Mode I is the crack opening or tensile mode where the crack surfaces move directly apart. In mode II, the sliding mode, the crack surfaces slide over each other in a direction normal to the crack front. Mode III is the tearing mode in which the crack



Mode I



Mode II



Mode III

Figure 5.12 Different Modes of Relative Crack Surface Displacements.

surfaces move relative to each other, and parallel to the crack front. Mode I is encountered in a majority of practical situations including the brittle failure of pipe. Hence, considerable attention has been given to the development of both analytical and experimental methods designed to quantify the mode I stress-crack length relationships (121). The following discussion concentrates on this mode of fracture.

Analysis of the stress distribution around the crack tip indicates that the presence of the crack causes stress intensification so that stresses in its vicinity are considerably higher than the remotely applied stress. This stress intensification is expressed in terms of a stress intensity factor K . For example, for an infinite plate loaded in mode I (Figure 5.11), the stress intensity factor is:

$$K_I = \sigma \sqrt{\pi a Y} \quad (5.3)$$

where σ is the remotely applied tensile stress,

Y is a geometrical factor depending on the crack geometry and loading configuration,

and K_I is the mode I stress intensity factor.

In linear elastic materials, as the stress intensity factor at the crack tip increases, a critical value is reached when catastrophic brittle failure occurs. This critical stress intensity factor, denoted as K_{IC} , is a material property that characterizes its fracture toughness. The value of K_{IC} is, however, affected by specimen thickness as the thickness

determines whether a major fraction of the crack front is under a biaxial (plane stress) or triaxial (plane strain) state of stress. K_{IC} can also be affected by temperature and the rate of loading.

An alternative energy balance approach to the fracture mechanics of linear elastic solids leads to the critical strain energy release rate, G_{IC} , as the LEFM fracture toughness parameter. It represents the critical rate of release of strain energy stored in the specimen at the onset of catastrophic failure. It is related to the fracture toughness parameter, K_{IC} , as follows.

$$G_{IC} = \frac{K_{IC}^2}{E} \quad (5.4)$$

The standard test for determining the LEFM fracture mechanics parameter, K_{IC} or G_{IC} , involves loading a specimen containing a notch to failure, and calculating the fracture toughness parameter from the conditions of load, crack length, specimen dimensions and configuration at the onset of catastrophic failure (122).

Real materials are rarely perfectly linear elastic solids, and some plastic yielding at the crack tip is unavoidable. Attempts have been made to modify the basic LEFM theory for small amounts of crack tip plastic yielding by assuming that the effective crack length is larger than the physical length by an amount equal to the size of the plastic zone (123,124). For the

determination of a valid LEFM toughness parameter such as K_{IC} or G_{IC} , the standard test requires that the dimensions of the specimen and the crack be much larger than the size of the plastic zone at the crack tip (122). For tough materials like plastics, however, a substantial amount of plastic deformation occurs at the crack tip. Moreover, in SFRTTP materials, a significant amount of slow stable crack growth usually occurs prior to unstable failure. Under such conditions, it becomes difficult to determine a valid LEFM toughness parameter.

In such cases, it is convenient to use the post-yield fracture mechanics concepts such as the path-independent J-integral to characterize the crack initiation resistance of the material.

5.3.4.1.2 The J-Integral Method

The J-integral concept has been put forward by Rice to characterize the resistance of a material to crack extension in the elastic-plastic regime (125,126). The concept is based on the deformation theory of plasticity, i.e., it assumes that the stress-strain relationship of an elastic-plastic material can be considered to be equivalent to that of a non-linear elastic body with the same stress-strain curve. For the two dimensional case, the J-integral is defined as follows (Figure 5.13):

$$J = \int_{\Gamma} (W dy - T_i \frac{\partial u_i}{\partial x} ds) \quad (5.5)$$

where Γ is any contour surrounding the crack tip,

T_i is the component of outward traction on the contour,

u_i is the component of the displacement vector at ds ,

ds is the incremental distance along the contour,

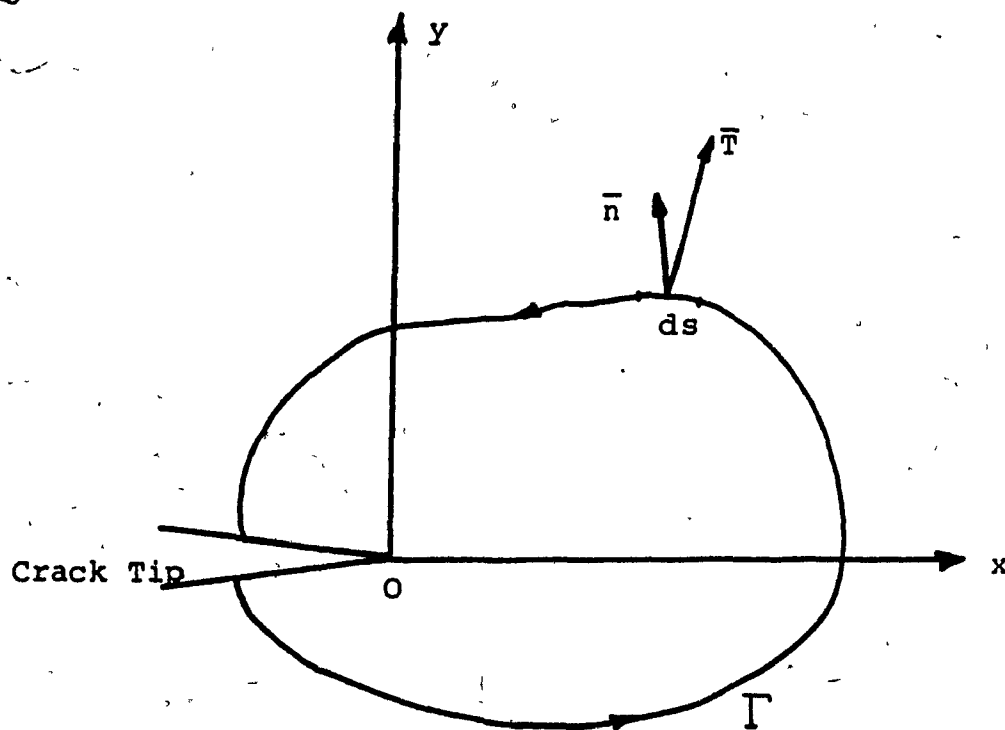
x, y are the Cartesian coordinates

and W is the strain energy density given as follows:

$$W = \int \sigma_{ij} d\epsilon_{ij} \quad (5.6)$$

σ_{ij} and ϵ_{ij} are components of the stress and strain tensors respectively.

It can be shown that the J-integral is path-independent when applied around the crack tip from one surface to another. In physical terms, the product $J \cdot da$ represents the energy available for crack extension by an amount da . Due to its path independence, the J-integral is a crack tip parameter, and hence can be used as a measure of the resistance of a material to crack extension. It must be noted that the assumption of the deformation theory of plasticity is not valid if unloading of the specimen occurs. Crack extension, however, leads to partial unloading of the test piece and the J-integral concept must thus be applied only for an infinitesimal increment in the crack length during which the amount of unloading is insignificant. Hence, the J-integral concept can be used to characterize only the crack initiation resistance of a material, and not its crack propagation resistance. It may be noted that for a linear elastic



$$J = \oint_{\Gamma} (W \, dy - T_i \frac{\partial u_i}{\partial x} \, ds)$$

Figure 5.13 Crack Tip Coordinate System and Arbitrary Line Integral Contour.

material, the J-integral is equivalent to the strain energy release rate, G.

5.3.4.1.3 Standard Method for Determining the Critical J-Integral Value (J_{IC})

The J-integral value at the initiation of crack extension is denoted as J_{IC} . ASTM standard test method E813 provides a procedure for determining this value (127). This method is based on the multiple test piece procedure developed by Bagley and Landes (128). Single edge notched bend test pieces such as the one shown in Figure 5.14 are convenient, but other types of test piece may also be used (127). The method consists of loading several identical test pieces having the same initial crack length (a_0) to different load point deflections in the three point bending mode, and then unloading. These test pieces thus exhibit different amounts of crack extension. After unloading, the test pieces are broken apart, and the crack advance (Δa) during loading is determined. Also the area U under the load-load point deflection is determined (Figure 5.15). The J-value at the point of unloading the test piece is determined as follows.

$$J = \frac{2 U}{B(D-a)} \quad (5.7)$$

where B, D and a are the test piece thickness, width and final crack length respectively.

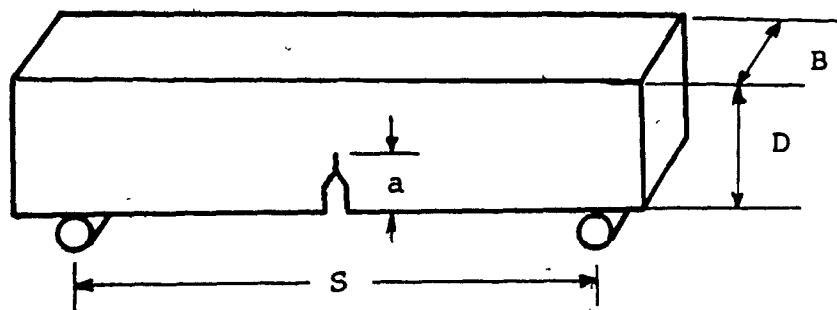
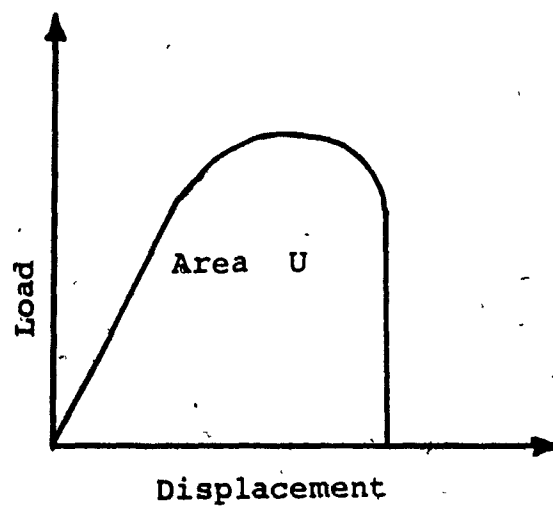


Figure 5.14 Single Edge Notched Three-Point Bending Test Piece



$$J = \frac{2 U}{B(D - a)}$$

Figure 5.15 The Area U under the Load versus Displacement Curve.

Various J-values are plotted against the crack advance (Δa), and a straight line fitted by linear regression is drawn through them. Also, a blunting line is drawn to account for the apparent crack advance due to the crack tip blunting according to the following equation.

$$J = 2 \sigma_y \Delta a \quad (5.8)$$

where σ_y is the yield strength of the material.

The J value corresponding to the point of intersection of the regression straight line with the blunting line is taken as a candidate J_Q value, and is considered a valid J_{IC} value if the certain criteria specified in the standard test procedure (127) are satisfied.

It must be noted that the J-integral concept and related fracture mechanics methods have been developed for a homogeneous, isotropic material. SFRTTP composites are non-homogeneous and non-isotropic materials. However, such methods are frequently used to study their fracture behaviour in as much as they represent the flow of energy at the crack tip during crack extension, and provide a basis for comparison (129-134). In the following, the J-integral method is used to characterize the resistance of the materials used in the study to crack initiation from a pre-existing notch.

5.3.4.2 Test Piece Preparation and Experimental Procedure

Single-layer plaques of the three materials, viz., high density unreinforced resin (PE 2906 M6), low density unreinforced resin (PE 74A) and SF RTP were compression molded as described in Section 5.3.2 with a nominal thickness of 6.3 mm (0.25 in). The plaques of SF RTP had a preferential fiber orientation along their longer side. Test pieces of dimensions 75 x 12.5 (width D) x 6.3 mm (thickness B) (3 x 0.5 x 0.25 in) were machined from the plaques in the L and T directions as shown in Figure 5.6. An initial crack of nominal depth of 5.3 mm was made in each test piece giving a ratio of initial crack length to test piece depth, $(a/D)_0$, of 0.42. The ASTM E813 procedure suggests the use of an initial crack produced by growing a fatigue crack from a machined notch. A crack made using a razor blade was, however, used due to its greater sharpness (91). To make the initial crack, a 3 mm deep prenotch was made with a band saw and a sharp razor blade was then pushed into the prenotch using a specially made notching tool to make a sharp crack of the desired depth. The notching tool is shown schematically in Figure 5.16. It has a base and a top blade holder. The blade is clamped between the two plates of the blade holder by a pair of screws such that a desired length extends out of the holder. The test piece is supported on the base, and the blade is slowly pushed into the prenotch on a laboratory press. Use of appropriate stoppers allows notches of desired depth to be made.

The notched test pieces were loaded in the three point bending mode so as to cause the desired amount of crack extension. All testing was carried out at room temperature and at

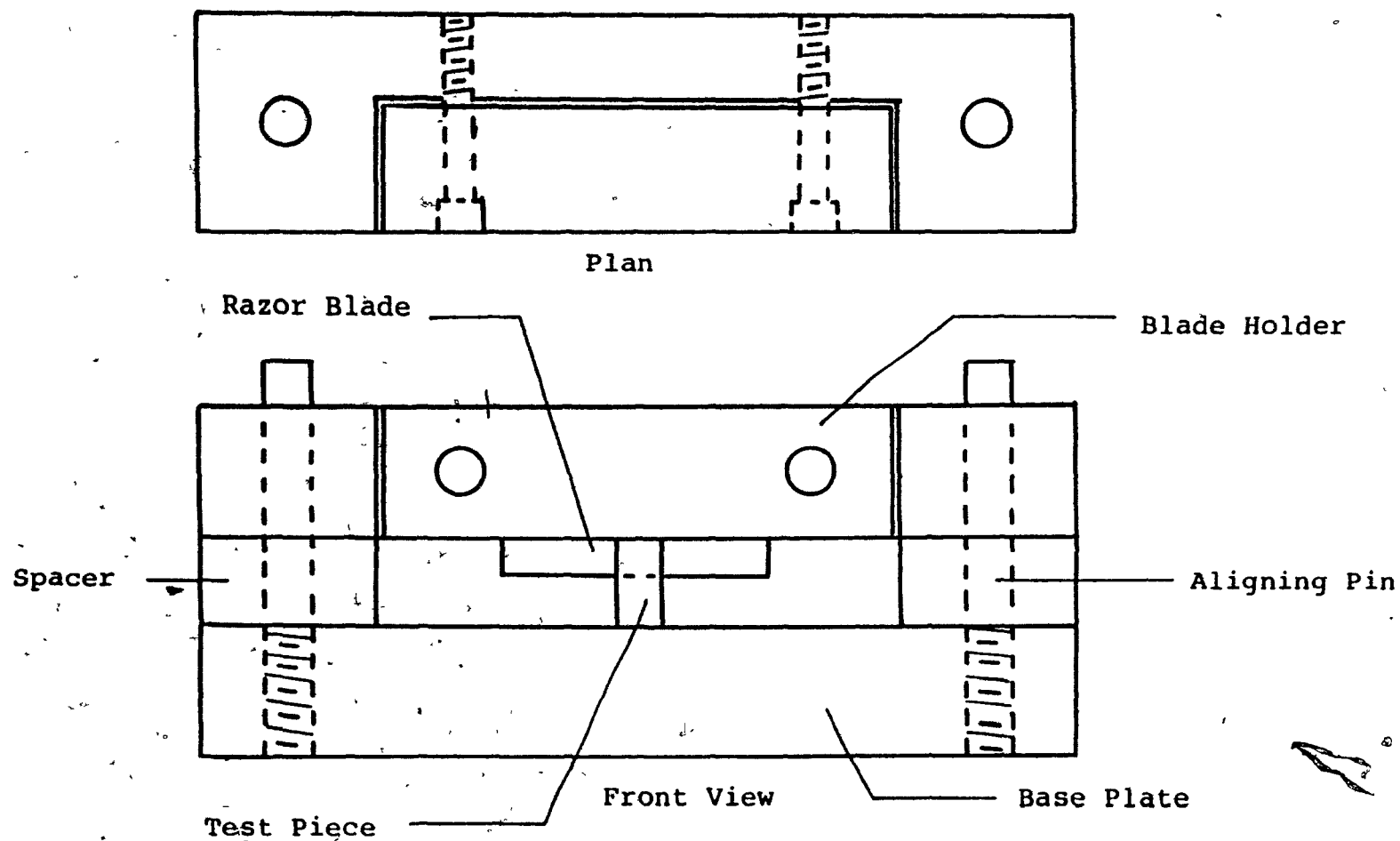


Figure 5.16 Notching Tool for Making Initial Crack

a loading rate of 0.5 mm/min. In order to measure the amount of crack extension, the test pieces were subsequently frozen in a bath of liquid Nitrogen, and broken apart. The characteristics of the fracture surface due to the brittle failure under frozen conditions are different from those due to crack extension at room temperature during loading. The crack front at the end of the loading could hence be easily identified, and the amount of crack extension from the initial notch could be measured, as shown in Figure 5.17. The crack extension was measured, using a low magnification optical microscope in the reflection mode to an accuracy of 0.024 mm. The crack extension is rarely uniform across the test piece thickness due to the change from plane stress conditions at the surfaces to plane strain conditions in the center. Seven equidistant measurements were made across the test piece thickness from one surface to the other, and an average value was taken to calculate the J-integral.

No significant difference was found in the results for the test pieces of the unreinforced materials taken in the L and T directions. In the case of SF RTP, however, a marked effect of fiber orientation was to be expected. In the test pieces of SF RTP along the L direction (direction of preferential fiber orientation), the fibers are preferentially oriented normal to the crack surface, as illustrated in Figure 5.18a, while in the test pieces taken along the T direction (direction normal to that of the preferential fiber orientation), the fibers lie parallel to the crack surfaces. There are two distinct possibilities in

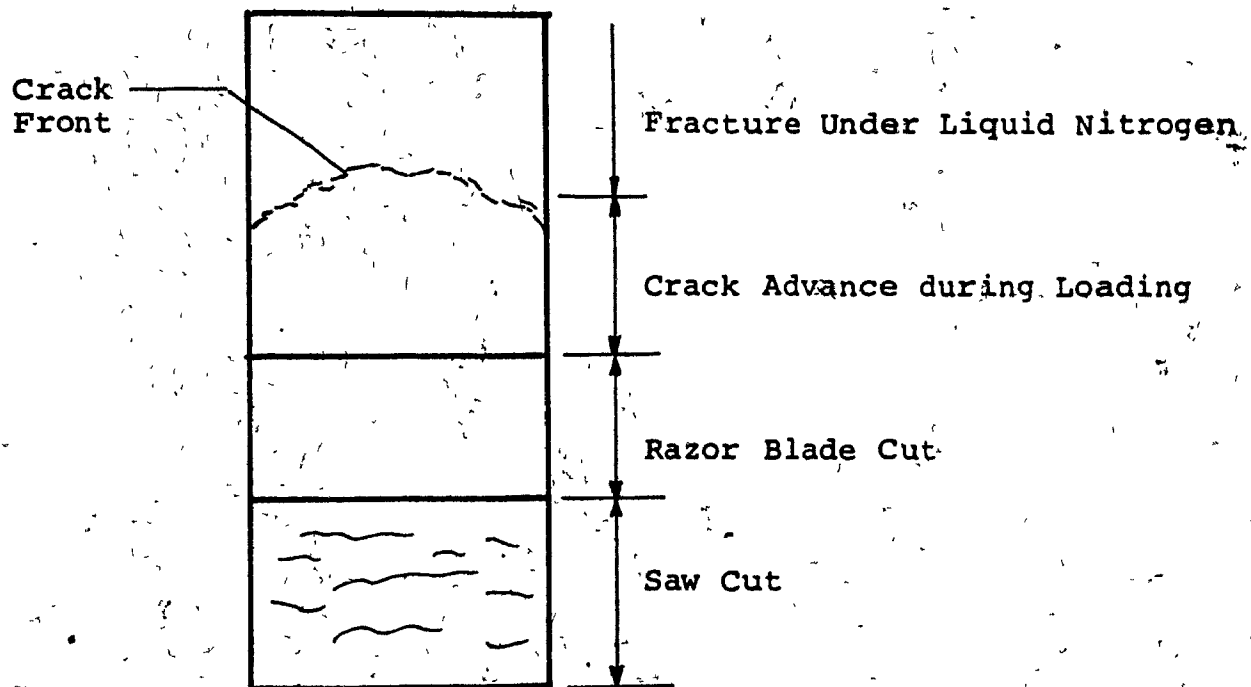


Figure 5.17 Crack Surface of a Test Piece.

the latter case; the fibers may be preferentially oriented in the width direction normal to the crack front, as shown in Figure 5.18b or in the thickness direction parallel to the crack front, as shown in Figure 5.18c. As the crack growth studies discussed later on show, there was no significant difference in the results for these two types of test piece. Hence these two cases of preferential fiber orientation along the crack surface are considered equivalent under the assumption of transverse isotropy.

5.3.4.3 Results and Discussion

The $J-\Delta a$ plots for the high density unreinforced resin (PE 2906 M6) and SF RTP (PE 2906 M6 + 30% fibers) with test pieces taken in both the L and T directions are shown in Figure 5.19. The blunting lines based on $J = 2 \sigma_y \Delta a$ are drawn using the previously determined values of yield strength (Section 5.3.3.2). The individual test piece dimensions, crack extension data and the J-integral values are tabulated in Appendix 5.6. The J_{IC} values determined from the intersection of the linear regression lines through the experimental data points and the blunting lines are summarized in Table 5.2. The scatter of the experimental data points around the regression lines is usually unavoidable due to the uneven crack growth across the crack front, particularly in the case of composite materials.

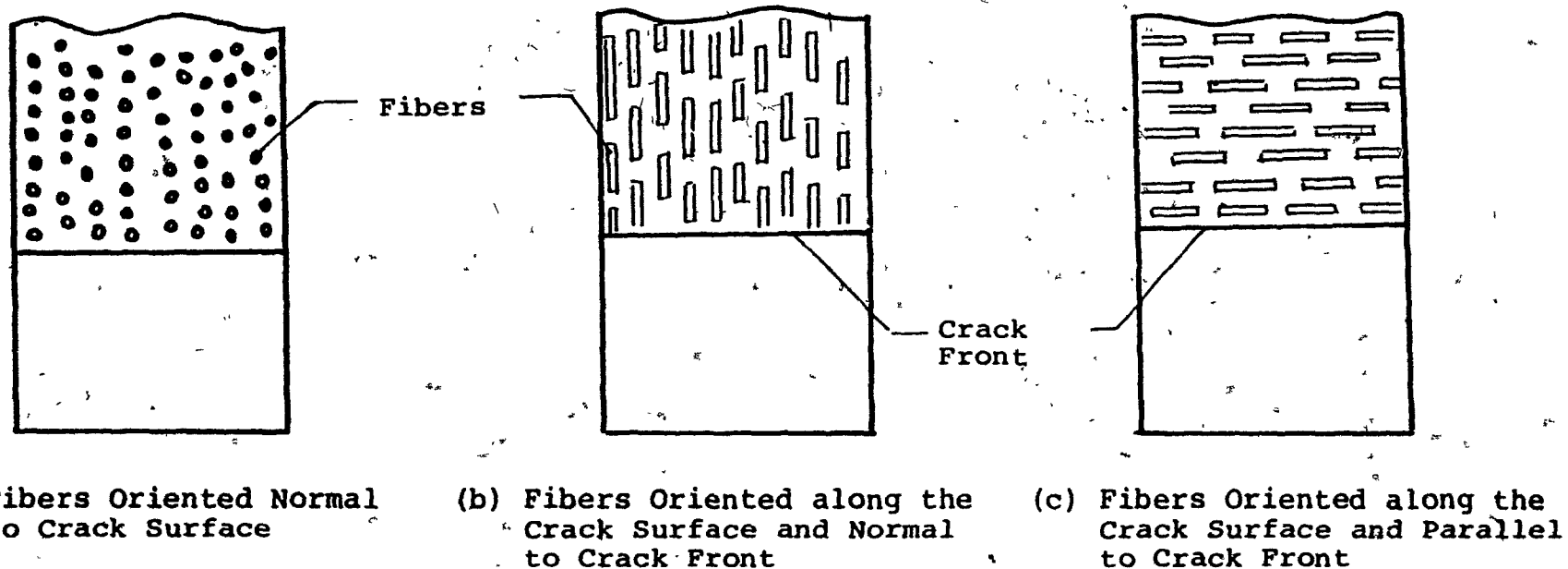


Figure 5.18 Different Cases of Fiber Orientation with respect to the Crack Surface.

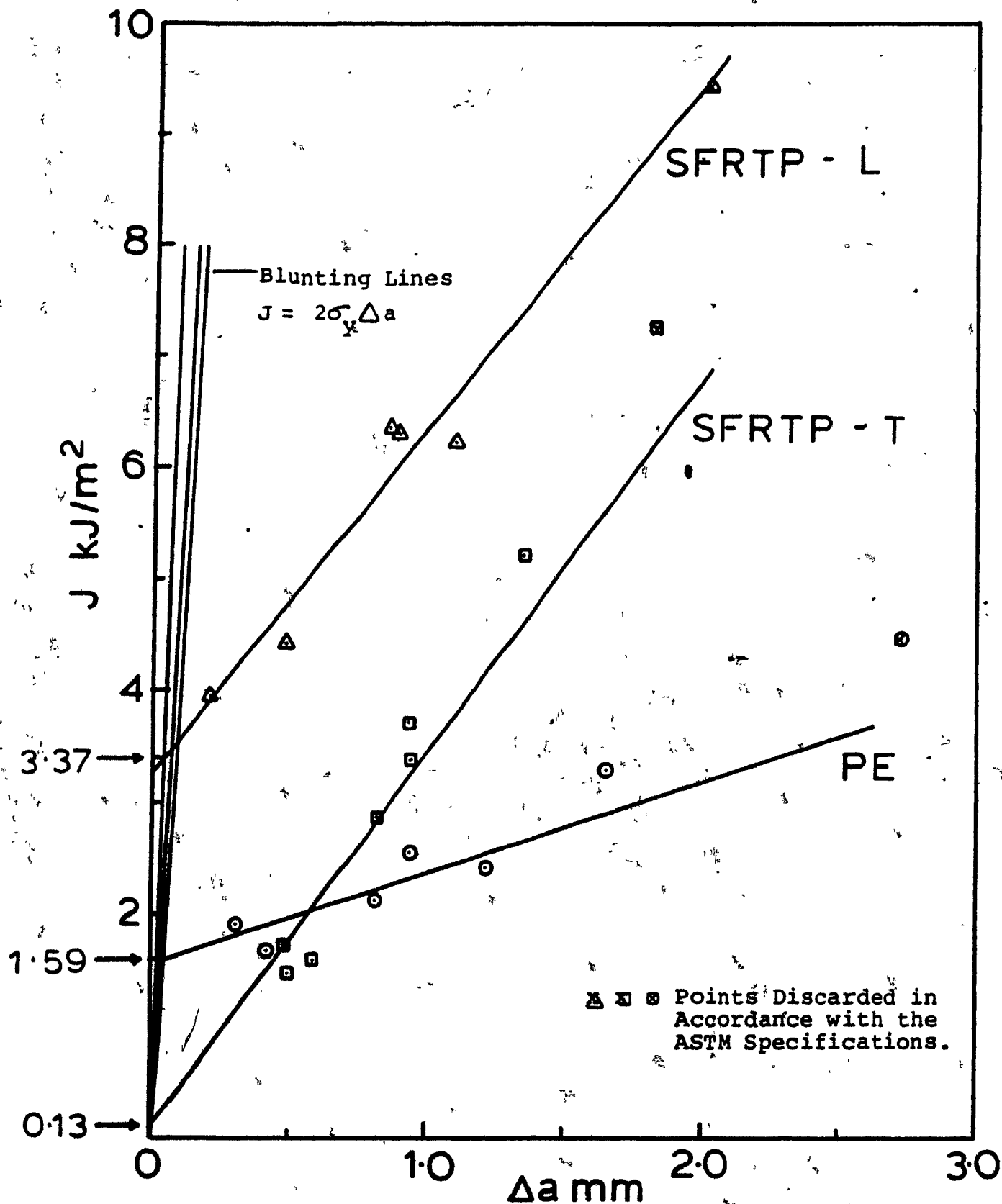


Figure 5.19 $J - \Delta a$ Plots for the High Density Unreinforced Polyethylene and SF RTP - L and T direction Test Pieces

Table 5.2**Crack Growth Characteristics of the Materials Used**

Material	J_{IC} (KJ/m ²)	K_R (MPa/m)
Unreinforced High Density (PE 2906 M6)	1.59	1.69 (Plateau)
SF RTP - L Direction	3.37	-
SF RTP - T Direction	0.125	-

Table 5.3**Plateau K_R Values for Multi-layer Test Pieces**

Plaque Type	Fractional Thickness of SF RTP Layer	Crack Growth Mode	K_R (Plateau) MPa/m
I	0.23	Unreinforced to SF RTP - L	1.88
II	0.58	Unreinforced to SF RTP - L	3.25
III	0.35	Unreinforced to SF RTP - T	1.87
IV	0.6	Unreinforced to SF RTP - T	2.16
V	0.61	SF RTP to Unreinforced - L	2.72
VI	0.69	SF RTP to Unreinforced - T	2.65

As seen from the J_{IC} values reported in Table 5.2, the resistance of the unreinforced high density resin to crack initiation is relatively poor, but it is significantly improved by the presence of fibers oriented normal to the crack surface (test pieces taken along the L direction). The crack initiation resistance is, however, sharply reduced, when the fibers are oriented parallel to the crack surface. As discussed in section 5.3.4.6, matrix characteristics and fiber-matrix adhesion have strong effects on the crack growth resistance. The presence of fibers changes the mode of matrix failure from a brittle to a ductile one involving localized matrix drawing during crack extension. This phenomenon together with fiber orientation is responsible for the observed increase and decrease in the J_{IC} values for the two cases of SF RTP.

The J-Aa curve for the unreinforced low density resin (PE 74 A) is shown in Figure 5.20. This material has a low stiffness ($E_f = 818$ MPa) and yield stress ($\sigma_y = 13$ MPa), and is extremely ductile. Just as in the case of test pieces of the high density resin, the presence of a sharp crack causes stress concentration at the tip. However, due to the material's low yield strength, the stress at the tip exceeds the yield value at a relatively low apparent stress. This causes large scale yielding or plastic deformation at the crack tip effectively blunting it. Crack advance is thus much impeded, as the blunt crack cannot be an effective stress concentrator. Thus, the material is almost completely notch insensitive. Any attempt to cause crack

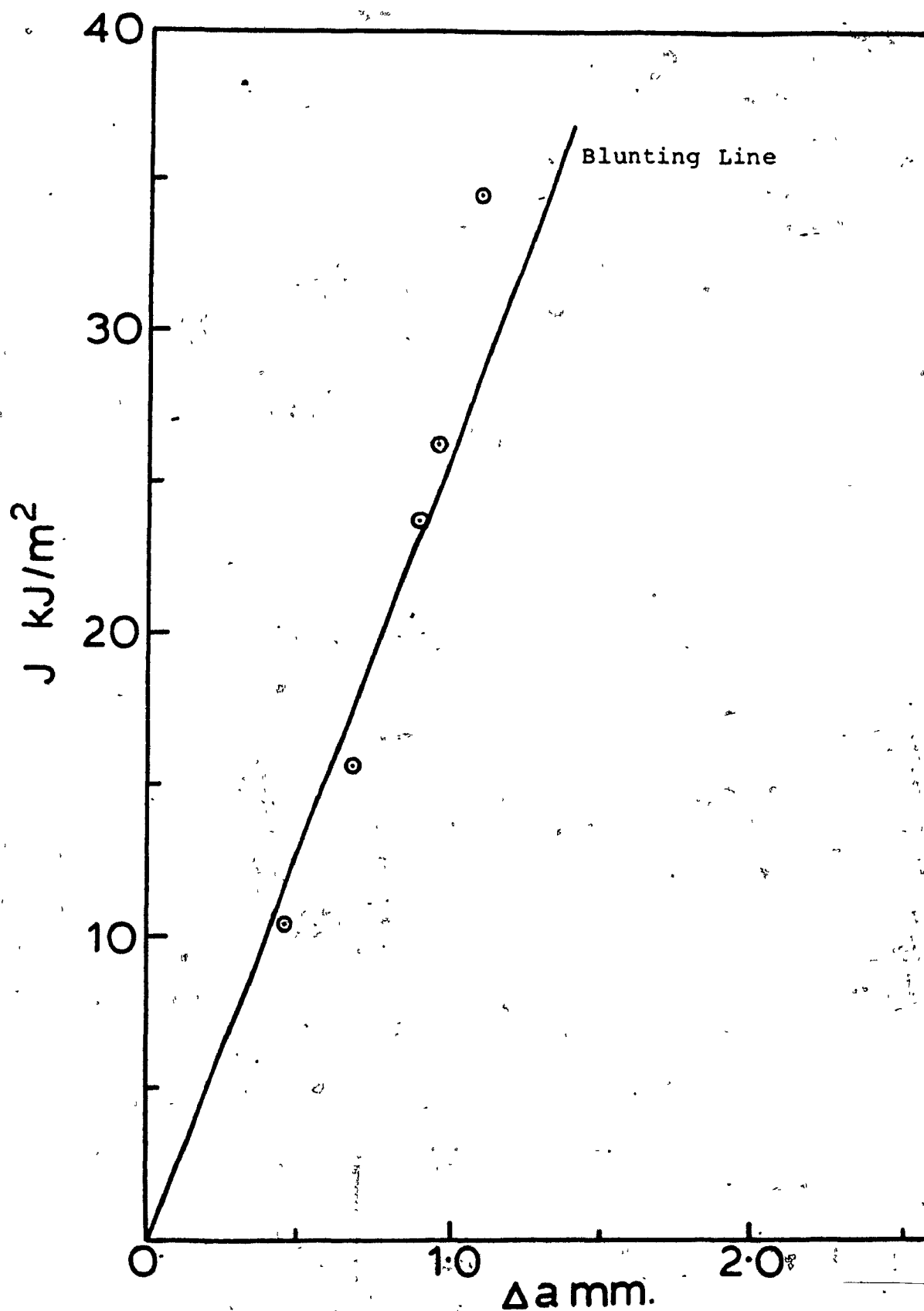


Figure 5.20 $J - \Delta a$ Plot for the Low Density Unreinforced Polyethylene (PE 74 A)

extension results in absorption of a large amount of unrecoverable strain energy at the crack tip due to the extensive plastic deformation, and as seen from the $J-\Delta a$ plot in Figure 5.20, all the points lie along the blunting line. Therefore, no J_{IC} value could be determined in this case. The material's failure is governed by ductile yielding even in the presence of a sharp notch, making it extremely tough. This is an excellent example of how a material possessing a low yield strength can be highly resistant to crack extension, and illustrates the characteristics desirable in a resin to be used for the surface layers of coextruded pipe in order to prevent the extension of cracks originating at the surface into the thickness of the wall (cf. Section 5.2.2).

5.3.4.4 Crack Propagation Resistance Characterization

As mentioned earlier, the J -integral concept is not valid for characterizing the resistance of a material to crack extension over larger distances. Also no suitable method based on elastic-plastic fracture mechanics (EPFM) is currently available for this purpose. Under the circumstances, the R curve method based on linear elastic fracture mechanics (LEFM) modified for limited crack tip plasticity was used in the following (135). The R curve is a plot of a parameter characterizing the developing fracture toughness of a material, for example the stress intensity factor, K_R , or the strain energy release rate, G_R , as the crack extends. During the slow stable crack propagation, the

developing crack growth resistance K_R is equal to the applied stress intensity factor K . Hence, under the conditions of slow stable crack growth, the R curve can be determined by increasing the stress intensity through increments of load or displacement applied to a notched test piece, and measuring the corresponding crack length increments. This crack length may include the small plastically deformed zone at the crack tip.

5.3.4.4.1 R Curve Determination

ASTM standard E561 describes a procedure to determine R curve using three test piece geometries and loading configurations, viz., axially loaded center-cracked tension plate, compact tension specimen and the crack-line-wedge-loaded specimen. It, however, points out that several other test piece geometries and loading configurations can also be used (135). The use of a single edge notched (SEN) test piece loaded in the three point bending mode (Figure 5.14) has been found convenient by several investigators to generate R curves, particularly due to the ease of test piece preparation (129,130). In this method, a three-point-bend notched test piece is loaded at a low rate of loading to obtain a slow stable crack advance until the point of instability, and a load-load point deflection record is generated. Several points are selected from the record to determine the corresponding applied stress intensity factors and crack lengths.

The applied stress intensity factor for a three-point-bend test piece is as follows:

$$K_I = \frac{P S}{B D^{3/2}} f(a/D) \quad (5.9)$$

where P is the applied load,

S is the test piece span,

B , D are the test piece thickness and width respectively,

and $f(a/D)$ is the crack length calibration factor.

For the above loading configuration, the calibration factor is given as follows (121):

$$f(a/D) = \frac{3(a/D)^{1/2} [1.99 - (a/D) \{1 - a/D\} \{2.15 - 3.93(a/D) + 2.7(a/D)^2\}]}{2[1 + 2(a/D)] [1 - (a/D)]^{3/2}} \quad (5.10)$$

In order to calculate $f(a/D)$ and, hence K_I , it is necessary to know the dimensionless crack length (a/D) at the selected points of the test record. In the following, the compliance method is used to determine the effective crack length. In this method, secants are drawn to the selected points from the origin (Figure 5.21). The reciprocal of the slope of such a secant is the compliance C (deflection/load) at that point. Knowing the compliance at a point, the corresponding crack length can be determined, if the compliance-crack length relationship is known. The following method was followed to determine analytically this relationship (129,135,136).

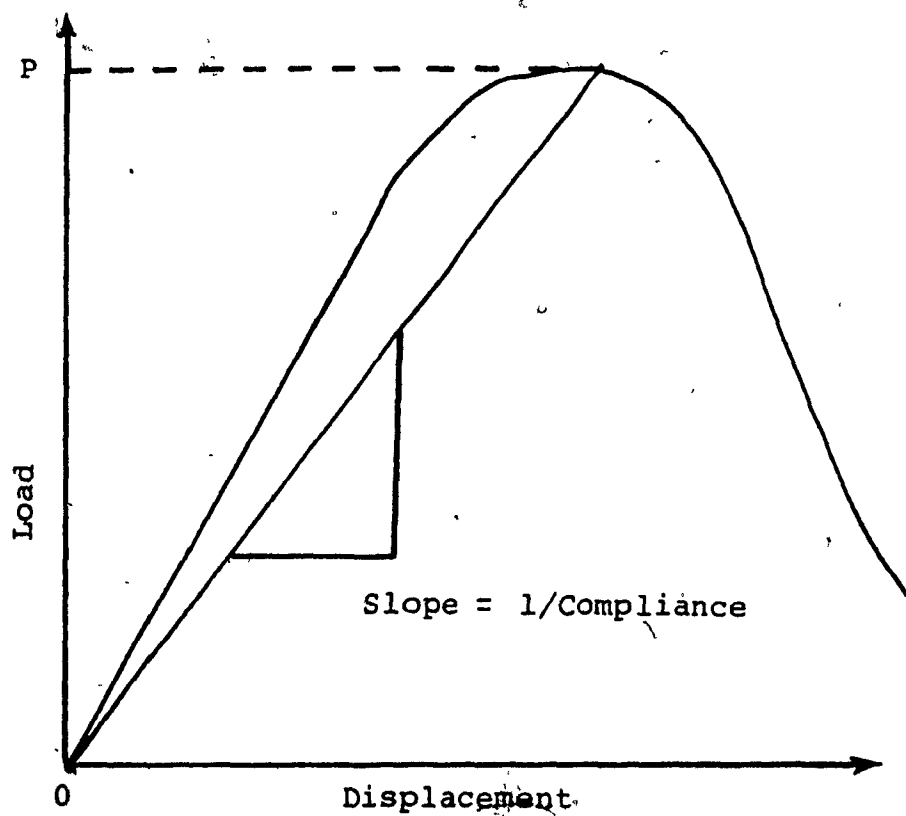


Figure 5.21 Effective Crack Length Determination by the Secant Method.

The strain energy release rate during crack propagation is as follows:

$$G = \frac{K_R^2}{E} = \frac{P^2}{2BD} \frac{dC}{d(a/D)} \quad (5.11)$$

where K_R is the stress intensity factor
and P is the applied load.

Combining Equations (5.9) and (5.11), a relationship between the compliance, crack length and the test piece dimensions is obtained.

$$\frac{dC}{d(a/D)} = \frac{2}{EB} (S/D)^2 [f(a/D)]^2 \quad (5.12)$$

Integration of the above equation gives the compliance-crack length relationship.

$$CEB = C_0EB + 2 (S/D)^2 \int_{(a/D)_0}^{(a/D)} [f(a/D)]^2 d(a/D) \quad (5.13)$$

In the above equation, the compliance is de-dimensionalized by the elastic modulus E and the test piece thickness B . The dimensionless compliance, C_0EB , corresponds to the initial crack length $(a/D)_0$, and can be determined from the initial slope of the load-deflection curve for a notched test piece having a known initial crack length $(a/D)_0$. The dimensionless compliance, CEB , corresponds to the effective crack length (a/D) , which is the physical crack length plus the length of the plastic zone ahead of the crack tip. The compliance versus crack length curve is shown in Figure 5.22. A BASIC programme to compute the relationship is listed in Appendix 5.7. Since the initial point

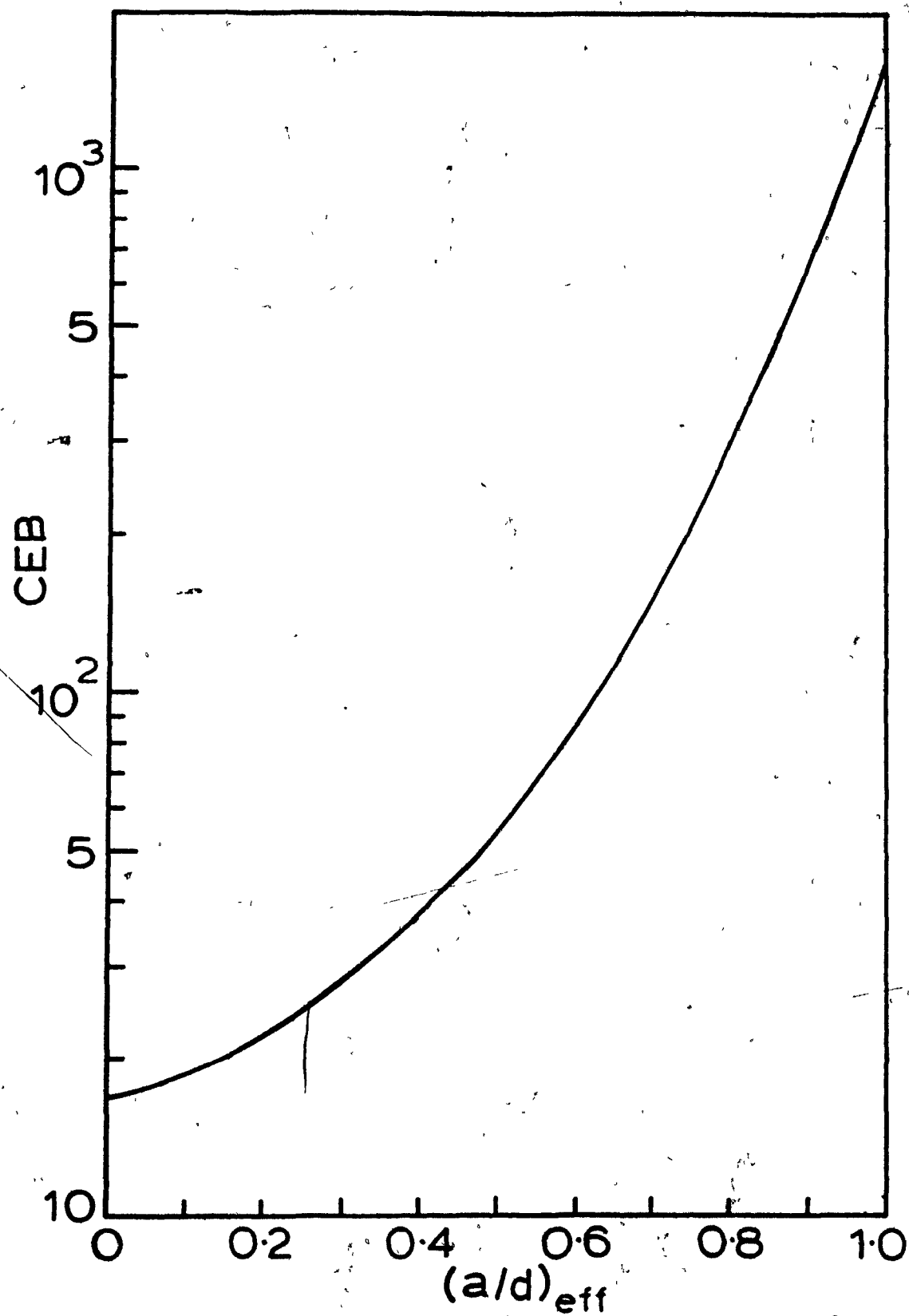


Figure 5.22 Compliance (CEB) versus Effective Crack Length $(a/D)_{eff}$ Relationship

$(C_{0EB}, (a/D)_0)$ represents a single calibration point, the curve in Figure 5.22 should be transposed vertically so as to pass through the initial point for each test piece (135). Moreover, since the compliance is de-dimensionalized with respect to stiffness, the curve in Figure 5.22 can be used for any homogeneous material.

Knowing the relationship between compliance (CEB) and crack length (a/D) , the crack length corresponding to any compliance can be determined. Knowing the crack length, the calibration factor $f(a/D)$ and hence, the stress intensity factor, K_R , can be determined. This is done for all selected points on the load-load point deflection test record, and the R curve is prepared as a plot of applied stress intensity factor, K_R , vs. effective crack length, (a/D) , or crack length extension $(\Delta a/D)$.

It may be noted that the R curve prepared in this way for a homogeneous material should be independent of the initial crack length $(a/D)_0$ providing the ligament in front of the crack tip remains predominantly elastic during the test (137).

The above-described procedure was used to characterize the crack growth resistance of the high density, unreinforced resin (PE 2906 M6) and SFRTF (PE 2906 M6 + 30% fibers) with test pieces taken in both L and T directions. The test could not be carried out for the low density, unreinforced resin (PE 74 A) since extensive yielding at the crack tip caused its blunting.

5.3.4.5 Results and Discussion

Test pieces similar to those used for J_{IC} determination were prepared to generate the R curves (Figure 5.14). The R curves are often plotted in terms of the effective crack extension (Δa), and are expected to be independent of the initial crack length (a_0), as mentioned above. However, for the R curves of the multi-layer test pieces to be described later, this is not the case. Hence, the R curves are plotted here in terms of the effective crack length, $((a/D)_{eff})$. Figure 5.23a to c show the R curves for the high density, unreinforced resin (PE 2906 M6), SF RTP-L and SF RTP-T test pieces respectively. The individual data points used in plotting the R curves are given in Appendix 5.8. At least two, and usually three, test pieces were used for each case to check the reproducibility. As can be seen from Figures 5.23, the R curves for the individual test pieces can be superimposed reasonably well by transposing them horizontally (with respect to the effective crack length) when the initial crack lengths are different, thus demonstrating their independence of $(a/D)_0$.

The R curve for the high density, unreinforced resin shows an initial rise, and then achieves a plateau, so that the subsequent crack extension occurs at a more or less constant value of K_R . For SF RTP-L test pieces with fibers oriented normal to the crack surface, on the other hand, the R curve increases continuously indicating an increasing resistance to the extending crack. A rising R curve is also obtained when fibers are oriented

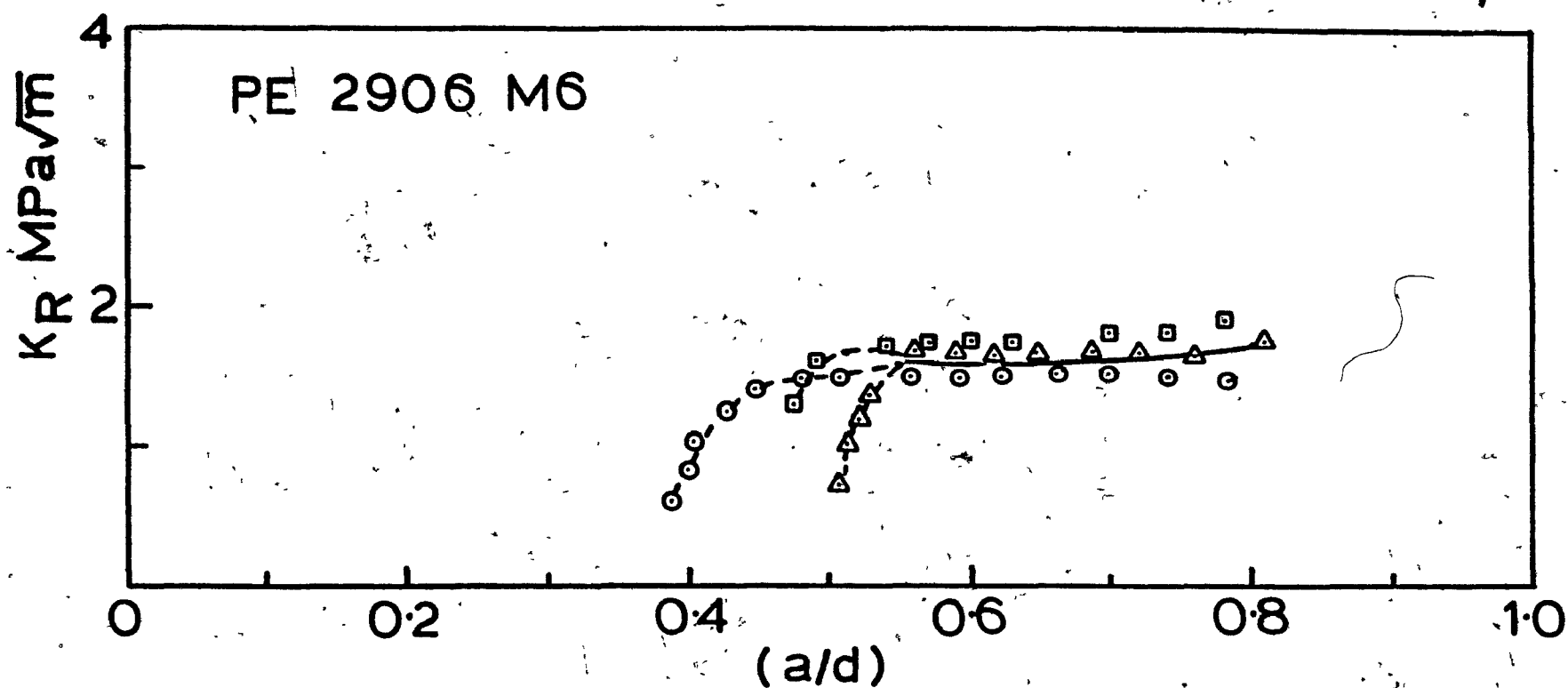


Figure 5.23a R Curve for the High Density Unreinforced Polyethylene (PE 2906 M6)

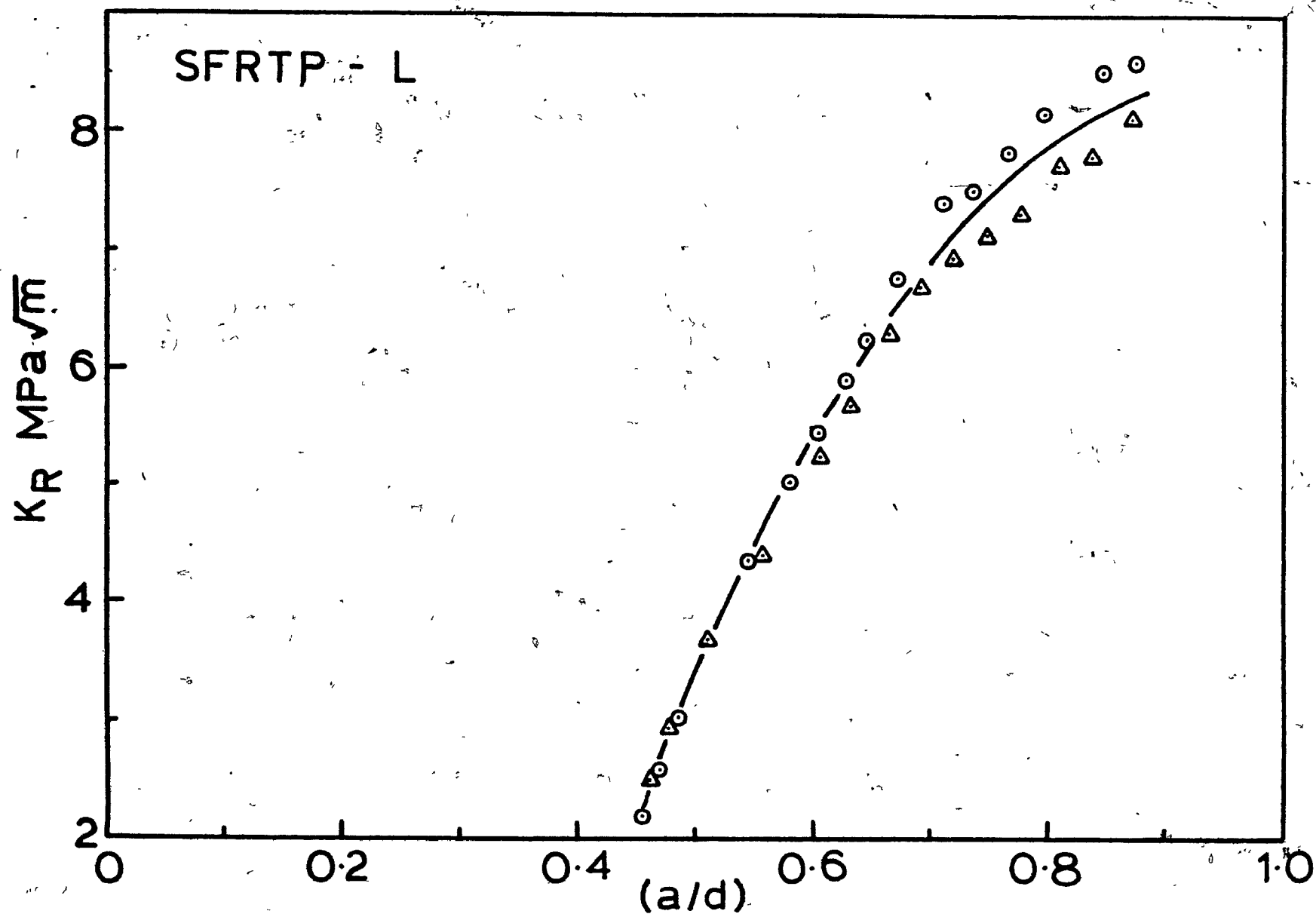


Figure 5.23 R Curve for SF RTP - L Direction Test Pieces.

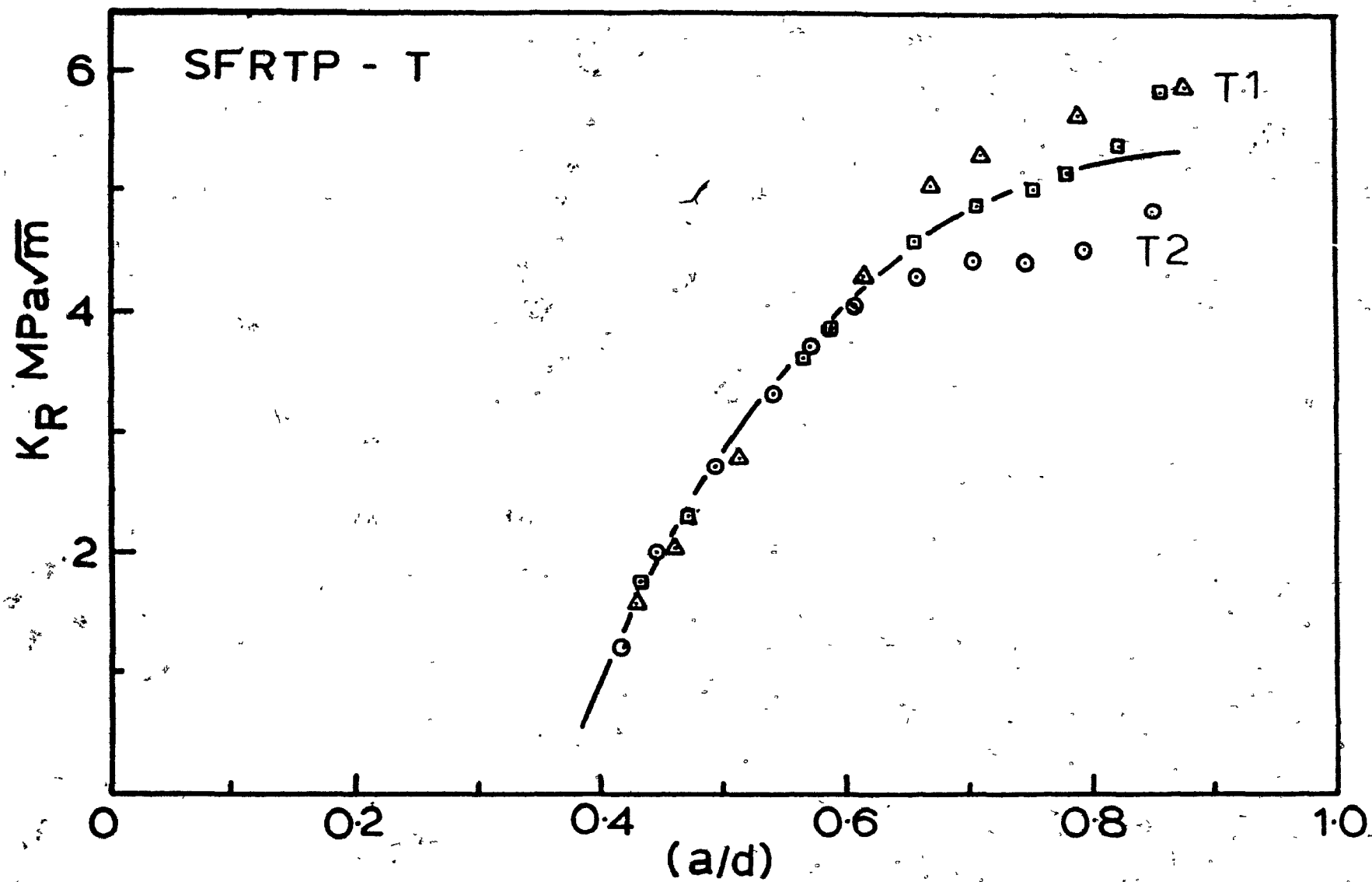


Figure 5.23c R Curve for SF RTP - T Direction Test Pieces.

along the crack surface, as in SF RTP-T test pieces, although the rise is less pronounced. Two different cases of T direction test pieces illustrated in Figure 5.18b and c are considered in Figure 5.23c. The curve marked T1 refers to the case where fibers lie along the crack surface in the width direction, and hence normal to the crack front (Figure 5.18b). The curve marked T2 refers to the case where fibers lie along the crack surface oriented in the thickness direction, and hence parallel to the crack front (Figure 5.18c). The difference between these two cases is not very large.

The above results show that the presence of fibers dramatically affects the fracture toughness of a material in slow crack growth. While a preferential orientation of fibers normal to the crack surface increases the crack initiation resistance (J_{IC}), a preferential orientation in the plane of the crack surface drastically reduces this resistance. In general, the presence of fibers leads to a rising R curve with significant improvement in the developing crack extension resistance. Again, the increase is more pronounced when fibers are oriented normal to the crack surfaces.

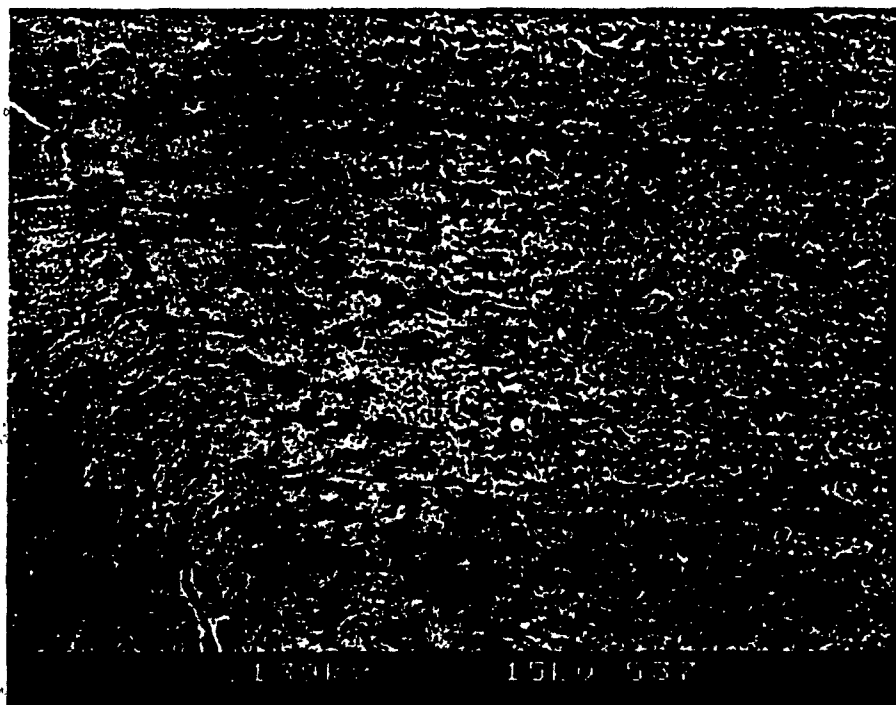
5.3.4.6 Fractographic Analysis

A fractographic examination of the crack surfaces of the unreinforced and SF RTP test pieces was carried out in order to

understand the effect of fibers on the crack growth resistance in physical terms.

Figure 5.24 shows a scanning electron micrograph of the fracture surface of the high density unreinforced resin (PE 2906 M6) near the crack front. The fracture surface is remarkably flat with little or no sign of even microscopic yielding, thus indicating a brittle cleavage. This material is extremely notch sensitive, although in the absence of a notch, it exhibits macroscopic yielding followed by non-uniform plastic deformation typical of a ductile plastic material. Such a behavior has been explained in terms of a hypothesis attributed to Ludvik (138). According to this hypothesis, for a given material, there is a critical shear stress for ductile failure to occur, and a critical normal stress for brittle failure to occur. Depending on the loading conditions, one of these is reached first causing the corresponding type of failure. It also suggests that a low yield stress may be advantageous to prevent brittle failure during slow crack growth.

Figure 5.25 shows a scanning electron micrograph of the fracture surface of a SFRTF-L test piece with fibers oriented normal to the crack surface. Unlike in the case of unreinforced resin, extensive localized matrix drawing surrounding the fibers is present. Moreover, during fracture, fibers are pulled out leaving holes behind. Improved crack initiation and propagation resistance is thus explained by the additional energy needed for



←
Crack

Figure 5.24 Scanning Electron Micrograph Showing Fracture Surface of a High Density Unreinforced PE Test Piece.



Figure 5.25 Scanning Electron Micrograph Showing Fracture Surface of a SF RTP - L Direction Test Piece.
(Fibers Preferentially Oriented Normal to the Crack Surface)

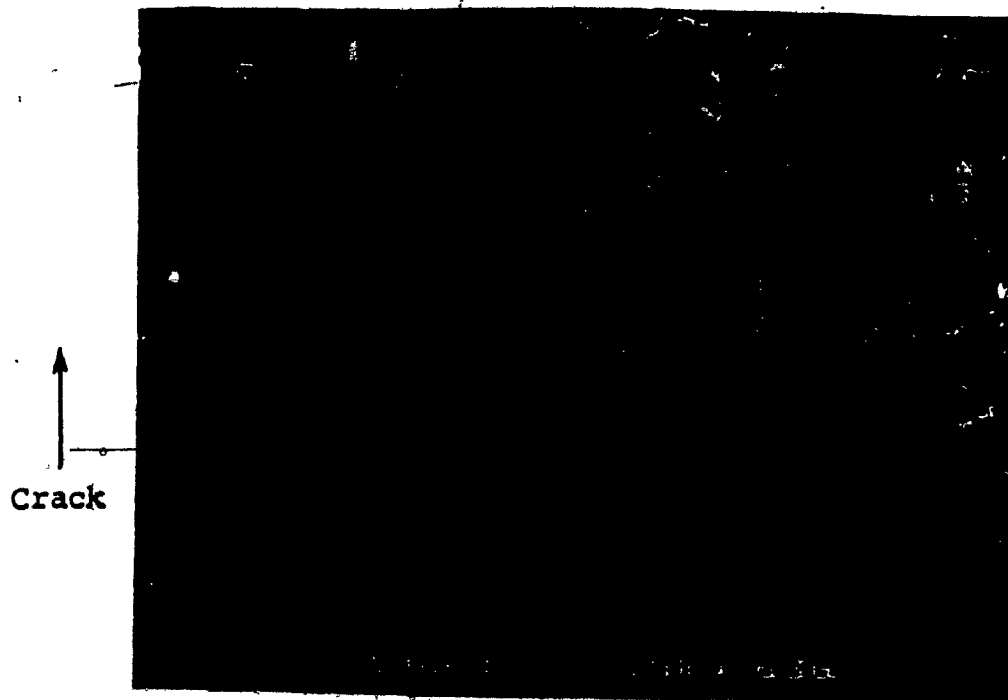


Figure 5.26 Scanning Electron Micrograph Showing Fracture Surface of a SF RTP - T Direction Test Piece.
(Fibers Preferentially Oriented Along the Crack Surface)

matrix drawing, fiber-matrix debonding and fiber pull-out. As can be seen, the fiber-matrix bonding is not very strong allowing some debonding to occur. This may, however, contribute positively to the fracture toughness through the requirement of additional energy to overcome interfacial friction during fiber pull-out as the crack propagates.

Figure 5.26 shows a scanning electron micrograph of the fracture surface of a SF RTP-T test piece with fibers oriented along the crack surface. While localized matrix drawing is still present, it is less than in the previous case. Also, very little fiber pull-out can occur, as most of the fibers are oriented in the plane of the crack surface. Consequently, the crack growth resistance, although better than that of the unreinforced resin, is not as good as when the fibers are oriented normal to the crack surface.

The differences in the crack initiation and propagation resistances of test pieces taken in the L and T direction clearly demonstrate the significant contribution of the debonding and pull-out processes on fracture toughness and hence the importance of fiber orientation. The shapes of the load-displacement curves obtained during the crack extension studies are sketched in Figure 5.27. As can be seen, the high density, unreinforced resin (PE 2906 M6) is reasonably linear up to the maximum load, due to the brittle nature of the failure. The non-linearity in the case of SF RTP test pieces is due to localized matrix yielding and

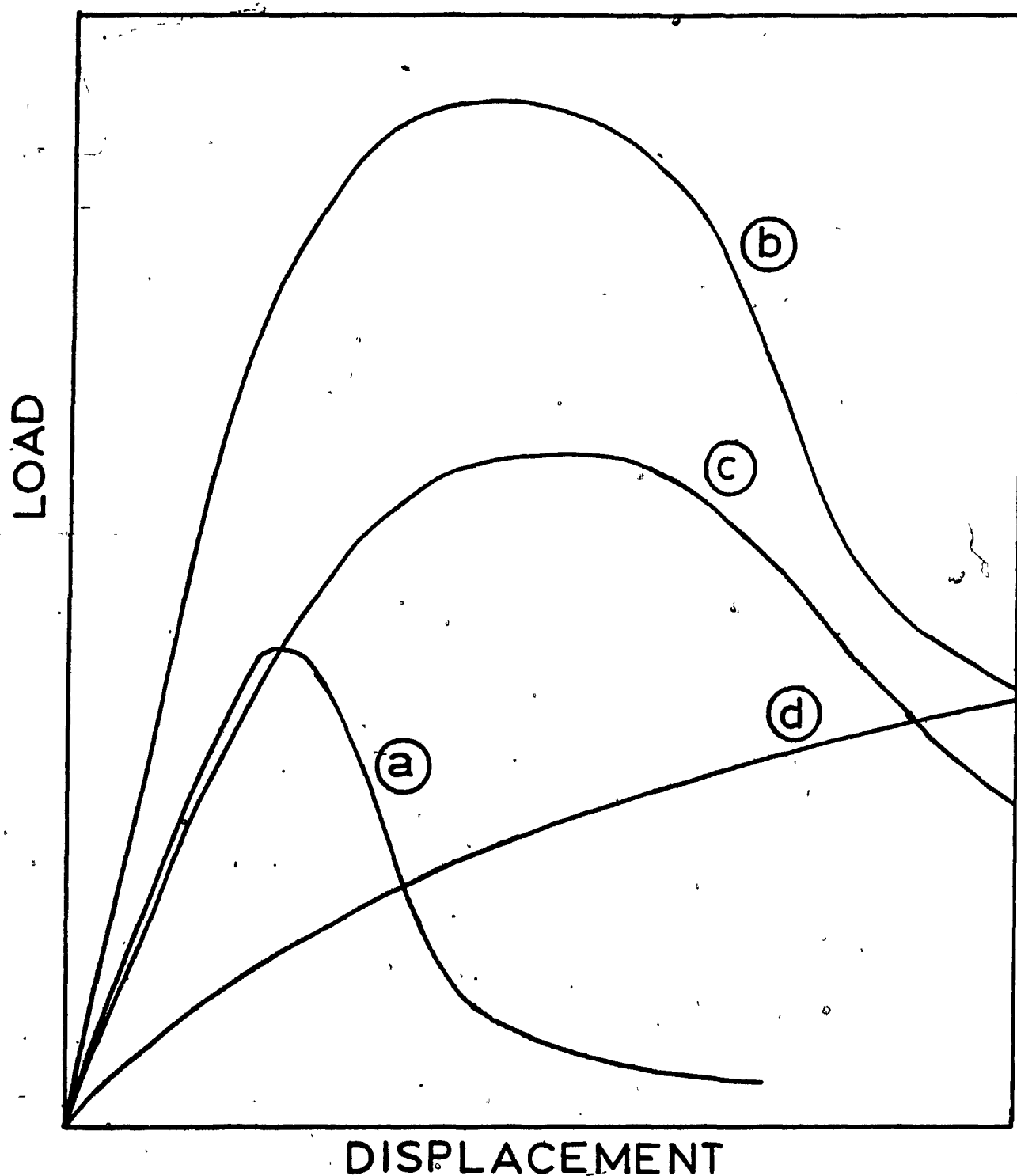


Figure 5.27 Typical Load versus Displacement Curves Obtained During Crack Propagation Studies.

- (a) High Density Unreinforced PE (PE 2906 M6)
- (b) SF RTP - L Direction Test Pieces
- (c) SF RTP - T Direction Test Pieces
- (d) Low Density Unreinforced PE (PE 74 A)

considerable crack stable crack extension prior to achieving the maximum load. The extensive nonlinearity in the case of the low density resin (PE 74 A) is due to the large amount of yielding at the crack tip and its consequent blunting.

For the sake of comparison, a micrograph of the fracture surface of a SF RTP-L test piece due to brittle failure after being frozen in liquid Nitrogen is shown in Figure 5.28. No localized matrix yielding is evident. The crack propagates by brittle cleavage of matrix and the breakage and pull-out of fibers close to the crack plane.

5.3.4.7 Crack Propagation in Multi-Layer Test Pieces

Coextruded SF RTP pipe has a three-layer wall structure. For such a pipe, it is interesting to study the crack growth characteristics, when a crack originating from a notch or defect in one layer, propagates toward the interface, and subsequently into the adjacent layer. The following exploratory study was carried out in order to simulate such a form of crack growth.

A rigorous analysis of stress distribution around a crack tip in a multi-layer test piece is extremely complex. The following study was hence carried out using certain nonconventional test procedures and empirical data treatment methods. The results provide an interesting comparison with the R curves of the individual materials reported above, and

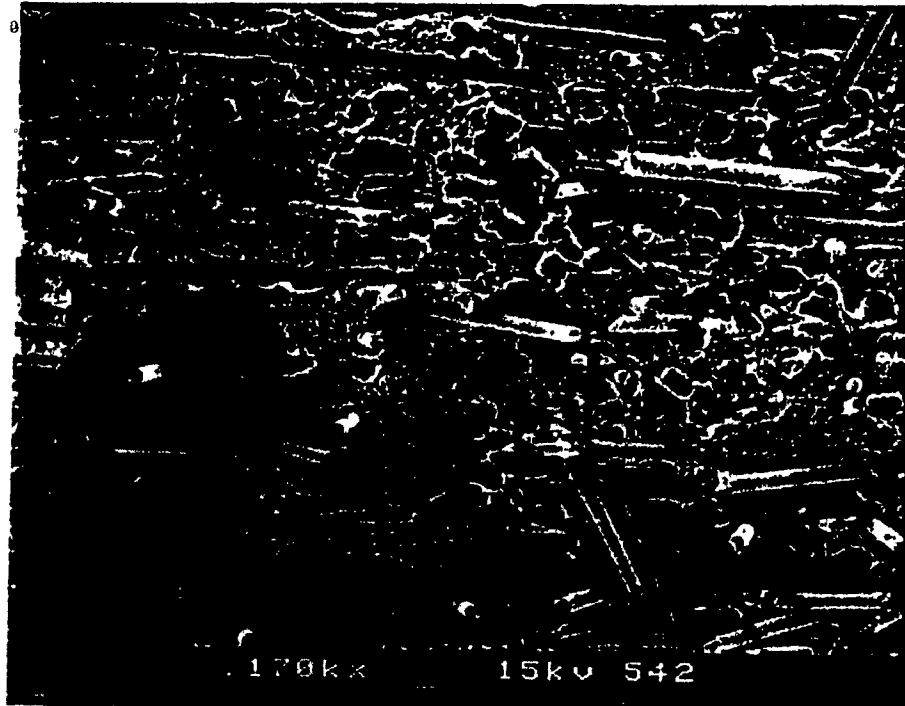


Figure 5.28 Scanning Electron Micrograph Showing Fracture Surface of a SF RTP - L Direction Test Piece after Freezing in Liquid Nitrogen.

qualitatively demonstrate special characteristics of coextruded pipe.

Two-layer plaques with one layer of high density, unreinforced resin (PE 2906 M6) and a second layer of SF RTP were prepared for these tests with different layer thickness ratios so as to obtain the same total nominal plaque thickness of 10 mm (0.4 in) by the compression molding procedure described in Section 5.3.2. Tests were limited to plaques with layers of the above two materials, as it was impossible to create brittle crack propagation in the low density, unreinforced resin (PE 74 A). Rectangular test pieces with dimensions of 75 x 10 (width D) x 6.3 mm (thickness B) (3 x 0.4 x 0.25 in) were machined from these plaques in both the L and the T directions (Figure 5.6), notched in the desired layer, and tested in the three point bending mode to generate the R curves. The following three important parameters were considered and their effects on the resulting R curves were studied.

- 1) Type of layer in which the crack originates,
- 2) relative thicknesses,
- 3) properties of the individual layers, particularly the elastic moduli, and hence fiber orientation in the SF RTP layer.

It may be noted that these R curves do not correspond to fundamental material properties, but represent experimental observations made using certain type of test pieces and test

conditions. However, they allow a qualitative comparison among themselves, and with the R curves of the individual materials obtained under similar conditions.

5.3.4.7.1 Experimental Procedure

The experimental procedure was similar to that followed to obtain the R curves of the individual materials as described in section 5.3.4.4.1 except for two points.

First, the initial crack depth was varied with the thickness of the layer in which the crack was introduced. The R curve for a homogeneous material, though expected to be independent of initial crack length in a certain range, may be affected by it when the initial crack length is very small. In the present case, the initial crack length was chosen to be equal to half the thickness of the layer in which the crack was introduced. The R curves were also generated using single-layer test pieces of the same geometry and initial crack length to facilitate comparison.

Secondly, a modification in the method used to calculate K_R was necessary. In the case of the single-layer test pieces, the effective crack length was estimated using a compliance-crack length relationship obtained by integration of Equation 5.12:

$$\frac{dC}{d(a/D)} = \frac{2}{E B} (S/D)^2 [f(a/D)]^2 \quad (5.12)$$

The elastic stiffness, E , of the ligament, of depth $(D-a)$ does not change during the crack growth process in a homogeneous test piece. However, this is not the case in a multi-layer test piece, because each individual layer makes a contribution to the overall stiffness depending on its thickness and, in the three point bending mode, on its location with respect to the neutral axis. Moreover, the effective stiffness of the ligament beyond the crack tip varies during crack extension. Consequently, Equation (5.12) cannot be used directly to obtain the compliance-crack length relationship.

As mentioned above, an exact analysis of crack extension in a multi-layer test piece can be extremely difficult, and does not seem to have been discussed in the literature. The problem was resolved here empirically by assuming that Equation (5.12) can be used for the multi-layer test pieces provided the stiffness E in this equation varies with the dimensionless crack length, (a/D) .

$$E = E(a/D) \quad (5.14)$$

Furthermore, it was assumed that this stiffness is equal to the equivalent flexural stiffness of the ligament of the test piece ahead of the crack tip. In other words, the stiffness E in Equation (5.12) was assumed to be that of a homogeneous material possessing the same flexural rigidity (product of the flexural stiffness and the second moment of inertia about the neutral axis) as the ligament beyond the crack tip. Under these

assumptions, Equation (5.12) can be integrated to obtain the following relationship.

$$CE_uB = C_0E_uB + 2 (S/D)^2 \int_{(a/D)_0}^{(a/D)} \frac{E_u}{E(a/D)} [f(a/D)]^2 d(a/D) \quad (5.15)$$

In this equation, E_u , the stiffness of the unreinforced resin is used for de-dimensionalization. The derivation of the equivalent flexural stiffness, $E(a/D)$, of the ligament as a function of the crack length, (a/D) , is given in Appendix 5.9. A BASIC program for computing $E(a/D)$ and CE_uB as functions of the crack length (a/D) is given in Appendix 5.7. Knowing $E(a/D)$, Equation (5.15) can be integrated numerically, and the previously described procedure can be used to estimate the effective crack length. The compliance (CE_uB) -crack length (a/D) curves for the various multi-layer test piece geometries used in the study are given in Appendix 5.10. As stated earlier, these curves should be transposed vertically so as to pass through the initial point $(C_0E_uB, (a/D)_0)$ determined from the initial slope of the test record and measured initial crack length.

5.3.4.8 Results and Discussion

a) Crack Growth from Unreinforced to SF RTP Layer

Crack propagation from unreinforced to SF RTP layer in a two-layer test piece simulates crack growth from the surface to the

core of a coextruded pipe wall. Two-layer plaques with ratios of SF RTP layer to the total thickness of 0.23 and 0.58 were prepared, and are referred as plaques of types I and II respectively. Test pieces were cut in the L and T directions. An initial notch was made in the unreinforced layer of each test piece to a depth equal to half the layer thickness. Thus it was possible to obtain some crack extension in the unreinforced layer before the crack reached the interface, and examine the effect of the presence of the stiffer second layer of SF RTP on the crack propagation resistance of the first unreinforced layer. The crack was subsequently allowed to propagate beyond the interface into the SF RTP layer, and complete R curves were obtained by the procedure described above.

The R curves for the two selected layer thickness ratios are shown in Figure 5.29a and b respectively for test pieces taken in the L direction of the plaques. The corresponding individual data points are given in Appendix 5.11. As seen from the figures, the portion of the R curves corresponding to crack propagation in the unreinforced layer first rises, and then reaches a plateau. In the region of the interface, however, the R curves start rising again in a very pronounced manner. The rise continues throughout the crack propagation in the SF RTP layer. For comparison, the R curves of the high density, unreinforced resin (PE 2906 M6) obtained using test pieces of the same geometry and initial crack length as the two-layer test pieces are also included in the figures.

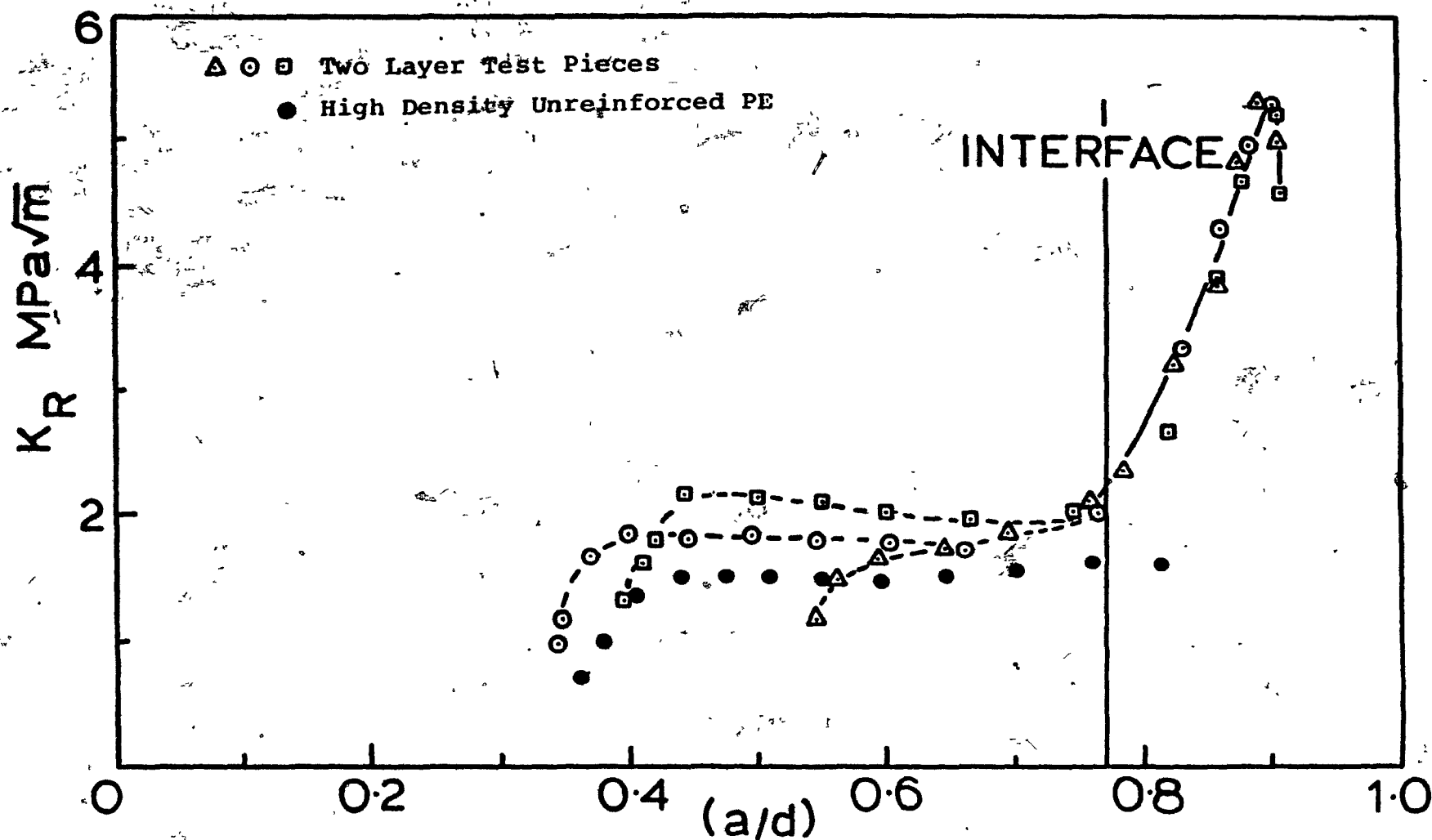


Figure 5.29a R Curve for Crack Propagation from Unreinforced to SF RTP Layer
 (Test Pieces in L Direction)
 Fractional Thickness of SF RTP Layer = 0.23

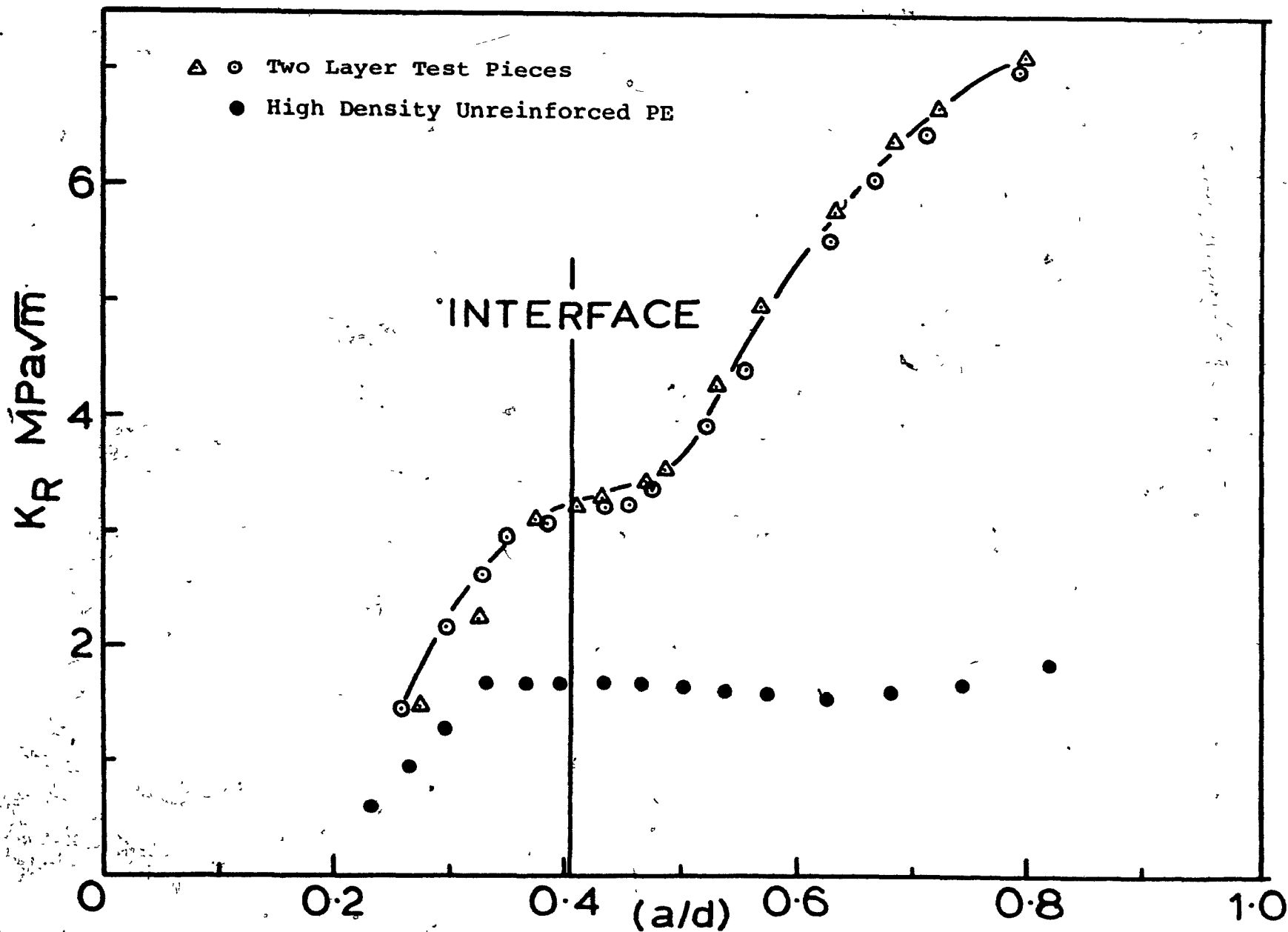


Figure 5.29b R Curve for Crack Propagation from Unreinforced to SF RTP Layer
(Test Pieces in L Direction)
Fractional Thickness of SF RTP Layer = 0.58

The plateau K_R values corresponding to crack extension in the unreinforced layers of the single and multi-layer test piece are listed in Table 5.3. As can be seen, in the case of test pieces with a thinner SF RTP layer, there is little effect of the presence of the second layer on the crack propagation resistance of the first layer. However, when the SF RTP layer is thicker, the plateau K_R value for the unreinforced layer is significantly higher. This seems to demonstrate the reinforcing effect of the stiffer second layer, and can be explained in terms of a critical crack mouth opening that is necessary for crack propagation to occur. When a stiffer SF RTP layer is present behind the unreinforced layer, it increases the overall stiffness (reduces the test piece compliance). Consequently, a higher stress intensity factor becomes necessary to cause a given amount of crack mouth opening and crack extension. The effect is obviously more pronounced for a thicker SF RTP layer. Such effects of changes in compliance on crack growth characteristics have also been described by Takemori and McGuire (139).

Once crack extension begins, the crack propagates in the unreinforced layer at a fairly constant value of stress intensity factor. Figure 5.30 shows sketches of typical load-displacement records for one and two-layer test pieces. As can be seen, after achieving the maximum value, the load rapidly drops indicating rapid crack propagation in the unreinforced layer of a two-layer test piece until the interface is reached. Since the crack growth resistance of the SF RTP layer is much higher, crack arrest can

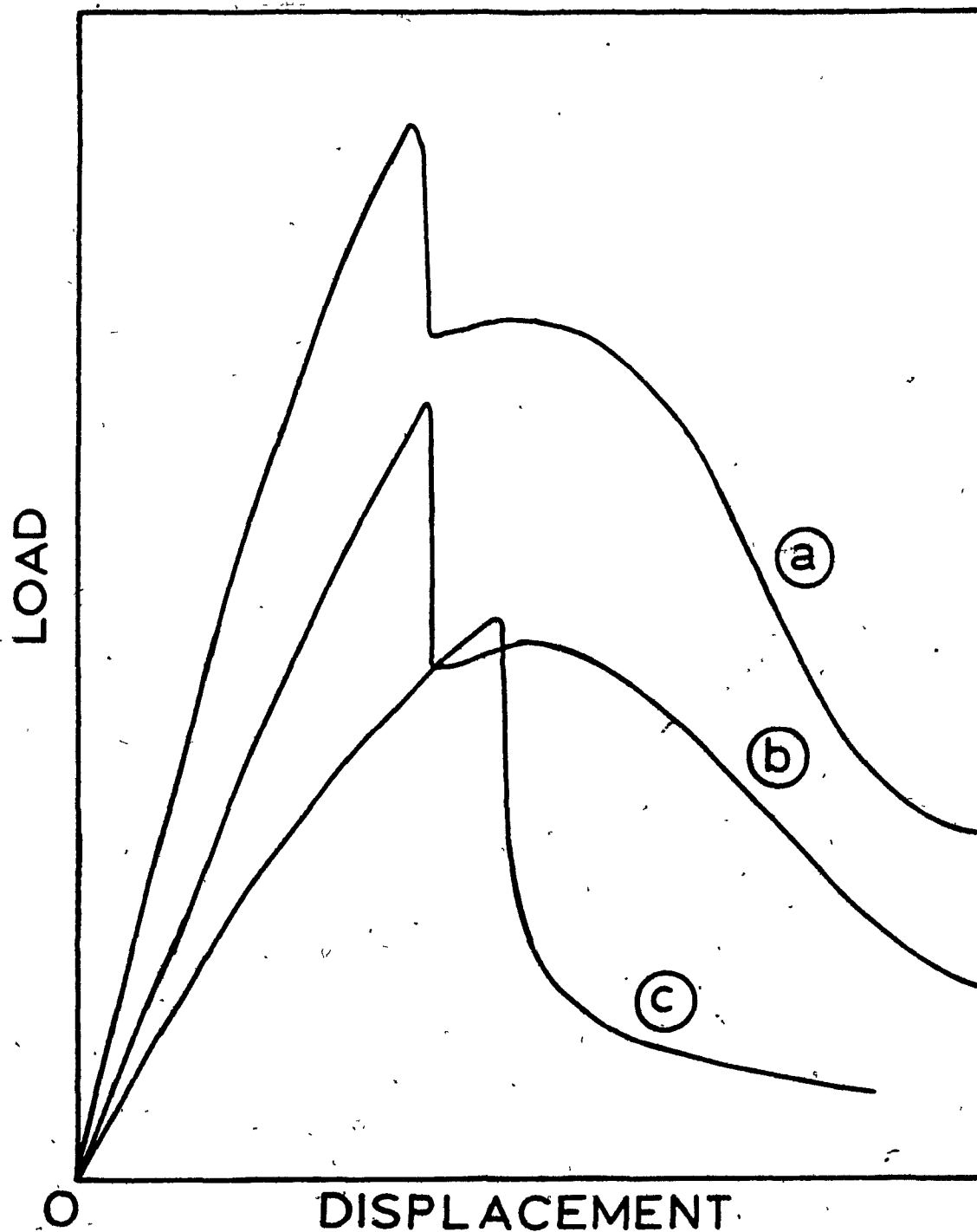


Figure 5.30 Typical Load versus Displacement Curves Obtained During Crack Propagation in Single and Two-Layer Test Pieces.

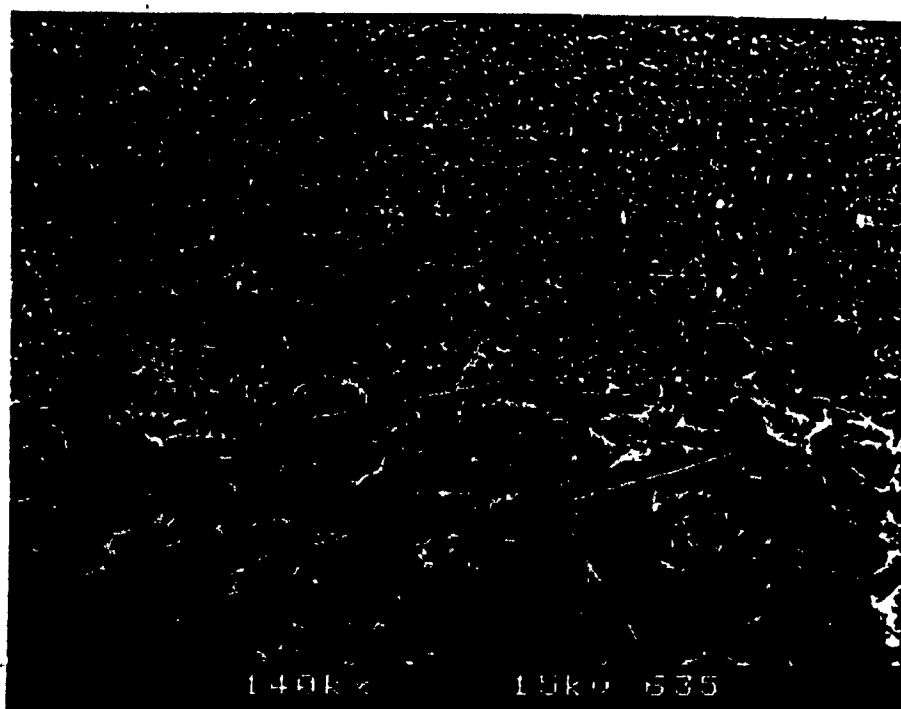
- (a) Crack Propagation from Unreinforced to Thicker SF RTP Layer
- (b) Crack Propagation from Unreinforced to Thinner SF RTP Layer
- (c) Crack Propagation in High Density Unreinforced Resin

occur at the interface unless the applied stress intensity factor is increased at this point. The subsequent R curve is equivalent to that for the single-layer test pieces of SF RTP.

Figure 5.31 shows a scanning electron micrograph of the fracture surface in the region of the interface of a two-layer test piece with fibers in the SF RTP layer oriented preferentially normal to the crack surface (L direction). The fracture surface in the unreinforced layer is more or less flat without any significant plastic deformation, but once the crack enters the SF RTP layer, a large amount of localized matrix drawing occurs as described earlier.

Similar results were also obtained when the two-layer test pieces cut in the T direction were tested to obtain R curves. For this purpose, plaques with SF RTP layer to total thickness ratios of 0.35 and 0.6 were prepared, and are referred to as plaques of types III and IV respectively. Test pieces were cut in the T direction so that fibers in the SF RTP layer were oriented in the crack plane in the thickness direction, i.e., parallel to the crack front (Figure 5.18c). The R curves for these cases are shown in Figure 5.32, while the individual data points are tabulated in Appendix 5.11. The plateau K_R values corresponding to crack propagation in the unreinforced layer are given in Table 5.3. The reinforcing effects of SF RTP layer, though still present, are less pronounced due to its lower stiffness. Again,

Crack



Unreinforced
Layer

SF RTP
Layer

Figure 5.31 Scanning Electron Micrograph Showing the Fracture Surface of a Two Layer Test Piece (Crack Propagation from Unreinforced to SF RTP Layer).

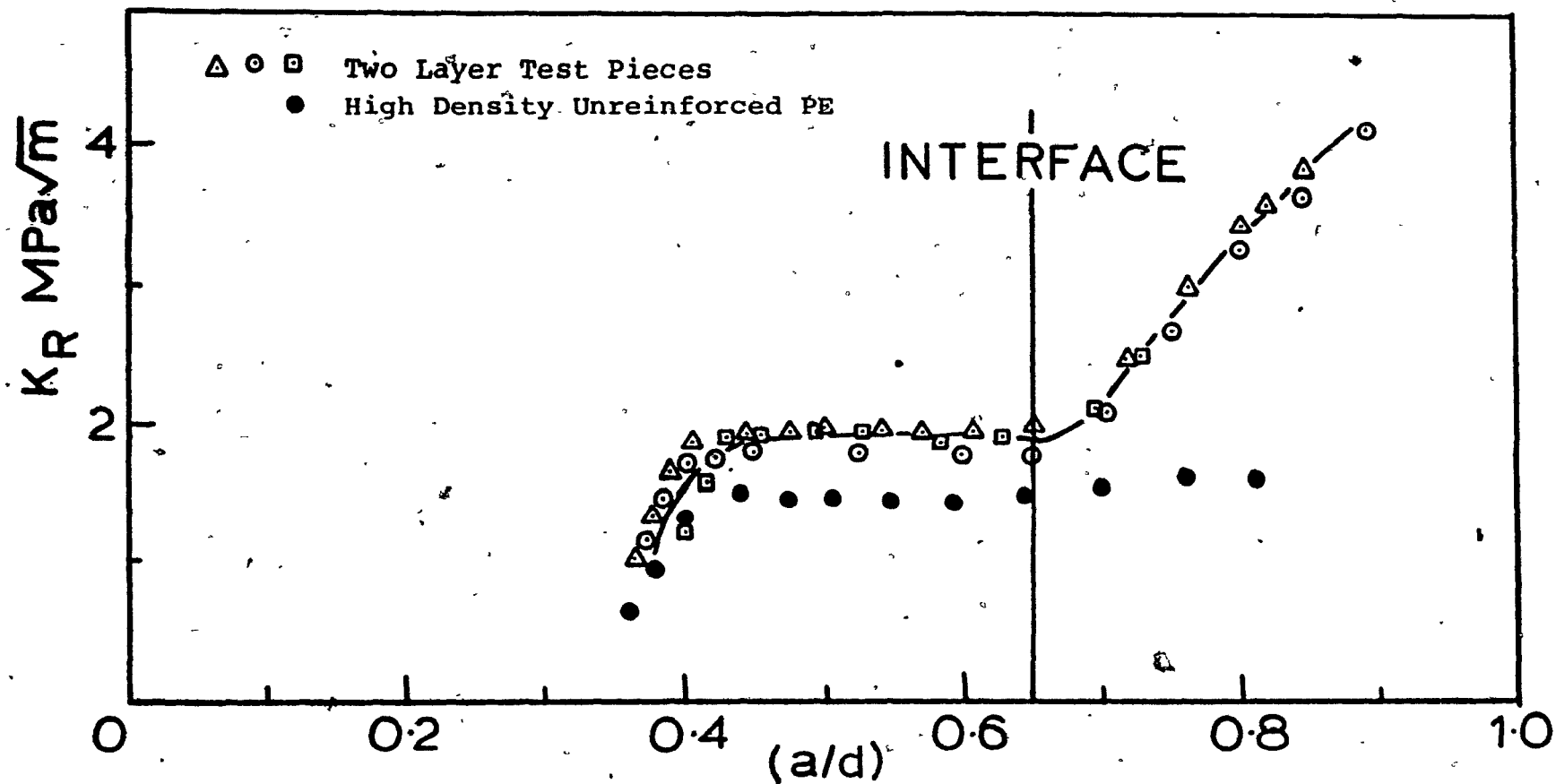


Figure 5.32a R Curve for Crack Propagation from Unreinforced to SF RTP Layer.
 (Test Pieces in T Direction)
 Fractional Thickness of SF RTP Layer = 0.35.

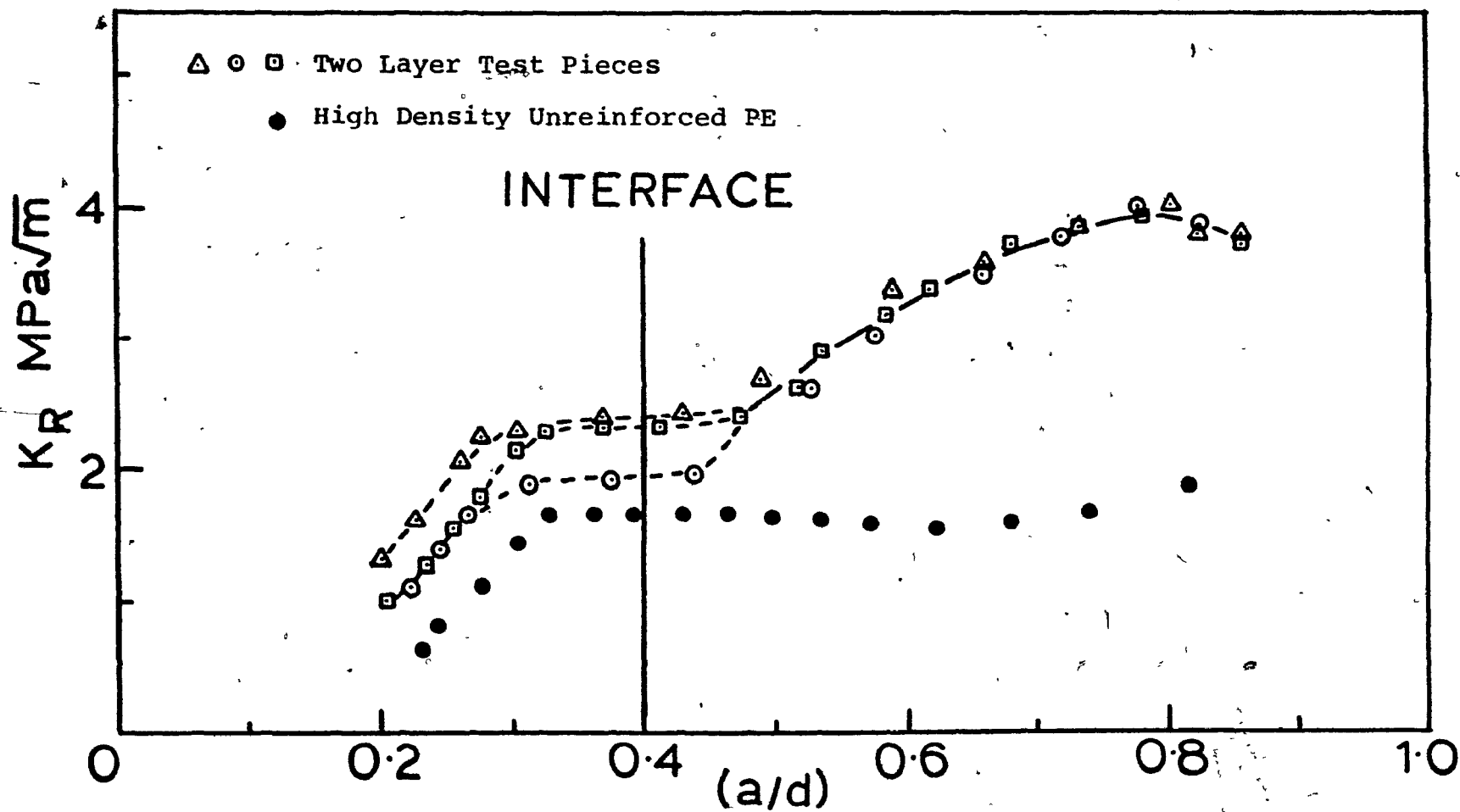


Figure 5.32b R Curve for Crack Propagation from Unreinforced to SF RTP Layer
(Test Pieces in T Direction)
Fractional Thickness of SF RTP Layer = 0.6

once the crack approaches the interface, the R curve starts rising due to the higher resistance of the SF RTP layer.

b) Crack Growth from SF RTP to Unreinforced Layer

It is also interesting to consider the case where a crack originating in the middle, SF RTP layer of coextruded pipe propagates toward the unreinforced surface layer. To study this case, two-layer plaques were prepared with ratios of SF RTP layer to total thickness of 0.61 and 0.69. Test pieces were machined in the L and T directions from these plaques. The initial notch was made in the SF RTP layer of each test piece to a depth equal to half its thickness, and crack propagation studies were carried out.

The R curves for the L and T test pieces are presented in Figure 5.33a and b, while the individual data points are tabulated in Appendix 5.12. The presence of the unreinforced layer behind the SF RTP layer is seen to have a detrimental effect on the overall resistance to crack propagation. Due to its lower stiffness, the unreinforced layer increases overall compliance, and hence allows a greater amount of crack mouth opening for a given stress intensity factor. As a result, crack extension occurs at a much lower value of the applied stress intensity factor. Test pieces in this case thus exhibit a poor crack propagation resistance. Also, once the crack reaches the interface, the K_{Rc} value starts dropping drastically due to the

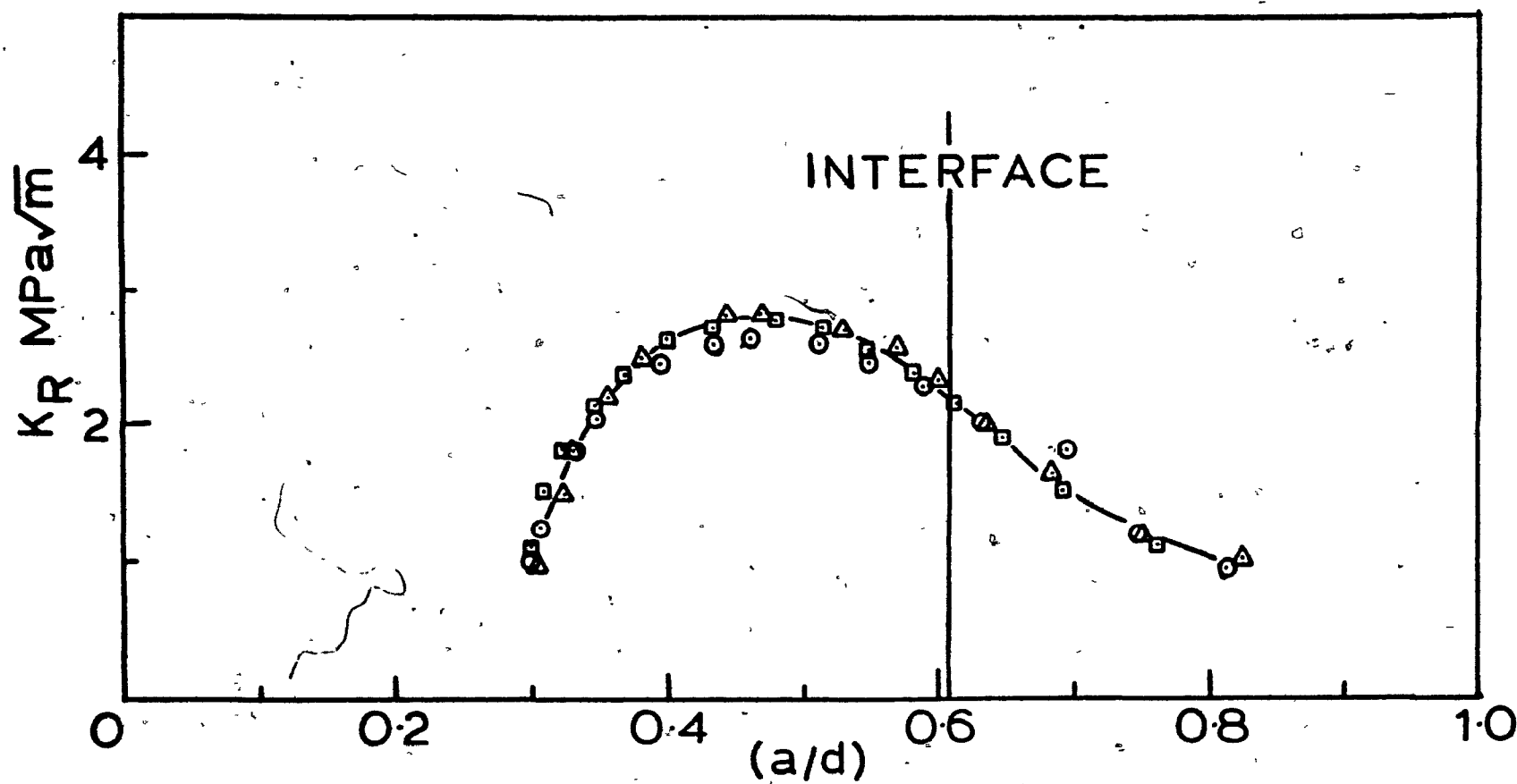


Figure 5.33a R Curve for Crack Propagation from SF RTP to the Unreinforced Layer
(L Direction Test Pieces).
Fractional Thickness of SF RTP Layer = 0.61.

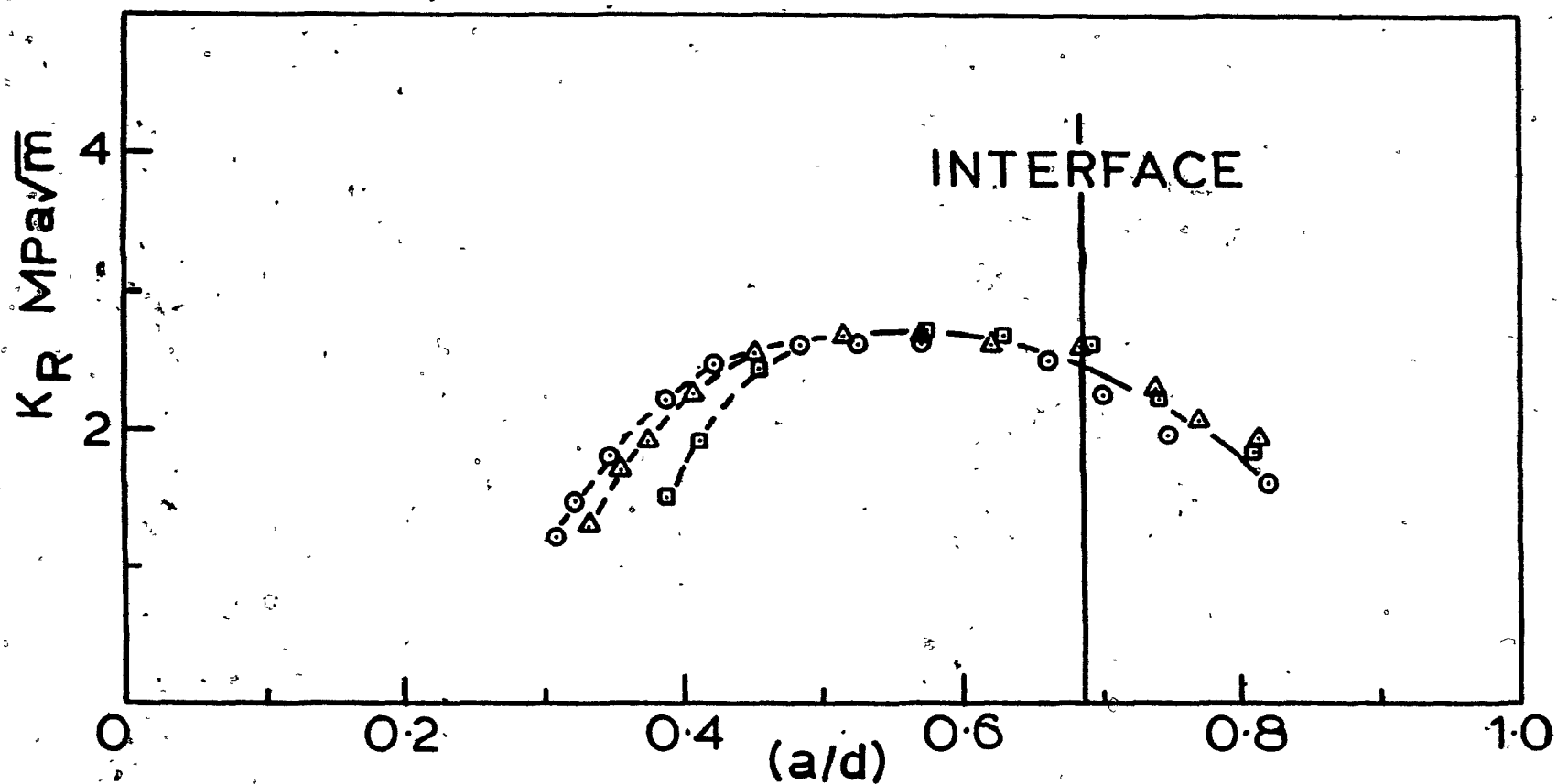


Figure 5.33b R Curve for Crack Propagation from SF RTP to the Unreinforced Layer
(T Direction Test Pieces).
Fractional Thickness of SF RTP Layer = 0.69.

poor crack propagation resistance of the high density, unreinforced resin.

Thus, if the crack originates in the stiffer SF RTP layer of the coextruded pipe wall, and propagates toward the unreinforced surface layer, a poor crack growth resistance may result. In the above experiments, the unreinforced layer of high density resin has a very poor crack growth resistance. However, as suggested earlier, if the surface layers consist of a tough material, the crack may actually be arrested at the interface. In some exploratory demonstration experiments, an attempt to generate such a form of crack growth was made using test pieces that had a two-layer structure of SF RTP and low density, unreinforced resin (PE 74 A). The crack extension proceeded from the SF RTP layer toward the interface. The load-displacement record is shown schematically in Figure 5.34. Crack extension proceeded up to the interface, but no further crack extension was possible due to the notch insensitivity of the unreinforced layer of low density resin. In this case, the interface itself formed the weakest link and subsequent loading merely caused layer debonding.

It must be remembered that the above results were obtained in the slow crack propagation mode, where crack growth is more or less stable. However, during dynamic crack propagation, a material's response can be significantly different. In addition, the crack speed at the interface may influence its extension across the interface (140-142). Nevertheless, the layer debonding

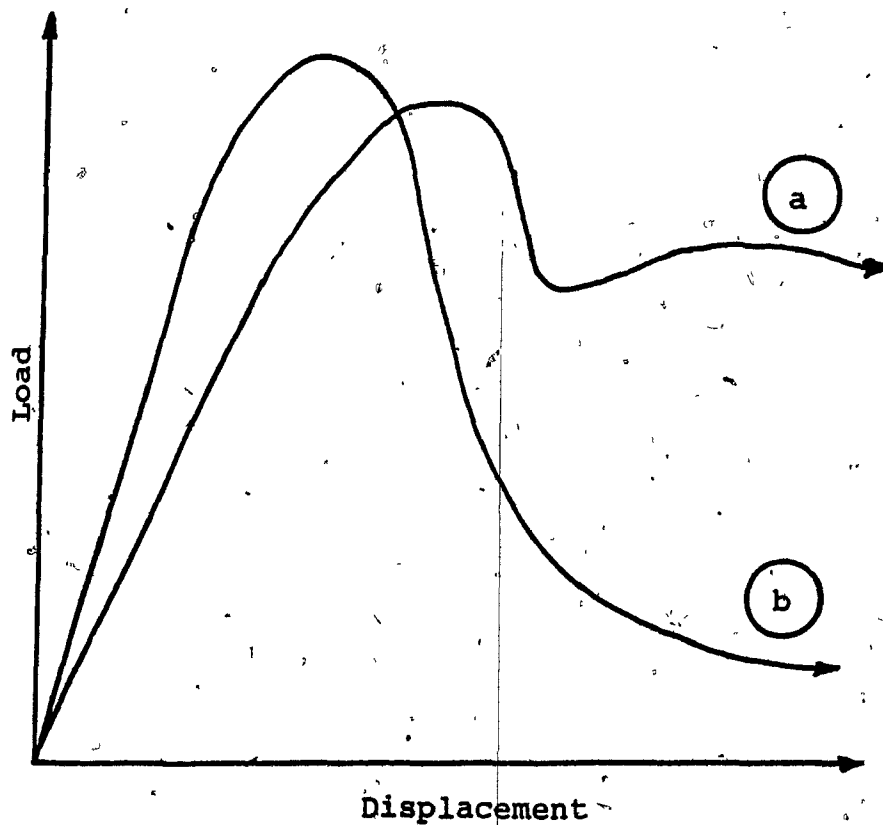


Figure 5.34 Load versus Displacement Curves for Crack Propagation from SF RTP to Unreinforced Layer (Schematic).

- (a) Unreinforced Layer of Low Density PE (PE 74 A)
- (b) Unreinforced Layer of High Density PE (PE 2906M)

mentioned above seems to be a highly energy consuming process, as seen from Figure 5.34; a high level of load is sustained due to the overall bending of the test piece accompanied by the layer debonding after the crack reaches the interface. It is not obvious without further study whether such a mode of failure (through layer debonding) can deter through-thickness failure resulting from crack growth across a coextruded pipe wall.

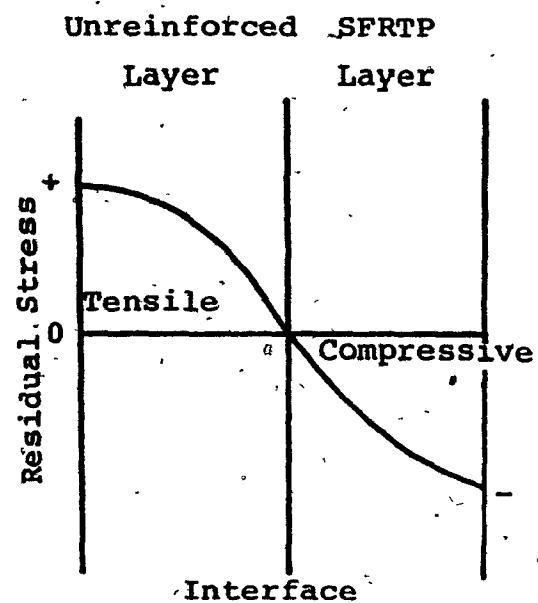
5.3.4.9 Physical Factors Affecting the R Curves

It should be noted that a variety of physical factors can affect the results reported above. Two important factors are interlayer bonding strength and residual stresses present in the test piece.

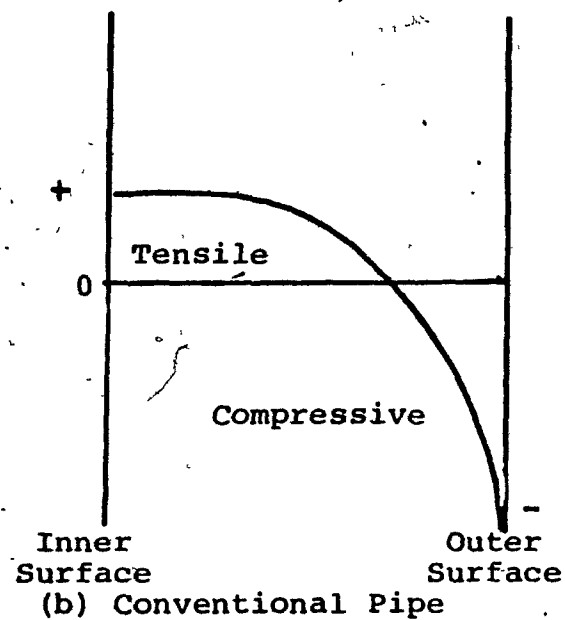
Good interlayer bonding is extremely important if the crack is to propagate from one layer to the next. The strain in the three point bending mode varies from zero at the neutral axis to a maximum at the surface. Because of the change in stiffness at the interface, the stresses in the two layers, however, are not the same. This introduces a shear stress at the interface. For crack propagation to occur from one layer to the next, the interface bonding should be strong enough to overcome the shear stress, and establish a continuity for crack propagation from one layer to the next. Even when the interlayer bonding is good, crack propagation across the interface may not occur if the second layer is extremely tough as seen above. The plaques for

the above tests were prepared by slow cooling in air from the molding temperature, and this procedure provided satisfactory interlayer bonding with good reproducibility.

The residual stress build-up in the plaques due to different crystallization and thermal contraction characteristics of the individual layers is another important factor. When a two-layer plaque solidifies, the unreinforced layer tends to contract more, since the contraction in the SF RTP layer is limited by the presence of glass fibers. This is clearly shown by the difference in the heat of melting of the two materials as measured by differential scanning calorimetry (DSC) (Appendix 5.13). The heat of melting of the high density, unreinforced resin is 51.7 cal/gm, while that of SF RTP is 35.5 cal/gm, i.e., about 30% lower. The differential contraction of the two layers can give rise to residual stresses in the plaque, introducing tensile stresses in the unreinforced layer and compressive stresses in the SF RTP layer, as shown schematically in Figure 5.35a. The slow cooling process used to obtain good interlayer bonding in the multi-layer plaques may actually promote the build-up of residual stresses by promoting crystallization. The results reported above may have been influenced to a certain extent by such stresses. However, due to the necessity of using certain larger thicknesses, the limitation on the total plaque thickness that can be molded, and the inherent nature of the differential contraction, it was virtually impossible to prepare completely stress-free plaques. The above results can, however,



(a) Two Layer Plaque



(b) Conventional Pipe

Figure 5.35 Residual Stresses in
(a) Two Layer Plaque and (b) Conventional Plastic Pipe

be viewed as conservative estimates in the case of crack growth from the unreinforced to the SF RTP layer, because the effect of residual stresses, if any, would have been to reduce the crack propagation resistance by superimposing the applied tensile stress.

Residual stresses are also unavoidable in actual pipe, and are generally considered undesirable, although their effect on pipe performance is not well understood (143-145). It is of interest to consider the build-up of residual stresses in coextruded pipe as against conventional plastic pipe. The extruded pipe is usually cooled in a water bath such that solidification occurs from the outer surface inward, although other methods have been suggested to control the build-up of residual stresses in pipe (146). In the conventional cooling method, the outer layer solidifies quickly, while the material, inside crystallizes and cools over a longer period of time. The associated contraction upon solidification causes a build-up of residual stresses that are compressive near the outer surface and tensile near the inner surface, as shown schematically in Figure 5.35b (143-145). In coextruded pipe, since the middle layer is of SF RTP that undergoes a lesser degree of contraction upon solidification, it may help reduce the residual stresses near the outer surface. Moreover, in such a pipe, it appears desirable to use a tough, low density resin for the surface layers. Such a resin usually has a lower degree of crystallinity, and hence may further help reduce the build-up of residual stresses.

Admittedly, these ideas are speculative, and more research will be necessary before concrete conclusions can be drawn.

5.4 Summary

In this chapter, results of mechanical tests carried out to explore possible advantages and disadvantages of the three-layer structure of coextruded pipe have been discussed. It is seen that stiffness, strength and ductility of coextruded pipe are significantly affected by the relative layer thicknesses and fiber orientation in the SFRTF layer. Considerable design flexibility can hence be achieved not only through choice of materials, but also through control of layer thicknesses and fiber orientation during extrusion.

Crack initiation and propagation studies showed that the presence of fibers can actually change the mode of failure in the matrix. The high density, unreinforced resin (PE 2906 M6) has a very poor crack growth resistance on its own, and fails in a brittle manner in the presence of a sharp notch. Its J_{IC} value is, however, significantly improved by fibers oriented normal to the crack surface due to significant localized matrix drawing, fiber-matrix debonding and fiber pullout. Fiber orientation in the plane of the crack surface, however, leads to poor crack initiation resistance. For this reason, it may be desirable to obtain a balanced orientation of fibers between the axial and hoop directions of the pipe, although the stiffness and strength

properties suggest otherwise. The low density, unreinforced resin (PE 74 A) exhibits an exceptional fracture toughness by yielding at the crack tip, and thus blunting it.

The presence of fibers also leads to a significant improvement in crack propagation resistance giving rise to a monotonically rising R curve. Again, the improvement is more pronounced when fibers are oriented normal to the crack surface than when they are oriented along the crack surface. Exploratory studies of crack propagation in two-layer test pieces shows that the crack propagation characteristics of the unreinforced layer are strongly influenced by the presence of the second layer. The stiffer layer of SF RTP improves the crack growth resistance of the first layer. The extent of this improvement is dependent on the stiffness of the SF RTP layer. The presence of a second layer of unreinforced material having a lower stiffness, on the other hand, drastically reduces the crack growth resistance of the first SF RTP layer.

It should be stressed that since the materials used in the above study are not pipe-grade resins, they may not represent suitable candidates for making a commercial coextruded pipe. This is particularly true for the high density, unreinforced resin (PE 2906 M6), which is an injection molding grade resin. However, its use in generating the above results has made it possible to demonstrate the interesting reinforcing effects of the middle SF RTP layer not only on the overall stiffness and strength, but

also on the resistance to crack propagation in the surface layers. Ideally, it would be desirable to have an unreinforced surface layer of a highly tough and ductile resin such as the low density resin (PE 74 A) used in the study. Surface layers of such a resin, when reinforced by the middle layer of SF RTP, can, in principle, lead to significant improvements in the resistance to surface cracks without compromise of overall stiffness and strength properties. The above results also clearly demonstrate the importance of controlling the fiber orientation in the SF RTP layer and the layer thickness distribution.

As mentioned earlier, the above study should not be considered to be a simulation of the field performance of coextruded pipe. Moreover, much controversy exists as to which properties are of prime importance in determining the field performance of plastic pipe. Improvements in the hoop stiffness, strength and resistance to crack growth are usually considered desirable, and as discussed above, the coextrusion process can offer a suitable practical way to achieve them. Within the scope of this project, it was not possible to carry out conventional pipe tests such as those described in Appendices 5.1 and 5.2. A study involving these types of tests could help evaluate the field performance of the pipe, and could allow more concrete conclusions to be drawn.

CONCLUSIONS AND RECOMMENDATIONS

A new process for the manufacture of short fiber-reinforced pipe featuring coextruded layers of reinforced and unreinforced thermoplastic resins has been developed. The process not only provides a smoother surface finish than achievable by simple extrusion of SF RTP, but also leads to significant processing advantages in terms of the control of fiber orientation and resulting improvements in mechanical properties of importance for plastic pipe. Specific conclusions of the research are summarized below.

6.1 Conclusions

- (1) Conventional pipe extrusion dies featuring axially converging geometry lead to preferential axial orientation of fibers, and are thus not desirable for the extrusion of plastic pipe incorporating a short fiber reinforcement. An expanding channel die featuring an increase in the perimeter along the flow direction, and consequently causing hoop stretching of the melt, can be used to promote hoop orientation of fibers in extruded SF RTP pipe.

- (2) The flow through an expanding channel die involves a superposition of a shear component in the flow direction and an extensional component in the hoop direction. The shear component tends to orient the fibers in the flow direction, while the extensional component tends to orient them in the hoop direction. A theoretical analysis based on Jeffrey's equations shows that, as a result, there is a change in the fiber orientation pattern across the extrudate thickness. In the core region away from the walls, the orientation is preferential in the hoop direction, while near the wall, it becomes preferential in the axial direction. These results are in qualitative agreement with experimental findings.
- (3) The theoretical analysis also shows that in simple extrusion through an expanding channel die, the total strain resulting from the flow, rather than the flow rate or the rate of strain, governs the final fiber orientation distribution at the die exit. The experimental results show no systematic dependence of orientation on the flow rate over two decades of the rate of extrusion studied.
- (4) The theoretical analysis suggests that the rheological characteristics, particularly the shear-thinning behavior, and geometry of the die can significantly influence the fiber orientation distribution in simple extrusion using the expanding channel die. A strongly shear-thinning behavior.

(lower power law index) leads to an increased degree of hoop orientation. A larger angle of expansion of the die, a higher degree of expansion, or a thicker channel gap can also increase the degree of hoop orientation of fibers within a certain range.

- (5) The process of coextrusion also allows the SF RTP melt layer to be kept away from the region of high shear rate near the wall. The theoretical analysis of coextrusion shows that such a flow pattern reduces the undesirable effect of shear on fiber orientation.
- (6) By selection of SF RTP and unreinforced resins with appropriate rheological characteristics, the flow of the SF RTP layer through the die can be "lubricated", making the velocity profile in that layer considerably flatter which promotes the desired hoop orientation of fibers. The theoretical analysis provides guidelines as to the processing conditions, rheological characteristics of the resins, and die geometry that allow improvement and control of this lubrication effect.
- (7) A coextrusion die featuring channels for the distribution of various melt streams to achieve the desired three-layer annular flow system as well as an expanding geometry near the exit was designed and constructed. The die was used in a

pipe coextrusion line to produce coextruded pipe using one SF RTP and two different unreinforced resins. For the selected resins, die design and processing conditions, a fairly balanced fiber orientation between the axial and hoop directions was obtained despite a possible reduction of hoop orientation during the post processing stage of cooling and take-off.

- (8) Studies related to the mechanical properties of coextruded pipe suggest that the three-layer structure offers the possibility of optimizing mechanical properties such as stiffness and strength by varying the layer thickness distribution and controlling the fiber orientation in the middle SF RTP layer.
- (9) Resistance to slow crack growth leading to brittle failure has been identified to be of particular importance in the long term performance of plastic pipe. Crack initiation and propagation studies of SF RTP and the selected high and low density unreinforced resins shows that the low density resin with low stiffness and yield strength exhibits excellent resistance to crack growth by yielding at the crack tip and blunting it. The high density resin with high stiffness and strength, on the other hand, exhibits a poor crack propagation resistance, although it shows general yielding in a tension test in the absence of a notch. Its resistance to crack propagation is improved somewhat by the presence of

reinforcing fibers due to the localized matrix yielding in the vicinity of individual fibers. The improvement is greater when they are oriented normal to the crack plane than when the fibers are oriented along the crack plane.

- (10) A study of crack propagation in multi-layer test pieces simulating the wall of a coextruded pipe shows that the layered structure of the coextruded pipe can, in principle, provide improved resistance to cracks originating at the surface. The presence of a reinforced layer can provide an increase in the overall stiffness, while the presence of the unreinforced surface layer of a suitably chosen resin can provide ductility in the vicinity of the surface where, cracks often originate. Both factors can lead to an improvement in the overall resistance to crack growth.

6.2 Recommendations for Further Work

In this study, the principles of controlling the fiber orientation pattern, and achieving improvements in the mechanical properties of the coextruded pipe have been demonstrated. The stiffness and strength properties of such a pipe are, in general, expected to be between those of conventional unreinforced plastic pipe and pipe reinforced by continuous filament winding. Such properties along with economical considerations will largely determine its potential field applications.

- The resins used in the study were not pipe-grade materials

for reasons given in Chapter 2. Further study using pipe-grade resins would allow a more direct comparison of the properties of coextruded pipe with those of commercial plastic pipe made by conventional pipe extrusion.

The theoretical analysis suggests that rheological properties and processing parameters can significantly influence the fiber orientation distribution in coextruded pipe. An experimental study using resins with specific rheological characteristics selected in order to maximize the above described lubrication effect will be helpful in getting further insight into the process of fiber orientation and its control.

The pipe produced in this research had a relatively thin wall for reasons of material economy and ease of calibration and cooling. Because of the limited materials, equipment and manpower resources, coextruded pipe of suitable quality and dimensions that could be subjected to the standard performance tests described in Appendices 5.1 and 5.2 could not be produced. The production of coextruded pipe with appropriate wall thickness and thickness distribution would allow rigorous testing by standard methods and the evaluation of its performance.

The pipe produced in this research had a nominal diameter of 50 mm (2 in). It is expected that the property improvements made possible by the coextrusion process would be economically more attractive in the larger diameter range (100 mm and larger). A

die for producing pipe with a larger diameter can also incorporate a greater degree of expansion and a thicker channel. As suggested by the theoretical analysis, both factors can further contribute to improving the degree of hoop orientation. A study of the processing and properties of the larger diameter coextruded pipe would be useful in assessing the technical and economical feasibility of the process. This would, however, require the use of large size extruders, and involve consumption of large quantity of resins and, hence, could be carried out suitably in an industrial environment.

As suggested by the results of Chapter 5, the properties of the multi-layer wall structure can, in principle, provide an improvement in the resistance to advance of cracks originating at the surface. On the other hand, reinforcing fibers in the SFRTF layer can themselves provide sites for stress concentration leading to crack initiation in the middle layer. In addition, several modes of failure are possible for coextruded pipe, such as through-thickness crack propagation and layer debonding. An interfacial crack arrest may also occur. An extensive study involving various modes of crack initiation, propagation and failure over long periods of time would be very useful in assessing the service performance of coextruded pipe. A study involving the optimization of properties for specific pipe applications by variation of the wall structure (resin selection, layer thickness distribution and fiber orientation distribution) would also be desirable.

An important advantage of plastic pipe is the ease of joining. Sections of polyethylene pipe and couplings are commonly joined by a variety of fusion techniques, although mechanical methods of joining can also be used as described in Appendix 6.1. These techniques, however, may require some adaptation in the case of coextruded pipe. The fused section has been known to be a potential site for initiation of failure (151), and is thus of critical importance in the performance of the piping system. A study exploring various possible methods of joining coextruded pipe, and the long term performance of joined sections would therefore be very useful.

Finally, this research has been limited to the use of short glass fibers as reinforcement. Several other particulate reinforcing agents can also be used for economy or to achieve specific pipe properties. A potentially attractive reinforcing agent is discontinuous carbon fibers. Carbon fibers, although relatively expensive, can not only offer attractive composite properties but can also impart partial conductivity to the pipe, an aspect extremely useful in tracing underground pipe and avoiding static build-up. The process of coextrusion can be extended to incorporate this and other suitable reinforcing agents.

6.3 Original Contributions

- (1) A novel process for the manufacture of a new type of short fiber-reinforced coextruded plastic pipe has been developed. While SF RTP composites have been used widely to make molded articles, this project establishes that their use can be extended to the extrusion of pipe.
- (2) A method of controlling fiber orientation in coextruded pipe has been developed. The method relies on a combination of expanding die geometry and the lubrication effect associated with the coextrusion process.
- (3) A theoretical analysis based on Jeffrey's equation has been developed to predict the fiber orientation distribution as a result of simple extrusion or coextrusion involving a flow with superimposed shear and extensional flow components for a geometry not studied previously. The analysis provides qualitatively correct predictions and guidelines for processing conditions, resin rheology and die geometry for the control of the structure of the coextruded pipe wall.
- (4) A special coextrusion die was designed to produce the desired coextruded pipe. Using the die, coextruded pipe has been successfully produced under various conditions of operation.
- (5) Crack initiation and crack propagation characteristics of SF RTP and unreinforced polyethylene resins have been studied and the results have been interpreted in terms of fractographic observations. In particular, a method of

analyzing experimental results of crack propagation in little studied multi-layer inhomogeneous test pieces has been developed and used to characterize the crack propagation resistance of the multi-layer structure of the coextruded pipe.

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Appendix 2.1

Viscosity - Shear Rate - Shear Stress Data

a) SF RTP System

Wall Shear Stress (τ_w) N/m ² x 10 ⁴	Apparent Wall Shear Rate ($\dot{\gamma}_A$) s ⁻¹	True Wall Shear Rate ($\dot{\gamma}_W$) s ⁻¹	Viscosity $\eta(\dot{\gamma})$ Pa.s
6.39	13.67	20.61	3103
8.10	27.34	41.23	1965
10.22	54.68	82.46	1240
13.38	136.70	206.15	649
18.04	273.40	412.29	437
23.19	546.80	824.58	281
27.39	1367.00	2061.45	133

Power-law Correlation : $\tau = 2.37 \text{ E}+4 \dot{\gamma}^{.33}$

b) High Density Unreinforced Resin (PE 2906 M6)

Wall Shear Stress (τ_w) N/m ² x 10 ⁴	Apparent Wall Shear Rate ($\dot{\gamma}_A$) s ⁻¹	True Wall Shear Rate ($\dot{\gamma}_W$) s ⁻¹	Viscosity $\eta(\dot{\gamma})$ Pa.s
4.06	13.67	19.25	2111
5.46	27.34	38.5	1417
7.14	54.68	77.01	927
10.11	136.7	192.53	525
12.97	273.4	385.06	337
16.97	546.8	770.13	220
23.50	1367.0	1925.32	122

Power-law Correlation : $\tau = 1.35 \text{ E}+4 \dot{\gamma}^{.38}$

c) Low Density Unreinforced Resin (PE 74 A)

Wall Shear Stress (τ_w) N/m ² x 10 ⁴	Apparent Wall Shear Rate ($\dot{\gamma}_A$) s ⁻¹	True Wall Shear Rate ($\dot{\gamma}_W$) s ⁻¹	Viscosity $\eta(\dot{\gamma})$ Pa.s
4.10	13.67	18.21	2252
5.69	27.34	36.42	1561
7.79	54.68	72.85	1070
12.05	136.7	182.12	662
16.47	273.4	364.24	452
20.95	546.8	728.48	288
28.37	1367.0	1821.20	156

Power - law Correlation : $\tau = 1.23E+4 \dot{\gamma}^{.43}$

Appendix 2.2

Molecular Weight Distributions

Molecular Weight	Cumulative Weight %	
	Matrix Resin of SFRTF	High Density Unreinforced Resin (PE 2906 M6)
1,000,000	2.62	3.34
700,000	4.0	5.22
500,000	5.64	7.40
300,000	9.41	11.75
100,000	25.75	25.64
10,000	86.53	80.59
5,000	93.34	90.03
2,000	98.41	97.50

Appendix 3.1

Assumptions Made in the Development of the Theoretical Model for Predicting Fiber Orientation Distribution

Several assumptions are made in the development of the theoretical model for predicting the fiber orientation distribution resulting from flow through an expanding channel die. While the justification for some of these assumptions is obvious, an explanation of the others is presented below.

An important assumption in the model development is the neglect of fiber-fiber and fiber-wall interactions. In a concentrated suspension, such as SFRTF, these interactions can significantly influence the fiber motion and the final orientation distribution. However, these interactions are very difficult to describe quantitatively, and their consideration leads to mathematical complexities. The assumption should thus be viewed as a simplifying one.

Another important assumption deals with the rheological properties of the suspension. While the suspension is assumed to follow power-law behavior on a global scale, in the vicinity of a fiber it is assumed to be Newtonian. In a realistic situation, the ratio of a characteristic dimension of the flow channel to that of the fiber can be of the order of 100 or more. This suggests that the variation in strain rate, and thus the strain

rate dependent properties, is insignificant on a local scale defined by the fiber dimension, as compared to that on a global scale defined by the flow channel dimensions. The power-law assumption also ignores the viscoelastic nature of the fluid. The inclusion of viscoelasticity, through the use of a complex constitutive equation, leads to major mathematical complexities and was hence avoided.

Another assumption concerns the derivation of the velocity profile. In the expanding channel die, the velocity u_z varies both across the channel thickness (direction y) and along the flow direction (direction z). Consequently, both the components of the rate of deformation tensor $(\partial u_z / \partial y)$ and $(\partial u_z / \partial z)$ are non-zero. In the computation of stress according to the generalized power-law equation (Equation 3.12), the contribution of the latter term to the second invariant of the rate of deformation tensor, however, is neglected. This implies that:

$$\left(\frac{\partial u_z}{\partial z} \right)^2 \ll \left(\frac{\partial u_z}{\partial y} \right)^2 \quad (\text{A.3.1.1})$$

Using the solution obtained in Equation 3.15, these terms can be evaluated. The ratio of the two terms, ϵ , is given as follows.

$$\epsilon \equiv \frac{(\partial u_z / \partial z)^2}{(\partial u_z / \partial y)^2} = \left(\frac{H/z}{1+1/n} \right)^2 \quad (\text{A.3.1.2})$$

Since $z \gg H$, then $\epsilon \ll 1$ and the term $(\partial u_z / \partial z)^2$ is negligible compared to $(\partial u_z / \partial y)^2$. This reasoning also justifies the neglect of the term $(\partial \tau_{zz} / \partial z)$ in the z -direction momentum equation in obtaining the velocity profile given by Equation 3.13. It may

further be noted that in obtaining the above solution, the inertial terms of the momentum equation are neglected because the flow profile is dominated by the viscous stresses due to the extremely high viscosity of the melt.

A final assumption is associated with the use of Jeffrey's equations for the nonhomogeneous flow field encountered in an expanding channel die. As mentioned earlier, the equations are derived for a homogeneous flow field where the components of the rate of deformation tensor are uniform throughout the flow field. However, the experiments of Goldsmith and Mason (49) suggest that these equations can be applied to a nonhomogeneous flow field if the local values of the components of the rate of deformation tensor are used. It may be noted that the nonhomogeneity of the flow field can cause translational slip of the fluid at the fiber surface, such that the fiber moves at a velocity lower than the local fluid velocity (67). In addition, the nonhomogeneous flow field can also cause transverse migration of the fiber (68). These effects can significantly influence its orientation. They are, however, poorly understood and difficult to describe quantitatively.

In view of these assumptions, the predictions of the analysis should not be expected to describe the experimental findings quantitatively. However, as seen in Chapters 3 and 4, they do support the experimental observations qualitatively and provide insight into the process of fiber orientation during processing.

APPENDIX 3.2

PROGRAM FOR CALCULATING THE CHANGE IN THE FIBER ORIENTATION IN SIMPLE EXTRUSION THROUGH THE EXPANDING CHANNEL DIE

```

/INFO MUSICA R(MCCONNEL) TI(100) PA(999)
/LOAD WATFIV
C   USED FOR THE FINAL RESULTS
C   PROGRAMME FOR THE SOLUTION OF PHI AND PSI BY EULER'S METHOD
C   POWER-LAW VELOCITY PROFILE
COMMON URT,Y1
COMMON I,J,K,Y,B,Z,DELZ,PHI,PHIO,RADCON,ALPHA,PI,PN,
1   SLOPE1,SLOPE2(10),PSI(10),PSIC(10),PSIO(10)
C   SPECIFY R0,R1,H,AS,ALPHAD,PN
PN = 0.5
172 R0 = 3./8.0
R1 = 1.0
H = 1./32.0
AS = 100.0
ALPHAD = 75.0
B = (AS*AS - 1.)/(AS*AS + 1.)
PI = 3.1415876
RADCON = PI/180.0
C   SPECIFY URT,Y1
C   URT = 3.0
C   Y1 = 0.5
C   CALCULATE INITIAL AND FINAL Z'S
ALPHA = ALPHAD* RADCON
Z0 = R0/SIN(ALPHA)
Z1 = R1/SIN(ALPHA)
Z0 = Z0/H
Z1 = Z1/H
C   START CALCULATION AT Y=0.0
Y = 0.00
DELY = 0.25
DELZ = (Z1 - Z0)/20.0
10 WRITE (6,20)Y,PN
20 FORMAT ('1',15X,'Y = ',F15.5,10X,'POWER-LAW INDEX = ',F7.3)
WRITE (6,30)
30 FORMAT (//6X,'Z',8X,'PHI',4X,'PSI(1)',3X,'PSI(2)',3X,
1'PSI(3)',3X,'PSI(4)',3X,'PSI(5)',3X,'PSI(6)',3X,'PSI(7)')
DO 90 I = 1,13
Z = Z0
PHI = (I - 1)* 15.0 * RADCON
PHIC = PHI/RADCON
DO 50 K = 1,7
PSI(K) = (K-1)*15.*RADCON
C   PSI(K) = (90.0 + (K-1)*15.0)*RADCON
PSIC(K) = PSI(K)/RADCON
50 PSIO(K) = PSIC(K)
ZL = 0.0
DO 60 J = 2,1000

```

```

C      IF ((Z-ZL).GE.((Z1-Z0)/25.0))GO TO 51
C      *****
C      IF (ZL.EQ.0.0)GO TO 51
C      *****
      GO TO 52
51     WRITE (6,70)Z,PHIC,(PSIC(K),K=1,7)
70     FORMAT (9(1X,F8.2))
      ZL = Z
52     DELZ = (Z1 - Z0)/20.0
      IF (Z - Z1) 80,91,91
80     CONTINUE
      PHIO = PHI
      CALL FUNCT1
      DO 160 K = 1,7
      CALL FUNCT2
160    CONTINUE
      ABS1 = ABS(SLOPE1)*DELZ
      IF (ABS1.GT.2.0*RADCON) DELZ = 2.0*RADCON/ABS(SLOPE1)
      DO 161 K = 1,7
      ABS2 = ABS(SLOPE2(K))*DELZ
      IF (ABS2.GT.2.0*RADCON) DELZ = 2.0*RADCON/ABS(SLOPE2(K))
161    CONTINUE
      PHI = PHI + DELZ*SLOPE1
      PHIC = PHI/RADCON
      DO 162 K = 1,7
      PSI(K) = PSI(K) + DELZ*SLOPE2(K)
162    PSIC(K) = PSI(K)/RADCON
      Z = Z +DELZ
60     CONTINUE
C      *****
C      91     WRITE (6,70) Z,PHIC,(PSIC(K),K=1,7)
C      *****
      WRITE (6,200)
200    FORMAT(//90(1H*)//)
      CALL DISTR
      WRITE (6,201)
201    FORMAT (//90(1H*)/90(1H*)//)
90     CONTINUE
      Y = Y + DELY
      IF (Y-0.85)10,171,171
C170    IF (PN.EQ.0.1)GO TO 171
C      PN = 0.1
C      GO TO 172
171    STOP
      END
C      *****
      SUBROUTINE FUNCT1
      COMMON URT,Y1
      COMMON I,J,K,Y,B,Z,DELZ,PHI,PHIO,RADCON,ALPHA,PI,PN,
1       SLOPE1,SLOPE2(10),PSI(10),PSIC(10),PSIO(10)
      YN = Y ** (1./PN)
      TERM1 = YN*(1. + B*COS(2*PHI))/(1. - YN*Y)
      TERM1 = TERM1 * (PN + 1.0)/(2.0*PN)
C      DNM = (Y1*Y1 - Y*Y) + URT*(1.0 - Y1*Y1)      ***
C      TERM1 = (1. + B* COS(2.*PHI))*Y/DNM          ***

```

```

C      IF ((Z-ZL).GE.((Z1-Z0)/25.0))GO TO 51
C      *****
C      IF (ZL.EQ.0.0)GO TO 51
C      *****
      GO TO 52
51     WRITE (6,70)Z,PHIC,(PSIC(K),K=1,7)
70     FORMAT (9(1X,F8.2))
      ZL = Z
52     DELZ = (Z1 - Z0)/20.0
      IF (Z - Z1) 80,91,91
80     CONTINUE
      PHIO = PHI
      CALL FUNCT1
      DO 160 K = 1,7
      CALL FUNCT2
160    CONTINUE
      ABS1 = ABS(SLOPE1)*DELZ
      IF (ABS1.GT.2.0*RADCON) DELZ = 2.0*RADCON/ABS(SLOPE1)
      DO 161 K = 1,7
      ABS2 = ABS(SLOPE2(K))*DELZ
      IF (ABS2.GT.2.0*RADCON) DELZ = 2.0*RADCON/ABS(SLOPE2(K))
161    CONTINUE
      PHI = PHI + DELZ*SLOPE1
      PHIC = PHI/RADCON
      DO 162 K = 1,7
      PSI(K) = PSI(K) + DELZ*SLOPE2(K)
162    PSIC(K) = PSI(K)/RADCON
      Z = Z +DELZ
60     CONTINUE
C      *****
C      91     WRITE (6,70) Z,PHIC,(PSIC(K),K=1,7)
C      *****
      WRITE (6,200)
200    FORMAT(//90(1H*))//)
      CALL DISTR
      WRITE (6,201)
201    FORMAT (//90(1H*)/90(1H*))//)
90     CONTINUE
      Y = Y + DELY
      IF (Y-0.85)10,171,171
C170   IF (PN.EQ.0.1)GO TO 171
C      PN = 0.1
C      GO TO 172
171    STOP
      END
C      *****
      SUBROUTINE FUNCT1
      COMMON URT,Y1
      COMMON I,J,K,Y,B,Z,DELZ,PHI,PHIO,RADCON,ALPHA,PI,PN,
1       SLOPE1,SLOPE2(10),PSI(10),PSIC(10),PSIO(10)
      YN = Y ** (1./PN)
      TERM1 = YN*(1. + B*COS(2*PHI))/(1. - YN*Y)
      TERM1 = TERM1 * (PN + 1.0)/(2.0*PN)
C      DNM = (Y1*Y1 - Y*Y) + URT*(1.0 - Y1*Y1)      ***
C      TERM1 = (1. + B* COS(2.*PHI))*Y/DNM          ***

```

```

TERM2 = B*SIN(2.*PHI)/(2.0*Z)
SLOPE1 = -(TERM1 + TERM2)
RETURN
END

```

C *****

```

SUBROUTINE FUNCT2

```

```

COMMON URT,Y1

```

```

COMMON I,J,K,Y,B,Z,DELZ,PHI,PHIO,RADCON,ALPHA,PI,PN,

```

```

1 SLOPE1,SLOPE2(10),PSI(10),PSIC(10),PSIO(10)

```

```

YN = Y ** (1./PN)

```

```

TERM1 = YN*SIN(2*PHIO)*SIN(2*PSI(K))/(1-Y*YN)

```

```

TERM1 = TERM1*(1.+PN)/PN

```

C DNM = (Y1*Y1 - Y*Y) + URT*(1. - Y1*Y1) ***

C TERM1 = 2.*Y*SIN(2.*PHIO)*SIN(2.*PSI(K))/DNM ***

```

TERM2 = -COS(2*PHIO)*SIN(2*PSI(K))/Z

```

```

TERM3 = 3.*SIN(2.*PSI(K))/Z

```

```

SLOPE2(K) = - B*(TERM1 + TERM2 + TERM3)/4.0

```

```

RETURN

```

```

END

```

C *****

```

SUBROUTINE DISTR

```

```

COMMON URT,Y1

```

```

COMMON I,J,K,Y,B,Z,DELZ,PHI,PHIO,RADCON,ALPHA,PI,PN,

```

```

1 SLOPE1,SLOPE2(10),PSI(10),PSIC(10),PSIO(10)

```

```

DIMENSION F0(10),F1(10),PSIM(10),PSIMC(10)

```

```

DO 210 L = 1,6

```

210 F0(L) = 1.0

```

DO 220 LL = 1,6

```

```

F1(LL) = F0(LL)*(PSIO(LL+1)-PSIO(LL))/(PSIC(LL + 1) - PSIC(LL))

```

```

PSIM(LL) = (PSIC(LL + 1) + PSIC(LL))/2.0

```

220 PSIMC(LL) = 180.0 - PSIM(LL)

```

WRITE (6,230) (F0(L),L=1,6),(F1(LL),LL=1,6),(PSIM(LL),LL=1,6)

```

```

1,(PSIMC(LL),LL=1,6)

```

230 FORMAT (// '0F0',5X,6(F8.3,2X)/'0F1',5X,6(F8.3,2X)/'0PSIM',3X,
16(F8.3,2X)/'0PSIMC',3X,6(F8.3,2X))

```

RETURN

```

```

END

```

C *****

/DATA

Appendix 3.3

Flexural Stiffness Data

a) Expanding Channel Die - Axial Direction

Rate of Extrusion	E_f	Mean E_f	95% Confidence Limit
cm ³ /s	MPa	MPa	MPa
0.0475	2886, 3133, 2809, 2819, 3288, 2988	2984	<u>+219</u>
0.2375	2549, 2557, 2880, 2910	2721	<u>+364</u>
0.475	2863, 2942, 2968, 3030, 2774, 2667	2871	<u>+155</u>
0.95	2812, 3122, 2716, 2770, 2646, 2758	2801	<u>+190</u>
2.375	2624, 2596, 2346, 2904, 2471, 2552	2580	<u>+215</u>

b) Expanding Channel Die - Hoop Direction

Rate of Extrusion	E _f	Mean E _f	95% Confidence Limit
cm ³ /s	MPa	MPa	MPa
0.0475	2175, 2261, 1997, 2378, 2556, 2477, 2525	2336	<u>+205</u>
0.2375	2581, 2600, 2101, 2083, 2226, 2258, 2427, 2503	2345	<u>+185</u>
0.475	2516, 2352, 2203, 2262, 2527, 2666, 2773	2469	<u>+209</u>
0.95	2198, 2136, 2398, 2302, 2437, 2426	2314	<u>+145</u>
2.375	2533, 2361, 2505, 2404, 2503, 2569	2477	<u>+92</u>

c) Expanding Channel Die - Core

Direction	E _f	Mean E _f	95% Confidence Limit
	MPa	MPa	MPa
Axial	1975, 1717, 1695, 1919	1826	<u>+362</u>
Hoop	2932, 2620, 2660, 2927, 2155	2659	<u>+440</u>

d) Straight Die - Axial Direction

Rate of Extrusion	E _f	Mean E _f	95% Confidence Limit
cm ³ /s	MPa	MPa	MPa
0.0475	4143, 4252, 4485, 4254, 4227, 4325	4276	<u>+104</u>
0.2375	4847, 5174, 4271, 4406, 5500, 4556, 3664, 4073	4562	<u>+320</u>
0.475	3854, 3695, 4257, 4687, 4069, 4063	4104	<u>+226</u>
0.95	5027, 4487, 3816, 4169, 3592, 3968, 4143, 4798	4250	<u>+298</u>
2.375	4080, 3915, 4092, 3695, 3566, 3588	3819	<u>+179</u>

e) Straight Die - Hoop Direction

Rate of Extrusion	E _f	Mean E _f	95% Confidence Limit
cm ³ /s	MPa	MPa	MPa
0.0475	1389, 1178, 1410, 1428, 1377, 1384	1361	<u>+81</u>
0.2375	1708, 1517, 1372, 1470, 1524, 1540	1522	<u>+86</u>
0.475	1352, 1291, 1326, 1547, 1522, 1421	1410	<u>+79</u>
0.95	1463, 1559, 1487, 1514, 1647, 1598	1544	<u>+68</u>
2.375	1572, 1411, 1477, 1421, 1543	1497	<u>+91</u>

Appendix 3.4

Tensile Strength Data

a) Expanding Die - Axial Direction

Rate of Extrusion	σ_y	Mean σ_y	95% Confidence Limit
cm ³ /s	MPa	MPa	MPa
0.0475	26.8, 25.4, 25.5, 24.1, 25.1, 23.6	25.1	<u>+1.3</u>
0.2375	23.4, 23.5, 26.0, 25.9, 22.1	24.2	<u>+2.4</u>
0.475	24.9, 23.3, 24.4, 24.5, 23.5, 24.3	24.2	<u>+0.8</u>
0.95	22.8, 24.1, 23.3, 23.9, 22.9, 22.1	23.2	<u>+0.8</u>
2.375	21.9, 21.6, 20.8, 22.4, 22.6, 22.3	21.9	<u>+0.8</u>

b) Expanding Die - Hoop Direction

Rate of Extrusion	σ_y	Mean σ_y	95% Confidence Limit
cm ³ /s	MPa	MPa	MPa
0.0475	30.4, 29.6, 30.0, 28.3, 32.2, 32.3	30.5	<u>+1.8</u>
0.2375	29.9, 29.7, 27.5, 27.9, 26.8, 28.5	28.4	<u>+1.4</u>
0.475	35.8, 33.2, 28.5, 27.6, 29.8, 28.3	30.5	<u>+3.8</u>
0.95	29.5, 28.2, 27.1, 28.8, 28.0, 30.2	28.6	<u>+1.3</u>
2.375	28.1, 28.3, 26.5, 26.4, 29.2, 23.0, 28.4, 30.0	27.5	<u>+2.0</u>

c) Expanding Channel Die - Core

Direction	σ_y	σ_y	95% Confidence Limit
	MPa	MPa	MPa
Axial	24.1, 26.4, 25.6, 20.7, 20.8, 21.6	23.2	<u>+2.6</u>
Hoop	38.4, 38.2, 34.3, 28.6, 28.6, 28.2, 25.1, 25.7, 33.2, 27.4	30.8	<u>+3.5</u>

d) Straight Die - Axial Direction

Rate of Extrusion	σ_y	Mean σ_y	95% Confidence Limit
cm ³ /s	MPa	MPa	MPa
0.0475	35.3, 40.3, 39.3, 38.7, 39.3	38.6	<u>+2.4</u>
0.2375	35.6, 32.4, 33.4, 34.7, 36.0	34.4	<u>+1.9</u>
0.475	32.3, 33.1, 30.1, 36.8, 35.8	33.6	<u>+3.3</u>
0.95	35.3, 34.4, 34.7, 39.2, 35.3	35.8	<u>+2.4</u>
2.375	34.1, 35.8, 33.1, 36.7, 34.4	34.8	<u>+1.8</u>

e) Straight Die - Hoop Direction

Rate of Extrusion	σ_y	Mean σ_y	95% Confidence Limit
cm ³ /s	MPa	MPa	MPa
0.0475	17.5, 17.4, 18.0, 17.9	17.7	<u>+0.5</u>
0.2375	19.0, 18.5, 17.8, 17.9	18.3	<u>+0.6</u>
0.475	17.2, 18.0, 17.4, 17.2	17.4	<u>+0.6</u>
0.95	18.4, 18.34, 18.8, 18.8	18.6	<u>+0.4</u>
2.375	17.2, 16.8, 16.2, 15.9	17.0	<u>+1.5</u>

Appendix 4.1

Numerical Solution of the Continuity Equation for the Derivation of Velocity Components

As discussed in Section 4.1.1, the normal component of velocity, u_n , can be obtained by numerical solution of the local continuity equation once the location of the interface is known. For this purpose, the domain of flow between the channel centerline and the wall along an incremental displacement Δr is discretized into several elements or control volumes, as shown in Figure A.4.1.1a. Each control volume has a grid point at its center. All the control volumes need not be of the same width. It is, however, convenient to locate them such that the melt interface and the channel wall correspond to the control volume faces and the point of interest, P (where the gradients of velocity components are desired), corresponds to a grid point, as shown in Figure A.4.1.1 a.

A typical control volume is shown in Figure A.4.1.1 b. It has a grid point P at its center, and is bounded by four faces a, b, c and d parallel to the r and n axes. The dimensions of the control volume are $\Delta r \times \Delta n \times l^C$. The radial and normal distances corresponding to the four bounding faces (r_a , r_b , n_c and n_d) and the velocity components across them (u_r^a , u_r^b , u_n^c and u_n^d) are also shown in Figure A.4.1.1 b. A material balance over the control volume gives the following discretized form of the local

CENTERLINE INTERFACE WALL

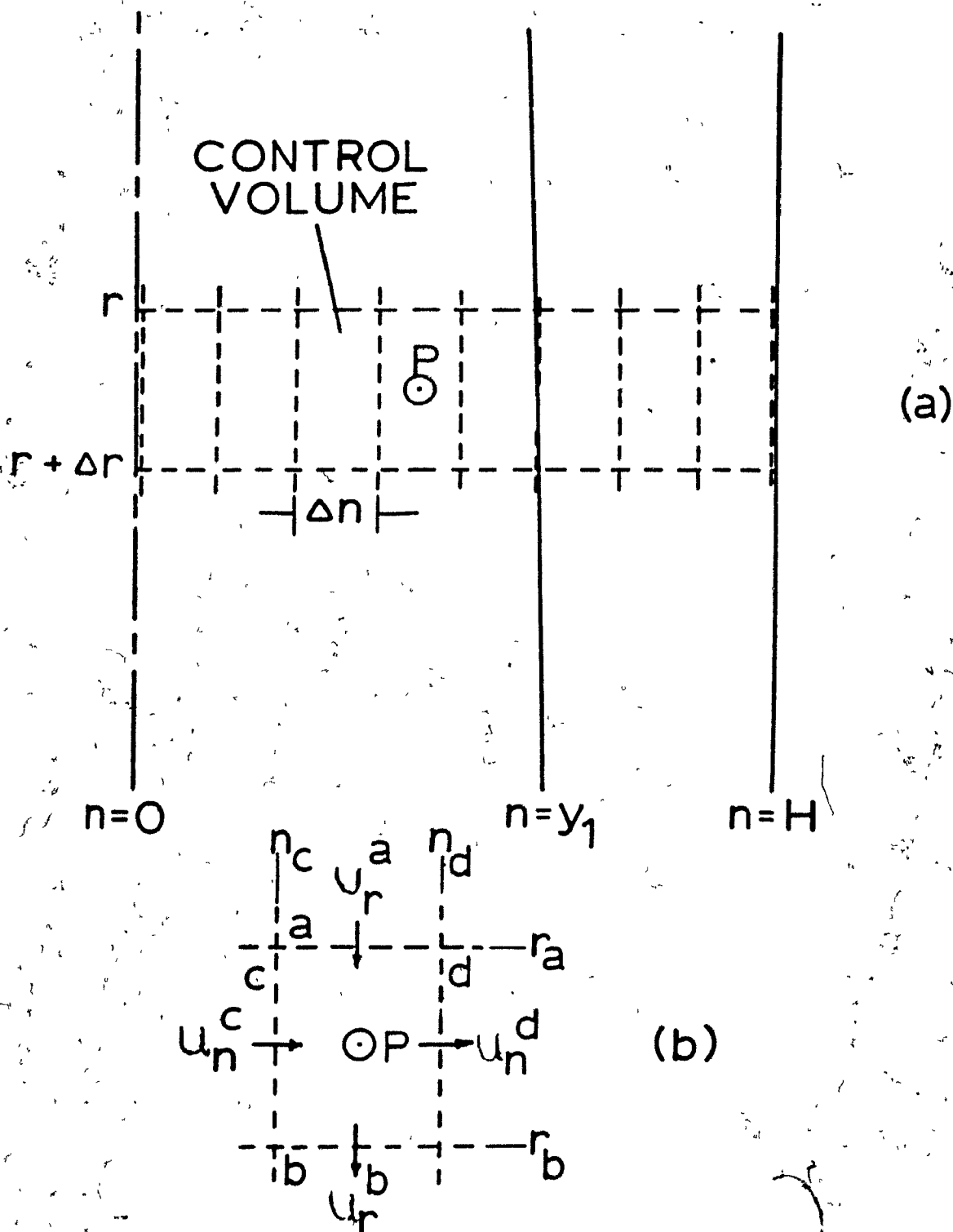


Figure A.4.1.1 (a) Discretization of the Flow Domain into Control Volumes.
(b) Typical Control Volume.

continuity equation.

$$(r_a \Delta n u_r^a - r_b \Delta n u_r^b) + 1/2(r_b^2 - r_a^2)(u_n^c - u_n^d) = 0 \quad (A.4.1.1)$$

It may be noted that the r - components of velocity u_r^a and u_r^b can be evaluated using Equation 4.5 or 4.6 depending on which layer of the flow field they correspond to. Equation A.4.1.1 can be rearranged to express the velocity component u_n^d in terms of the other velocity components as follows.

$$u_n^d = u_n^c + \frac{1}{r_p} \frac{\Delta n}{\Delta r} [r_a u_r^a - r_b u_r^b] \quad (A.4.1.2)$$

where

$$r_p = \frac{r_a + r_b}{2} \quad \text{and} \quad \Delta r = r_b - r_a$$

The normal velocity component u_n^d can thus be calculated if the velocity component u_n^c is known. Equation A.4.1.2 can be written for all the control volumes from the channel centerline to the wall to obtain a set of linear algebraic equations with u_n components of velocity as unknowns. These equations can be solved using the following boundary condition.

$$u_n = 0 \quad \text{at} \quad n = \pm H$$

It may be noted that due to the assumed symmetry of the velocity profile about the channel centerline, the above solution should give:

$$u_n = 0 \quad \text{at} \quad n = 0 \quad (A.4.1.3)$$

The above procedure thus gives the velocity components at the bounding faces of the control volumes considered. If desired, the velocity components at the grid points can be evaluated by a linear interpolation within the control volume. The gradients of the velocity components at the grid point can be evaluated using the following difference formulae.

$$\left(\frac{\partial u_r}{\partial r} \right)_P = \frac{u_r^b - u_r^a}{\Delta r} \quad \left(\frac{\partial u_n}{\partial n} \right)_P = \frac{u_n^d - u_n^c}{\Delta n} \quad (\text{A.4.1.4})$$

Accuracy of such a solution can be increased by considering a larger number of control volumes. In the results presented in Chapter 4, computations were carried out using 30 to 50 control volumes between the channel centerline and the interface, and 20 control volumes between the interface and the wall to obtain the desired accuracy with a grid size independent solution.

APPENDIX 4.2

PROGRAM FOR CALCULATING THE CHANGE IN THE FIBER ORIENTATION IN COEXTRUSION THROUGH THE EXPANDING CHANNEL DIE

```

/INFORMUSICA TI(100)
/SYS REG=MAX
/LOAD WATFIV
C      /INFO MVS PAG(9999) TIM(0100) CL(10)
C      // EXEC WATFIV
C      //GO.SYSIN DD *
C      $WATFIV ,TIME=100,PAGES=9999
C      PROGRAMME FOR CALCULATING THE INTERFACE POSITION ASSUMING
C      A MEAN DIE RADIUS - R AND SYMMETRICAL FLOW DISTRIBUTION
C      OF THE LAYER THICKNESSES.
COMMON I4
COMMON R0,R1,H,ALPHA,PI,RADCON,ASR
COMMON C1,C2,PN1,PN2,PN11,PN12,PN21,PN22,PNI,PWR
COMMON Q1S,Q2S,QLIM1,QLIM2,QS,Q1,Q1F
COMMON Z,Z0,ZF,Z1,Z2,ZS,DZ,DL
COMMON Y,YS,YG,DY
COMMON Y1S,Y1Z,Y1F,Y1Z1,Y1Z2,Y11,Y1,DY1
COMMON S,DS,VS
COMMON VZ,VZS,VZ1,VZ2,VZ1T,VZ2T
COMMON VYS,VYZ,VY1,VY2
COMMON FRS,FRZ,FRZ1,FRZ2,FRC
COMMON DVZDZ,DVZDY,DVYDY
COMMON PHI,PHIC,PHIO,SLOPE1
COMMON VZS2,VYS1,VYS2,VYIN,VYSF
COMMON PSI(10),PSIC(10),PSIO(10),PSIM(10),SLOPE2(10)
COMMON F0(10),F1(10)
CALL DIEGM
CALL PWRLAW
Z = Z0
C      *****
C      Q2S = 5.0
C      Y1Z = 0.4
C      DO 102 I = 1,6
C      Y1Z = Y1Z + 0.1
C      IF (Y1Z.GE.1.0) Y1Z = 0.95
C      Q2S = Q2S * (35.0 - I*I)/36.0
C      *****
C      CALL FLWCND
C      WRITE (6,10)
C 10  FORMAT
C      DO 20 I1 = 1,7
C      I1 = 3
C      Z = Z0
C      DS = (ZF - Z0)/100.0
C      PHI = (I1 - 1.0)*15.0*RADCON
C      PHIC = PHI/RADCON
C      DO 30 I2 = 1,7

```

```

C      PSI(I2) = (I2 - 1.0)*15.0 * RADCON
C      PSIC(I2) = PSI(I2) / RADCON
C 30   PSIO(I2) = PSIC(I2)
      ZL = 0.0
C      DO 40 I3 = 2,2000
C      DS = (ZF - Z0) / 100.0
C      DZ = DS
C      IF (Z - ZF) 80,90,90
C 80   CONTINUE
C      PHIO = PHI
      ZS = Z
      CALL SOLVE
102   CONTINUE
C      *****
      CALL PROFIL
C      *****
      GO TO 101
      Y1Z = Y1S
      FRZ = FRS
      IF (Z.NE.Z0) GO TO 100
      Y = 0.0
100   YS = Y
      YR = Y/Y1S
      CALL CALVZ1
      VZ = VZS
      IF (Z.EQ.Z0) GO TO 110
      Z1 = Z - DZ/2.0
      ZS = Z1
      CALL SOLVE
      Y1Z1 = Y1S
      FRZ1 = FRS
      YS = Y
      CALL CALVZ1
      VZ1 = VZS
      GO TO 120
110   Z1 = Z0
      Y1Z1 = Y1Z
      FRZ1 = FRZ
      VZ1 = VZ
120   Z2 = Z + DZ/2.0
      ZS = Z2
      CALL SOLVE
      Y1Z2 = Y1S
      FRZ2 = FRS
      YS = Y
      CALL CALVZ1
      VZ2 = VZS
      CALL VZGRAD
      CALL CALVY
      VYZ = VYS
      VYIN = VYS1
      VYSF = VYS2
      VS = SQRT (VZ*VZ + VYZ*VYZ)
      IF ( (Z - ZL).GE.( (ZF - Z0)/20.0) ) GO TO 50
      GO TO 60

```

```

50  WRITE (6,131) Y,Z,Y1Z,VZ,VYZ,VYIN,VYSF,DVYDY,FRZ
131  FORMAT (/3X,F10.5,1X,F10.5,1X,F10.5,1X,F15.5,1X,F10.5,1X,F15.5,
1    1X,F15.5,1X,F10.5,1X,F10.5)
    WRITE (6,70) Y, Z, Y1Z, PHIC, (PSIC(K),K = 1,7)
70  FORMAT (/11(1X,F10.3))
    ZL = Z
60  CALL FUNCT1
    DO 140 I4 = 1,7
    CALL FUNCT2
140  CONTINUE
    ABS1 = ABS ( SLOPE1 ) * DS
    IF ( ABS1 . GT. 2.0 * RADCON ) DS = 2.0 * RADCON / ABS(SLOPE1)
    DO 150 I5 = 1,7
    ABS2 = ABS ( SLOPE2(I5) ) * DS
    IF ( ABS2 . GT. 2.0*RADCON ) DS = 2.0*RADCON / ABS(SLOPE2(I5))
150  CONTINUE
    PHI = PHI + DS * SLOPE1
    PHIC = PHI / RADCON
    DO 160 I6 = 1,7
    PSI(I6) = PSI(I6) + DS * SLOPE2 (I6)
160  PSIC(I6) = PSI(I6)/RADCON
    Z = Z + VZ*DS/VS
    Y = Y + VYZ*DS/VS
    40  CONTINUE
    90  WRITE (6,170)
170  FORMAT (//90(1H*))//)
    CALL DISTR
    WRITE (6,170)
    20  CONTINUE
101  STOP
    END

```

C
C
C

SUBROUTINE USER

```

COMMON I4
COMMON R0,R1,H,ALPHA,PI,RADCON,ASR
COMMON C1,C2,PN1,PN2,PN11,PN12,PN21,PN22,PN1,PWR
COMMON Q1S,Q2S,QLIM1,QLIM2,QS,Q1,Q1F
COMMON Z,Z0,ZF,Z1,Z2,ZS,DZ,DL
COMMON Y,YS,YG,DY
COMMON Y1S,Y1Z,Y1F,Y1Z1,Y1Z2,Y11,Y1,DY1
COMMON S,DS,VS
COMMON VZ,VZS,VZ1,VZ2,VZ1T,VZ2T
COMMON VYS,VYZ,VY1,VY2
COMMON FRS,FRZ,FRZ1,FRZ2,FRC
COMMON DVZDZ,DVZDY,DVYDY
COMMON PHI,PHIC,PHIO,SLOPE1
COMMON VZS2,VYS1,VYS2,VYIN,VYSF
COMMON PSI(10),PSIC(10),PSIO(10),PSIM(10),SLOPE2(10)
COMMON F0(10),F1(10)
ENTRY DIEGM
DEFINE DIE GEOMETRICAL PARAMETERS
SPECIFY R0, R1, H, ALPHAD
R0 = 5./16.

```

C
C

```

        RI = 9.0/8.0
        H = 1./16.0
        ALPHAD = 45.0
C      NON - DIMENSIONALIZE THE PARAMETERS
        R0 = R0/H
        R1 = R1/H
        PI = 3.1415876
        RADCON = PI/180.0
        ALPHA = ALPHAD*RADCON
        Z0 = R0/SIN(ALPHA)
        ZF = R1/SIN(ALPHA)
        DELZ = (ZF - Z0)/10.0
        AS = 100.0
        ASR = (AS*AS - 1.0)/(AS*AS + 1.0)
        RETURN

```

```

C      *****
C
C

```

```

C      ENTRY PWRLAW
C      SPECIFY THE POWER - LAW PARAMETERS
        C1 = 23758.532
        C2 = 13536.407
        PN1 = 0.3298
        PN2 = 0.37969
C      CALCULATE THE NECESSARY RELATIONSHIPS
        PN11 = 1. + 1./PN1
        PN12 = 2. + 1./PN1
        PN21 = 1. + 1./PN2
        PN22 = 2. + 1./PN2
        PNS = PN2 - PN1
        IF (PN1.EQ.PN2) PNS = 1.0
        PNSI = 1./PNS
        PWR = (C2/C1)**PNSI
        PNI = PN2/PN1
        RETURN

```

```

C      *****
C
C

```

```

C      ENTRY FLWCND
C      SPECIFY THE FLOW RATES AND LIMITS FOR CONVERGENCE
C      Q1 --- FLOW RATE OF THE FILLED CORE LAYER
C      Q2 --- THE "TOTAL" FLOW RATE OF BOTH THE INNER AS WELL AS
C              THE OUTER UNFILLED LAYERS

```

```

        Q1S = 9.473
        Q2S = 3.14
C      QLIM1 = Q1S * 1.0 E - 05
C      QLIM2 = Q2S * 1.0 E - 05
        Q1S = Q1S * PWR / H ** 3.0
        Q2S = Q2S * PWR / H ** 3.0
        QLIM1 = Q1S*1.0 E -05
        QLIM2 = Q2S*1.0 E -05
        Y1Z = 0.62747
        GO TO 290

```

```

C      *****
C      CALCULATION OF FRZ FOR THE REDUCED SHEAR RATE (DIMENSIONLESS)

```

```

C      AT THE INTERFACE = 1.0
      FRZA = (1.0 - (Y1Z**PN21))/PN21
      FRZB = (Y1Z**PN21)/PN11
      FRZ = FRZA/(FRZB + FRZA)
      *****

C
C
C      FRZ= (1.0 - (Y1Z**PN11))
      FRZ = 0.7
      FRZLIM =FRZ * 1.0 E -06
      DQ2 = 0.1 * Q2S
      QE21 = Q2S
      ZS = Z
      Y1 = Y1Z
      CALL CALQ
      FRZ1 = FRC
      Q1 = QS
      Q2S = Q2S + DQ2
      CALL CALQ
      FRZC =FRC
      Q1 = QS
      ITER = 1
      DO 250 IQ2 = 1,200
      SLOPE = (Q2S - QE21)/(FRZC - FRZ1)
      Q2S = QE21 + SLOPE * (FRZ - FRZ1)
      CALL CALQ
      FRZC =FRC
      Q1 = QS
      RESID = ABS(FRZ - FRZC)
      IF (RESID.LE.FRZLIM) GO TO 260
      ITER = ITER + 1
250  CONTINUE
      WRITE (6,270)
270  FORMAT (/5X,'NO CONVERGENCE ACHIEVED - Q2/FRZ')
260  FRZ =FRZC
      Y1Z =Y1
      Q1S =Q1
C      Q1SA = Q1S * H ** 3.0/ PWR
C      Q2SA = Q2S * H ** 3.0 / PWR
      QLIM1 = Q1S * 1.0 E - 06
      QLIM2 = Q2S * 1.0 E - 06
      WRITE (6,280) Q1S ,Q2S ,QLIM1,QLIM2,FRZ,Y1Z,FRZLIM
280  FORMAT (/5X,'Q1SA = ',F15.5,5X,'Q2SA = ',F15.5/
1      5X,'QLIM1 = ',F15.5,5X,'QLIM2 = ',F15.5/
2      5X,'FRZ = ',F15.5,5X,'Y1Z = ',F15.5,5X,'FRZLIM = ',F15.5)
290  RETURN
      END

C
C
C      *****

C      SUBROUTINE SOLVE
C      THIS SUBROUTINE SOLVES FOR Y1 AND FR FOR THE SPECIFIED
C      FLOW RATES, POWER - LAW PARAMETERS AND ZS
C      Y1S IS THE INTERFACE LOCATION --- "SYMMETRICAL"
C      FRS IS THE INTERFACE VELOCITY AS A FRACTION OF THE MAXIMUM
C      CENTER - LINE VELOCITY.

```

```

COMMON I4
COMMON R0,R1,H,ALPHA,PI,RADCON,ASR
COMMON C1,C2,PN1,PN2,PN11,PN12,PN21,PN22,PN1,PWR
COMMON Q1S,Q2S,QLIM1,QLIM2,QS,Q1,Q1F
COMMON Z,Z0,ZF,Z1,Z2,ZS,DZ,DL
COMMON Y,YS,YG,DY
COMMON Y1S,Y1Z,Y1F,Y1Z1,Y1Z2,Y11,Y1,DY1
COMMON S,DS,VS
COMMON VZ,VZS,VZ1,VZ2,VZ1T,VZ2T
COMMON VYS,VYZ,VY1,VY2
COMMON FRS,FRZ,FRZ1,FRZ2,FRC
COMMON DVZDZ,DVZDY,DVYDY
COMMON PHI,PHIC,PHIO,SLOPE1
COMMON VZS2,VYS1,VYS2,VYIN,VYSF
COMMON PSI(10),PSIC(10),PSIO(10),PSIM(10),SLOPE2(10)
COMMON F0(10),F1(10)
IF (Q2S.EQ.0.0) GO TO 340
DY1 = 0.01
Y11 = Y1Z
Y1 = Y11
CALL CALQ
Q11 = QS
Y1 = Y1 + DY1
CALL CALQ
Q1 = QS
ITER = 1
DO 300 IY1 = 1,200
SLOPE = (Y1 - Y11)/(Q1 - Q11)
Y1 = Y11 + SLOPE * (Q1S - Q11)
CALL CALQ
Q1 = QS
RESID = ABS(Q1S - Q1)
IF ( RESID.LE.QLIM1) GO TO 310
ITER = ITER + 1
300 CONTINUE
WRITE (6,320)
320 FORMAT (/5X,'NO CONVERGENCE ACHIEVED')
310 FRS = FRC
Y1S = Y1
Q1S = QS
GO TO 350
340 Y1S = 1.0
FRS = 0.0
350 WRITE (6,330) Y1S, Q1S,FRS,Q2S,Z,ITER
330 FORMAT ('1',5X,'Y1 = ',F15.5,
1      5X,'Q1 = ',F15.5,5X,'FR = ',F15.5/5X,
1      'Q2S = ',F15.5,5X,'Z = ',F15.5,5X,'ITER = ',I4)
RETURN
END

*****

SUBROUTINE CALQ
THIS SUBROUTINE CALCULATES THE VALUE OF Q1 FOR THE
SPECIFIED VALUE OF Y1

```



```

COMMON I4
COMMON R0,R1,H,ALPHA,PI,RADCON,ASR
COMMON C1,C2,PN1,PN2,PN11,PN12,PN21,PN22,PN1,PWR
COMMON Q1S,Q2S,QLIM1,QLIM2,QS,Q1,Q1F
COMMON Z,Z0,ZF,Z1,Z2,ZS,DZ,DL
COMMON Y,YS,YG,DY
COMMON Y1S,Y1Z,Y1F,Y1Z1,Y1Z2,Y11,Y1,DY1
COMMON S,DS,VS
COMMON VZ,VZS,VZ1,VZ2,VZ1T,VZ2T
COMMON VYS,VYZ,VY1,VY2
COMMON FRS,FRZ,FRZ1,FRZ2,FRC
COMMON DVZDZ,DVZDY,DVYDY
COMMON PHI,PHIC,PHIO,SLOPE1
COMMON VZS2,VYS1,VYS2,VYIN,VYSF
COMMON PSI(10),PSIC(10),PSIO(10),PSIM(10),SLOPE2(10)
COMMON F0(10),F1(10)
A = (1.0 - (Y1**PN22)) / PN22
B = 1.0 - Y1 - A
C = (PN21 * Q2S / B) ** PN1
ZTERM = (4.0 * PI * ZS* SIN (ALPHA)) ** (1.0 - PN1)
D = (Y1 ** PN12) * ZTERM / PN12
E = Q2S * (1.0 - (Y1 ** PN21))/ B
QS = D * C + E * Y1
F = C * ZTERM * (Y1 ** PN11)/ PN11
FRC = E / (E + F)

```

C

```

ZA = 4.0 * PI * ZS* SIN(ALPHA)
ZB = ZA ** PN1
VTERM1 = (Y1 **PN11) * C / (PN11 * ZB)
VTERM2 = E / ZA
RETURN
END

```

C
C
C

SUBROUTINE VELGRD

```

COMMON I4
COMMON R0,R1,H,ALPHA,PI,RADCON,ASR
COMMON C1,C2,PN1,PN2,PN11,PN12,PN21,PN22,PN1,PWR
COMMON Q1S,Q2S,QLIM1,QLIM2,QS,Q1,Q1F
COMMON Z,Z0,ZF,Z1,Z2,ZS,DZ,DL
COMMON Y,YS,YG,DY
COMMON Y1S,Y1Z,Y1F,Y1Z1,Y1Z2,Y11,Y1,DY1
COMMON S,DS,VS
COMMON VZ,VZS,VZ1,VZ2,VZ1T,VZ2T
COMMON VYS,VYZ,VY1,VY2
COMMON FRS,FRZ,FRZ1,FRZ2,FRC
COMMON DVZDZ,DVZDY,DVYDY
COMMON PHI,PHIC,PHIO,SLOPE1
COMMON VZS2,VYS1,VYS2,VYIN,VYSF
COMMON PSI(10),PSIC(10),PSIO(10),PSIM(10),SLOPE2(10)
COMMON F0(10),F1(10)
ENTRY CALVZ1

```

C
C

THIS SUBROUTINE CALCULATES VZ FOR SPECIFIED VALUES OF
YS,ZS,Y1S,FRS IN PHASE 1 - THE FILLED CORE PHASE

```

A = (2.0*PN1 + 1.0)/(PN1*(1.0 + FRS) + 1.0)
B = A / (4.0* PI * ZS * SIN (ALPHA) * Y1S)
C = Q1S / PWR
D = 1.0 - ((YS/Y1S)**PN11)
E = FRS + (1.0 - FRS)*D
VZS = B * C * E
RETURN

```

```

*****

```

```

ENTRY CALVZ2

```

```

THIS SUBROUTINE CALCULATES VZ FOR THE SPECIFIED VALUES
OF Y,Z,Y1Z,FRZ IN PHASE 2 - THE UNFILLED OUTER PHASE.

```

```

A = (2.0 * PN1 + 1.0) / (PN1 * (1.0 + FRS) + 1.0)
B = A * FRS / (4.0 * PI * ZS * SIN(ALPHA) * Y1S)
C = Q1S/ PWR
D1 = 1.0 - (YS ** PN21)
D2 = 1.0 - (Y1S ** PN21)
D = D1/D2
VZS2 = B * C * D
RETURN

```

```

A = (Q2S/PWR) / (4.0 * PI * ZS * SIN(ALPHA))
B = (1.0 - (Y1S**PN22) / PN22
C = 1.0 - Y1S - B
D = 1.0 - (YS * PN21)
VZS2 = A * D / C
RETURN

```

```

*****

```

```

ENTRY VZGRAD

```

```

THIS SUBROUTINE CALCULATES THE GRADIENTS OF THE VZ
COMPONENT OF VELOCITY

```

```

A = (2.0*PN1 + 1.0)*(1.0 - FRS)/(PN1*(1.0 + FRS) + 1.0)
B = A / (4.0 * PI * ZS * SIN(ALPHA) * Y1S * Y1S)
C = Q1S/ PWR
D = PN11 * ( (YS/Y1S) ** (1.0/PN1) )
DVZDY = - C * B * D

```

```

DVZDZ = (VZ2 - VZ1)/(Z2 - Z1)

```

```

RETURN

```

```

*****

```

```

ENTRY VGRAD2

```

```

THIS SUBROUTINE CALCULATES THE GRADIENTS OF THE VZ
COMPONENT OF VELOCITY IN THE UNFILLED PHASE 2.

```

```

A = (2.0*PN1 + 1.0)* FRS / (PN1*(1.0 + FRS) + 1.0)
B = A / (4.0 * PI * ZS * SIN(ALPHA) * Y1S )
C = Q1S/ PWR
D = (PN21 * (YS ** (1.0/PN2)))/(1.0 - ((Y1S)**PN21))
DVZDY = - C * B * D

```

```

RETURN

```

```

END

```

```

*****

```

C

SUBROUTINE CALVY

COMMON I4

COMMON R0,R1,H,ALPHA,PI,RADCON,ASR

COMMON C1,C2,PN1,PN2,PN11,PN12,PN21,PN22,PN1,PWR

COMMON Q1S,Q2S,QLIM1,QLIM2,QS,Q1,Q1F

COMMON Z,Z0,ZF,Z1,Z2,ZS,DZ,DL

COMMON Y,YS,YG,DY

COMMON Y1S,Y1Z,Y1F,Y1Z1,Y1Z2,Y11,Y1,DY1

COMMON S,DS,VS

COMMON VZ,VZS,VZ1,VZ2,VZ1T,VZ2T

COMMON VYS,VYZ,VY1,VY2

COMMON FRS,FRZ,FRZ1,FRZ2,FRC

COMMON DVZDZ,DVZDY,DVYDY

COMMON PHI,PHIC,PHIO,SLOPE1

COMMON VZS2,VYS1,VYS2,VYIN,VYSF

COMMON PSI(10),PSIC(10),PSIO(10),PSIM(10),SLOPE2(10)

COMMON F0(10),F1(10)

C

THIS SUBROUTINE CALCULATES VY FOR SPECIFIED VALUES OF
YS AND ZS USING THE LOCAL CONTINUITY.

C

C

IF (YS.EQ.0.0) GO TO 650

DY = Y/49.5

YG = DY/2.0

VY2 = 0.0

FACTOR = 2.0*DY / (Z2*Z2 - Z1*Z1)

DO 600 IVY, = 1,50

VY1 = VY2

ZS = Z1

YS = YG

Y1S = Y1Z1

FRS = FRZ1

CALL CALVZ1

VZ1T = VZS

ZS = Z2

YS = YG

Y1S = Y1Z2

FRS = FRZ2

CALL CALVZ1

VZ2T = VZS

C

C

C

MATERIAL BALANCE OVER A FINITE ELEMENT TO CALCULATE VYS

VY2 = VY1 + (Z1 * VZ1T - Z2 * VZ2T) * FACTOR

YG = YG + DY

600 CONTINUE

VYS = (VY1 + VY2)/2.0

DVYDY = (VY2 - VY1)/DY

YB = Y + DY / 2.0

GO TO 660

C

650 VYS = 0.0

DVYDY = 0.0

VY2 = 0.0

YB = 0.0

```

660  DY = ( Y1Z - YB ) / 20.0
      YG = YB + DY / 2.0
      FACTOR = 2.0 * DY / (Z2*Z2 - Z1*Z1)
      DO 610 IVY = 1,20
        VY1 = VY2
        ZS = Z1
        YS = YG
        Y1S = Y1Z1
        FRS = FRZ1
        CALL CALVZ1
        VZ1T = VZS
        ZS = Z2
        YS = YG
        FRS = FRZ2
        Y1S = Y1Z2
        CALL CALVZ1
        VZ2T = VZS
        VY2 = VY1 + (Z1* VZ1T - Z2* VZ2T) * FACTOR
610  YG = YG + DY
      VYS1 = VY2
      IF (Q2S.EQ.0.0) GO TO 630

```

C

```

      DY = (1.0 - Y1Z) / 20.0
      YG = Y1Z + DY/2.0
      FACTOR = 2.0 * DY / (Z2*Z2 - Z1*Z1)
      DO 620 IVY = 1,20
        VY1 = VY2
        ZS = Z1
        YS = YG
        Y1S = Y1Z1
        FRS = FRZ1
        CALL CALVZ2
        VZ1T = VZS2
        ZS = Z2
        YS = YG
        Y1S = Y1Z2
        FRS = FRZ2
        CALL CALVZ2
        VZ2T = VZS2
        VY2 = VY1 + (Z1 * VZ1T - Z2 * VZ2T) * FACTOR
620  YG = YG + DY
      VYS2 = VY2
      GO TO 640
630  VYS2 = 0.0
640  RETURN
      END

```

C
C
C

SUBROUTINE FUNCT

COMMON I4

COMMON R0,R1,H,ALPHA,PI,RADCON,ASR

COMMON C1,C2,PN1,PN2,PN11,PN12,PN21,PN22,PN1,PWR

COMMON Q1S,Q2S,QLIM1,QLIM2,QS,Q1,Q1F

COMMON Z,Z0,ZF,Z1,Z2,ZS,DZ,DL

```

COMMON Y,YS,YG,DY
COMMON Y1S,Y1Z,Y1F,Y1Z1,Y1Z2,Y11,Y1,DY1
COMMON S,DS,VS
COMMON VZ,VZS,VZ1,VZ2,VZ1T,VZ2T
COMMON VYS,VYZ,VY1,VY2
COMMON FRS,FRZ,FRZ1,FRZ2,FRC
COMMON DVZDZ,DVZDY,DVYDY
COMMON PHI,PHIC,PHIO,SLOPE1
COMMON VZS2,VYS1,VYS2,VIIN,VYSF
COMMON PSI(10),PSIC(10),PSIO(10),PSIM(10),SLOPE2(10)
COMMON F0(10),F1(10)
ENTRY FUNCT1
TERM1 = (1.0 + ASR*COS(2.0*PHI))*DVZDY/2.0
TERM2 = (DVYDY - DVZDZ)*SIN(2.0*PHI)*ASR/2.0
SLOPE1 = (TERM1 - TERM2)/VS
RETURN

```

```

C
ENTRY FUNCT2
TERM1 = DVZDY*SIN(2.0*PHIO)*SIN(2.0*PSI(I4))
TERM2 = DVYDY*SIN(2.0*PSI(I4))*(3.0 + COS(2.0*PHIO))
TERM3 = DVZDZ*SIN(2.0*PSI(I4))*(3.0 - COS(2.0*PHIO))
SLOPE2(I4) = ASR*(TERM1 + TERM2 + TERM3)/(4.0*VS)
RETURN

```

```

C
ENTRY DISTR
DO 700 L = 1,6
700 F0(L) = 1.0
DO 710 LL = 1,6
F1(LL) = F0(LL)*(PSIO(LL+1) - PSIO(LL))/(PSIC(LL+1) - PSIC(LL))
710 PSIM(LL) = (PSIC(LL+1) + PSIC(LL))/2.0
WRITE (6,720) (F0(L),L=1,6),(F1(L),L=1,6),(PSIM(L),L=1,6)
720 FORMAT (// '0F0',5X,6(F8.3,2X)/ '0F1',5X,6(F8.3,2X)/
1 '0PSIM',3X,6(F8.3,2X))
RETURN
END

```

```

C
SUBROUTINE PROFIL
COMMON I4
COMMON R0,R1,H,ALPHA,PI,RADCON,ASR
COMMON C1,C2,PN1,PN2,PN11,PN12,PN21,PN22,PNI,PWR
COMMON Q1S,Q2S,QLIM1,QLIM2,QS,Q1,Q1F
COMMON Z,Z0,ZF,Z1,Z2,ZS,DZ,DL
COMMON Y,YS,YG,DY
COMMON Y1S,Y1Z,Y1F,Y1Z1,Y1Z2,Y11,Y1,DY1
COMMON S,DS,VS
COMMON VZ,VZS,VZ1,VZ2,VZ1T,VZ2T
COMMON VYS,VYZ,VY1,VY2
COMMON FRS,FRZ,FRZ1,FRZ2,FRC
COMMON DVZDZ,DVZDY,DVYDY
COMMON PHI,PHIC,PHIO,SLOPE1
COMMON VZS2,VYS1,VYS2,VIIN,VYSF
COMMON PSI(10),PSIC(10),PSIO(10),PSIM(10),SLOPE2(10)
COMMON F0(10),F1(10)

```

```

C THIS SUBROUTINE CALCULATES THE VZ COMPONENT VELOCITY
C PROFILE AT THE SPECIFIED VALUE OF Z.

```

```

C      ZS = ZO
C      FRS = FRZ
C      Y1S = Y1Z
      YS = 0.0
      CALL CALVZ1
      VZPR1 = VZS*H*2.54
      CALL VZGRAD
      WRITE (6,2010) YS, VZPR1, DVZDY
      DO 2000 I = 1,5
      YS = I * Y1S/5.0
      CALL CALVZ1
      VZPR = VZS*H*2.54
      CALL VZGRAD
      VZPR = VZPR/VZPR1
      WRITE (6,2010) YS, VZPR, DVZDY
2000  CONTINUE
      WRITE (6,2030)
2030  FORMAT ('0',80(1H*))
      YS = Y1S
      CALL CALVZ2
      VZPR = VZS2 * H * 2.54
      CALL VGRAD2
      VZPR = VZPR/VZPR1
      WRITE (6,2010) YS, VZPR, DVZDY
      DO 2020 I = 1,5
      YS = Y1S + I * (1.0 - Y1S)/5.0
      CALL CALVZ2
      VZPR = VZS2*H* 2.54
      CALL VGRAD2
      VZPR = VZPR/VZPR1
      WRITE (6,2010) YS, VZPR,DVZDY
2020  CONTINUE
2010  FORMAT ('0',5X,'Y = ',F10.5,5X,'VZ = ',F15.5,5X,'DVZDY = ',F15.5)
      RETURN
      END

```

\$DATA

\$

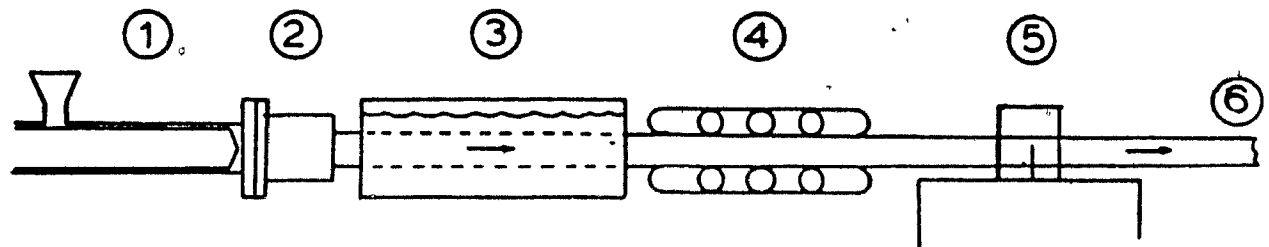
//

Appendix 4.3

Procedure for the Start-Up and Continuous Pipe Manufacture

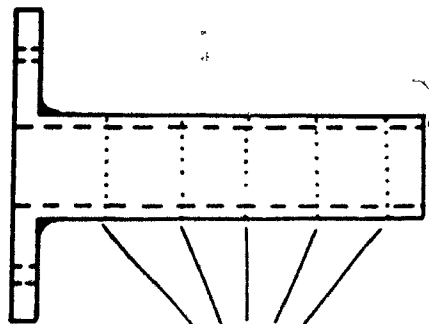
The manufacture of plastic pipe involves extrusion, calibration, cooling and take-off. A typical pipe extrusion line is shown schematically in Figure A.4.3.1. It consists of an extruder, a pipe die, a calibration-cooling tank and a take-off system. The die is mounted on the extruder supplying the polymer melt. The extrudate coming out of the die enters the calibrator mounted at the front end of the cooling tank. A typical pipe calibrator is shown in Figure A.4.3.2. The pipe freezes along the cooling tank and is subsequently carried away by the take-off system usually consisting of a Caterpillar haul-off and an automatic saw cutter.

In order to maintain the tubular shape of the extrudate as it comes out of the die and produce a pipe with desired dimensions, it is important to calibrate it properly, freeze the outer surface as quickly as possible, and attain a synchronization between the rate of extrusion, cooling and the take-off speed. A common method of calibration involves maintaining the cooling tank under vacuum; while an atmospheric pressure is maintained inside the pipe. For the start-up, a sufficient length of the extrudate is produced continuously and passed through the calibrator and the cooling tank. The front end of the extrudate is sealed off and air is blown inside it through the mandrel by a



- (1) EXTRUDER (4) HAUL - OFF
 (2) PIPE DIE (5) SAW CUTTER
 (3) COOLING TANK (6) EXTRUDED PIPE

Figure A.4.3.1 Typical Pipe Extrusion Line.



Holes for Entry of Water

Figure A.4.3.2 Pipe Calibrator

special air channel provided in the die. At the same time, the cooling tank is flooded with water and maintained under vacuum. As the extrudate enters the calibrator, it is subjected to a pressure differential, the pressure being higher inside the extrudate. Under the differential pressure, it is pushed against the cold wall of the calibrator, and its outer surface freezes quickly. Subsequent cooling of the extrudate occurs as it moves along the cooling tank, and the extrudate is completely frozen forming the desired pipe at the exit of the tank. A rubber gasket is provided at the rear end of the cooling tank with a circular hole in the center that is slightly smaller than the outer diameter of the pipe. Once a calibrated pipe of desired dimensions is formed, the rubber gasket seals the rear end of the tank, thus maintaining the vacuum inside it. No air blowing inside the extrudate should, in principle, be necessary as the steady state conditions are established. Blowing at a slow rate is, however, usually necessary in order to overcome some fluctuations in the extruder output and small leaks at the two ends of the cooling tank. The pipe is subsequently taken-off and cut to pieces of desired length by the take-off system.

APPENDIX 4.4

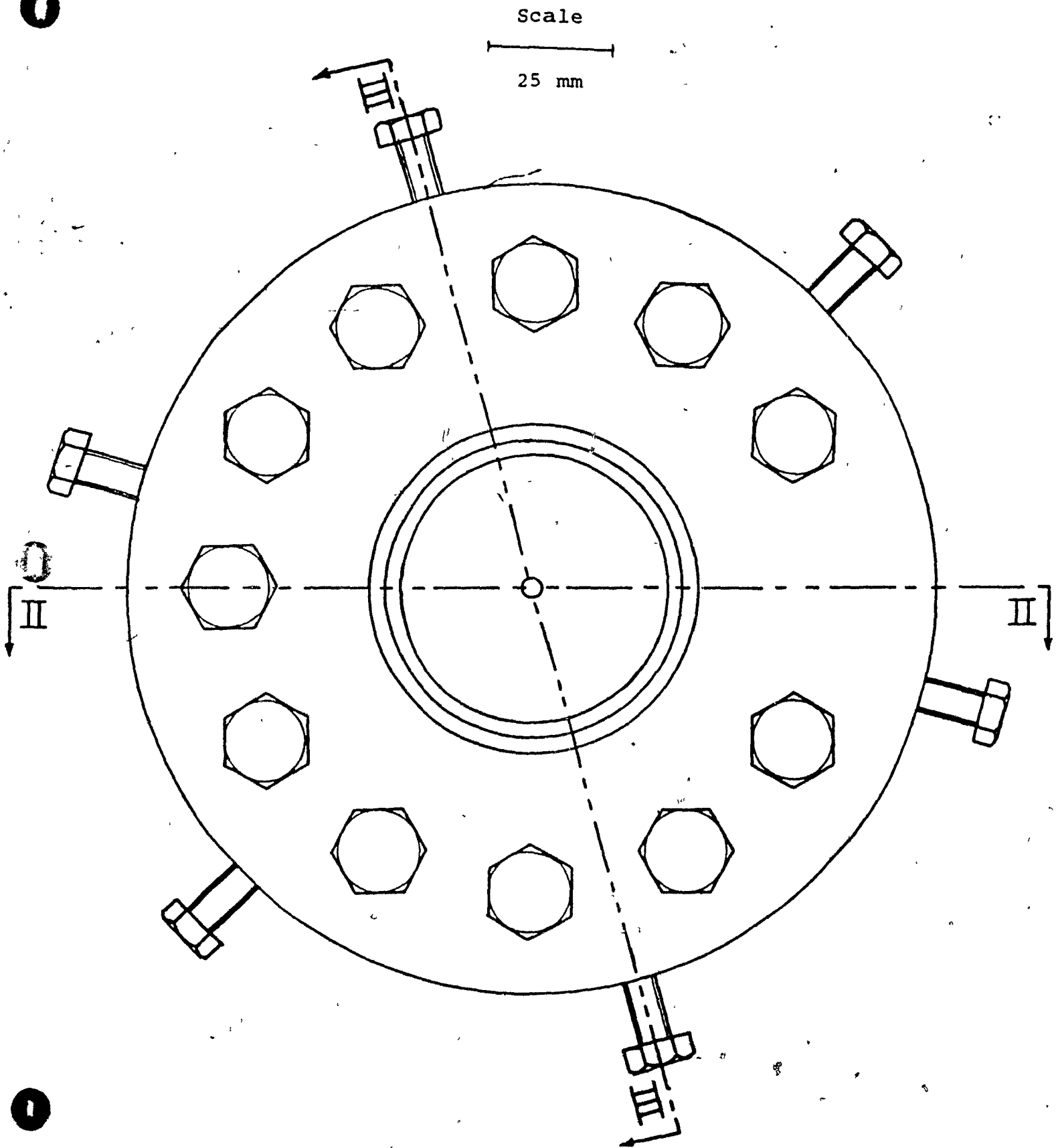


Figure A.4.4.1 Front View of the Coextrusion Die.

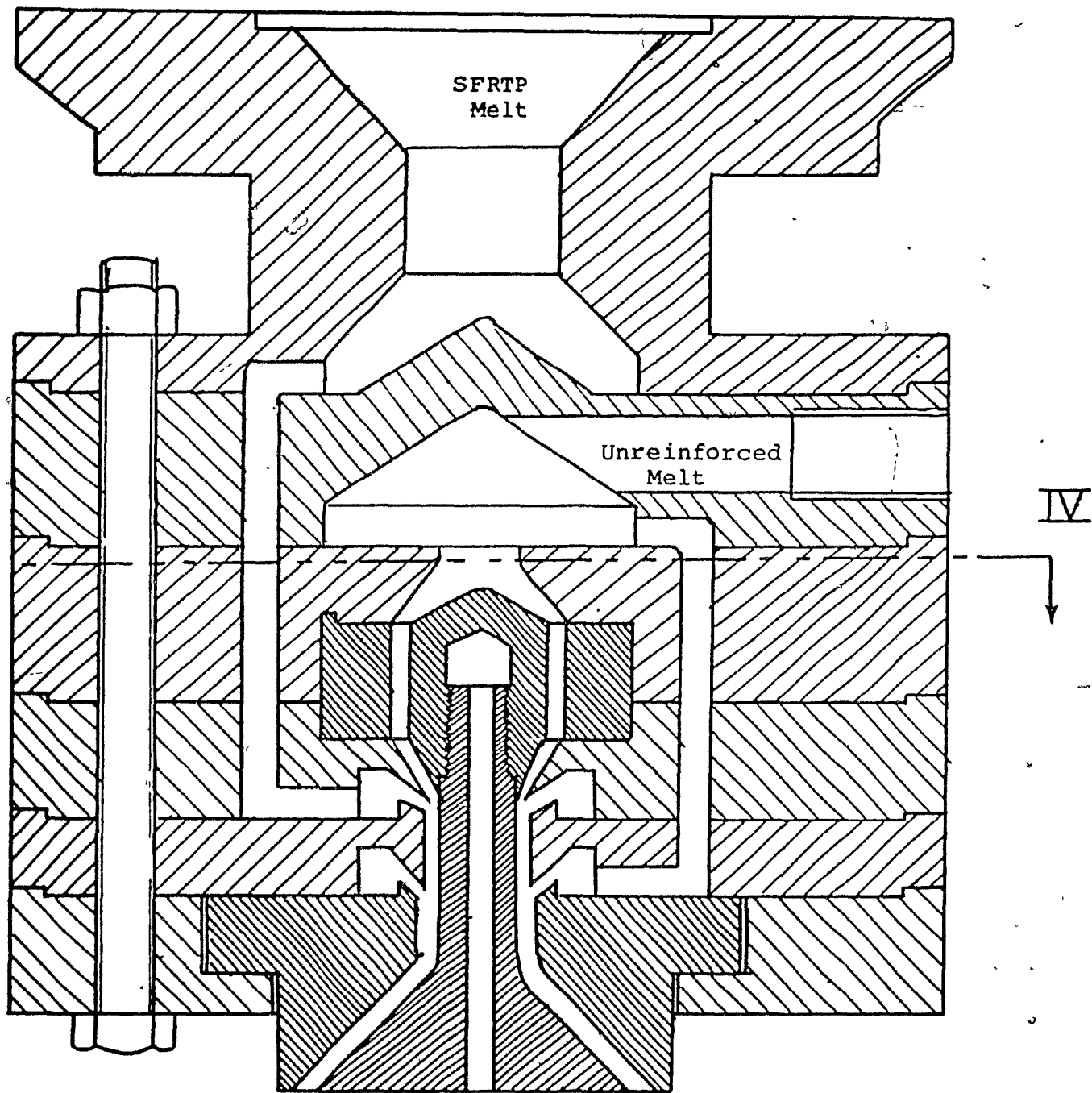


Figure A.4.4.2 Sectional Plan View along Line II - II of Figure A.4.4.1

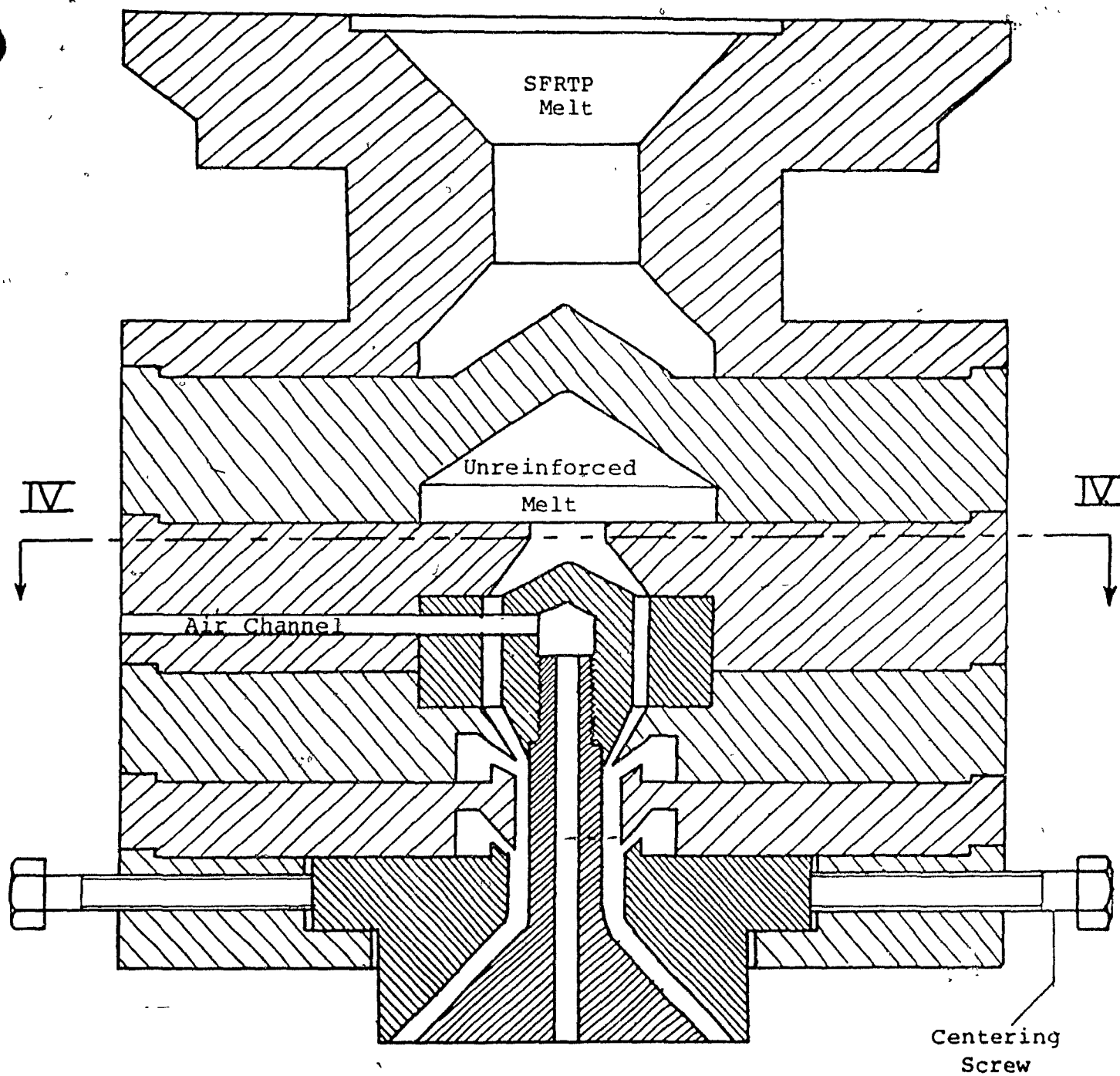


Figure A.4.4.3 Sectional Plan View along Line III - III of Figure A.4.4.1.

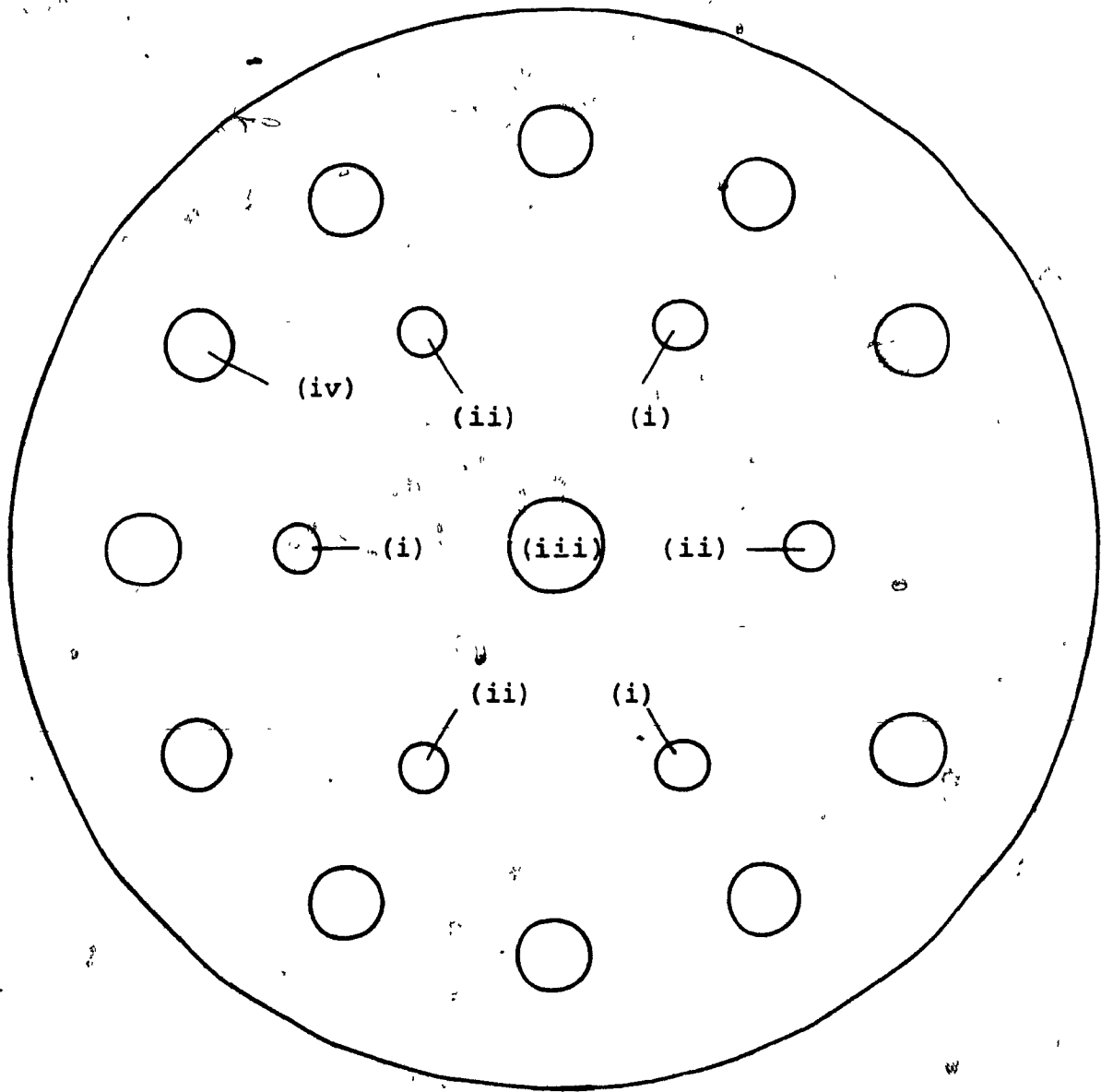


Figure A.4.4.4 Sectional View along Line IV - IV of
Figures A.4.4.2 and A.4.4.3.

- (i) SF RTP Melt Channel (Middle Layer)
- (ii) Unreinforced Melt Channel (Outer Layer)
- (iii) Unreinforced Melt Channel (Inner Layer)
- (iv) Holes for Bolts

Appendix 4.5

Tensile Strength Data

(a) Axial

Conditions of Extrusion	σ_y MPa	Mean σ_y MPa	95% Confidence MPa
A	21.7, 21.4, 22.9, 27.9, 28.7, 23.6, 26.3	24.7	± 2.7
B	26.7, 23.8, 29.3, 27.8, 26.1, 25.5, 28.1, 26.3, 28.2	26.9	± 1.3
C	30.5, 32.2, 33.0, 34.2 33.1, 34.5, 35.8, 31.4	33.1	± 1.5
D	24.7, 27.2, 25.4, 28.5 26.1	26.4	± 1.9
Simple Extrusion	37.5, 37.5, 39.4, 38.8	38.3	± 1.5

(b) Hoop

A	19.9, 19.4, 19.7, 22.1, 20.7, 21.8, 23.6, 21.5, 22.4, 23.5, 21.2, 21.3, 21.5, 22.2	21.5	± 0.7
B	22.6, 23.0, 23.4, 27.0, 22.9, 22.6, 26.1, 25.0, 24.5, 25.3, 23.3, 24.9	24.5	± 0.8
C	27.1, 27.2, 27.5, 24.0, 27.0, 24.3, 26.3, 26.3, 25.6, 22.9, 26.2, 25.7, 25.7, 25.6, 26.3	25.9	± 0.7
D	30.5, 26.5, 29.3, 28.7, 26.7, 31.2, 25.2, 24.6, 25.8, 29.6, 25.2, 29.5	27.7	± 1.5
Simple Extrusion	17.0, 16.0, 15.9, 16.4, 17.2	16.5	± 0.7

Appendix 5.1

Standard Test Methods for Plastic Pipe

Annual Book of Standards (Vol. 9, Sec. 8) published by the American Society for Testing and Materials (ASTM) describes several tests to evaluate and rank piping materials and to measure properties that can be used for designing piping systems. A brief description of the important tests is presented below.

Tests D1598, D1599, D2837 and D2992 are intended specifically for pressure pipe. Test D1598 describes a method to determine the relationship between stress due to internal hydraulic pressure and time to failure for plastic pipe. The test consists of subjecting pipe specimens to different internal pressures so as to cause different levels of wall stress under controlled environmental conditions. The time to failure is recorded for each specimen. The stress-time to failure relationship can be used to determine the hydrostatic design basis for the pipe as described by method D2837.

Test D1599 describes a short term burst stress determination under internal pressurization of a pipe specimen or a complete piping system including fittings. The pressure is increased continuously until failure occurs so as to obtain the maximum stress the pipe specimen or the piping system can withstand.

However, the long term strength is usually considerably lower than the short term strength obtained during the burst test described above. ASTM D2837 describes a method of determining the long term hydrostatic strength (LTHS). Several pipe specimens are subjected to different levels of stress (different internal pressures) and the corresponding failure times are recorded, as described in the test method D1598. The test is carried out for failure times up to at least 10 000 hours. The stress-time to failure data are then plotted using log scales and a regression straight line through the data is extrapolated to 100 000 hours. The stress corresponding to 100 000 hours is taken to represent the LTHS of the pipe. A hydrostatic design basis (HDB) can be determined from the LTHS and used for designing piping systems.

Instead of subjecting pipe specimens to steady internal pressure as in D1598 or D2837, they may also be subjected to a cyclic internal pressure so as to determine the number of cycles to failure as described in the test D2143. Mean internal pressure-cycles to failure data can then be extrapolated to 150×10^6 cycles to obtain the long term hydrostatic strength (LTHS) as described in test D2992.

While all of the above tests are based on internal pressurization of pipe specimens, several other tests have also been standardized to characterize the performance of pipe under a variety of different loading conditions.

Test method D2290 describes a method of determining the hoop strength of a pipe material. A ring shaped specimen is machined so as to reduce its width at two diametrically opposite points. The ring then is subjected to tensile testing using a split disc fixture described in the test such that the two diametrically reduced areas act as gauge sections, and the tensile stress at the failure is determined to obtain the hoop strength of the pipe.

Similarly, test D2105 describes a method of determining the axial tensile properties of the pipe. A length of the pipe is subjected to axial tensile loading and various properties such as stiffness, yield strength and yield strain are determined from the load-displacement record of the test.

Underground pipe and conduit are often subjected to lateral crushing loads. Test method D2402 describes a method to determine the resistance of pipe to such loads. A length of the pipe is held longitudinally between two parallel plates. The plates are made to approach each other at a controlled rate crushing the pipe specimen laterally, and the load-deflection record is obtained. The slope of the record is a measure of the pipe stiffness against such loads.

Many times during shipping, handling, installation and/or in service, pipe is subjected to unforeseen impacts. Impact properties are of particular importance in the case of brittle

pipe such as rigid PVC conduit or pipe made of certain thermosetting materials. ASTM D2444 provides a method to measure the resistance of the pipe under impact by means of a falling tup. The probability of failure under impact can be determined from the data obtained in the test.

Pipe may also be subjected to conditions of differential pressures such that the external pressure exceeds the internal pressure. ASTM method D2586 and D2924 describe tests in which a pipe specimen is subjected to external pressure in a chamber. Internal volume changes and the maximum failure stress due to the external pressure can be determined in these tests.

Appendix 5.2

New Test Methods for Plastic Pipe

As described in Appendix 5.1, ASTM method D2837 describes a procedure for determining the long term hydrostatic strength (LTHS) by extrapolation of stress versus time to failure data. The method, however, does not differentiate between ductile and brittle modes of failure. As discussed in Section 5.1, field failures of plastic pipe usually involve crack propagation in the brittle mode. Hence, several tests have recently been proposed that focus on resistance to crack growth and attempt to simulate brittle type failures.

One approach to the development of such tests is based on the activated rate process or Arrhenius theory of failure (147). According to this theory, the failure of a material is assumed to be a rate process impeded by an energy barrier. The overcoming of this barrier and, hence, the rate of failure is determined by the applied stress and the absolute temperature at which the failure occurs. Based on this theory, Bragaw (101) has suggested a rate process method for determining the LTHS of plastic pipe. In this method, the internal pressure testing of the pipe specimens is carried out at several temperatures (usually 80°C, 65°C and 50°C) in addition to the room temperature. While failure at room temperature usually occurs in the ductile mode, that at higher temperature may occur in either the ductile or brittle mode. The

stress versus failure time data thus obtained at various temperatures are correlated statistically using the following equation.

$$\log t = A + \frac{B}{T} + C \log P \quad (\text{A.5.2.1})$$

where t is the failure time

P is the applied pressure

T is the absolute test temperature ($^{\circ}\text{K}$)

and A, B, C are the coefficients obtained by multiple regression.

Knowing the coefficients, the equation can be used to determine the long term hydrostatic strength corresponding to a temperature of 300°K (23°C) and a failure time of 50 years (148).

A similar method commonly used in Europe has been suggested by International Standards Organization (ISO). This method, called the Standard Extrapolation Method, uses the stress versus time to failure data at various temperatures to evaluate the coefficients of the following equation.

$$\log t = A + \frac{B}{T} + C \frac{\log P}{T} \quad (\text{A.5.2.2})$$

The high temperature data often exhibit a knee indicating a ductile-brittle transition as shown schematically in Figure A.5.2.1. It is assumed that such a knee also occurs at room temperature (20°C), but at a longer failure time. Thus, the extrapolation at 20°C is carried out such that the contour is similar to that for the higher temperature data. The stress at

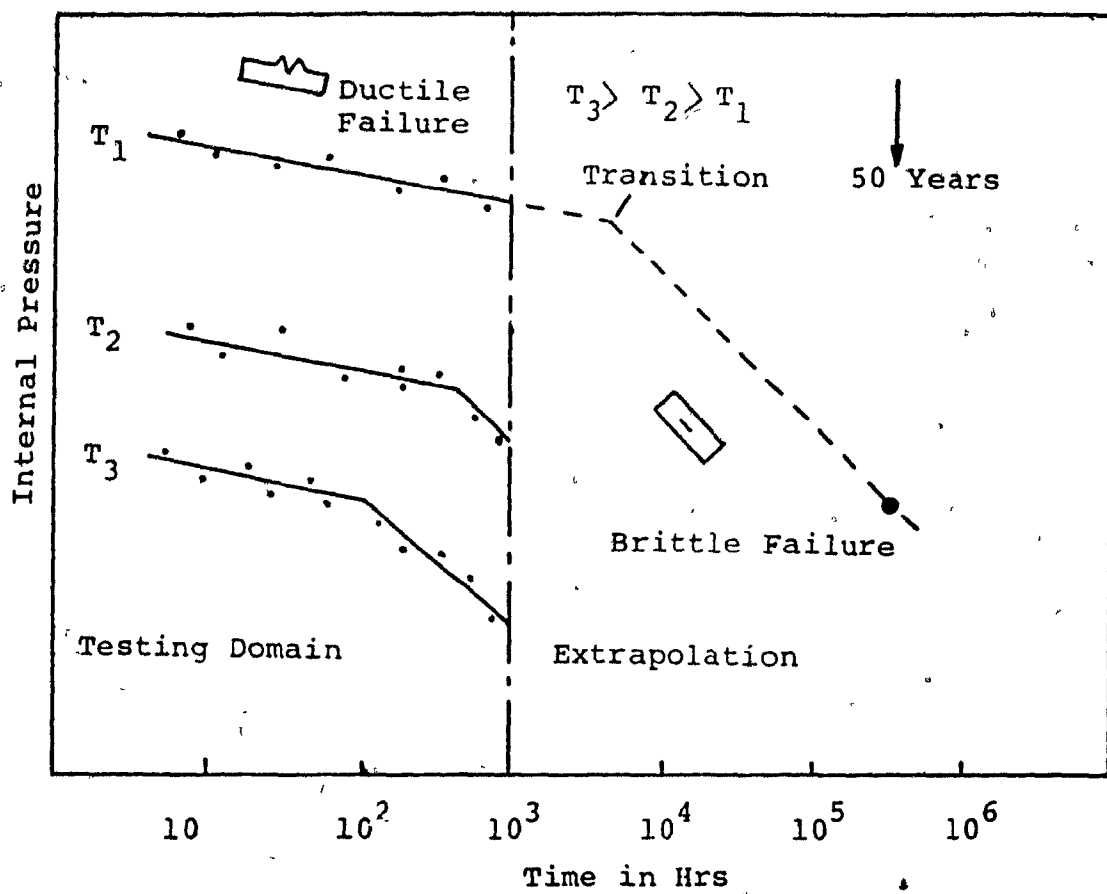


Figure A.5.2.1 Knee Characterizing Ductile-Brittle Transition in Internal Pressure Testing of Plastic Pipe.

20°C, 50 years failure time is taken to be the LTHS. Several variations of the equation used to fit the multi - temperature data have recently been proposed that incorporate three to six coefficients (102). Also, the Plastic Pipe Institute (PPI) has recently proposed the use of similar methods to validate the LTHS determined by the standard ASTM D2837 method (149).

The so-called "Logit" method, common in France, does not distinguish between ductile and brittle failures. The data obtained at a single temperature (usually room temperature) are fitted to the following equation (98).

$$\log \frac{P}{P_{\max} - P} = A + B \log t \quad (A.5.2.3)$$

Here $P_{\max}$ is the short term burst pressure at the same temperature. Extrapolation to 50 years then gives the LTHS.

All of the above tests are based on the internal pressurization of the pipe. Several tests involving direct crack extension from an existing notch under imposed stresses have also been proposed. The slow crack growth (SCG) test involves the use of "C" shaped test pieces prepared from the pipe (104). Several test pieces are notched and loaded to create crack extension in Mode I through their thickness. Plots of crack extension versus time and applied stress intensity factor versus apparent crack growth rate are claimed to be useful in ranking piping materials.

The Constant Tensile Load (CTL) test (105) involves cutting

a ring from pipe, notching it at diametrically opposite points and subjecting it to a tensile load using a split disc fixture to generate the stress versus time to failure data. The failure can be ductile or brittle depending on the loading conditions. The failure process can be significantly accelerated by immersing the specimen in a solution of a surface active stress cracking agent such as Igepal or by increasing the test temperature. The stress versus failure time data obtained in this manner have been shown to exhibit a knee, corresponding to the ductile-brittle transition, similar to that found in the high temperature internal pressure tests described above (Figure A.5.2.1). The test has thus been claimed to simulate brittle failures in plastic pipe and the results are considered to be useful for quality control and the relative ranking of the materials.

Tests have also been proposed to measure the Rapid Crack Propagation (RCP) in plastic pipe (100). An adequately supported pipe specimen containing an axial notch is struck by a hammer to dynamically initiate the propagation of a high speed crack. The crack arresting characteristics of the pipe can be studied and used to design piping systems to avoid RCP.

Appendix 5.3

Specifications for the Brabender Extruder

Screw diameter = 19 mm(0.75 in)

Screw L / D Ratio = 25 : 1

Screw Type - Single Stage

Screw Compression Ratio = 3 : 1

Barrel Heating / Cooling : 3 Zones

Electrical Heating

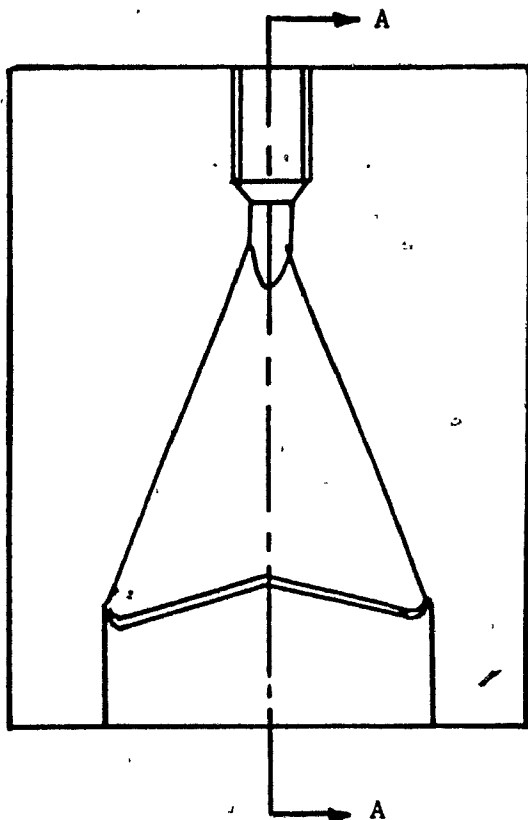
Air Cooling

Die Heating :

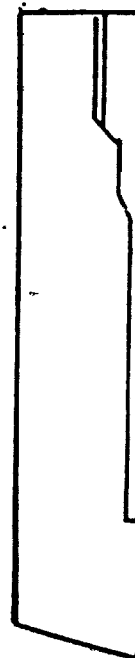
Single Zone

Electrical Heating

No Cooling



Plan



Section AA

Sectional Side View

Figure A.5.3.1 Sheet Die Used to Make Extruded Strips.

Appendix 5.4

Flexural Stiffness Data

f = Fractional thickness of SF RTP layer

a) Test Pieces in the L direction

f	E_f (MPa)	Mean E_f (MPa)	95% Confidence Limit (MPa)
0	1195, 1242, 1299, 1253	1247	± 68
0.1	1130, 1224, 1075, 1206	1159	± 109
0.35	1422, 1192, 1168, 1246	1257	± 182
0.52	1617, 1512, 1549, 1555	1558	± 69
0.68	2077, 1987, 1891, 1936	1973	± 127
1.0	3997, 4341, 4352, 4447, 4136	4254	± 227

b) Test Pieces in the T Direction

f	E_f (MPa)	Mean E_f (MPa)	95% Confidence Limit (MPa)
0	1281, 1198, 1232, 1299	1252	± 73
0.1	1126, 1231, 1165, 1082	1151	± 100
0.35	1322, 1322, 1322, 1322, 1323	1322	± 0.8
0.52	1079, 1170, 1116, 1193	1140	± 82
0.68	1126, 1165, 1066, 1231, 1082	1134	± 106
1.0	2129, 2171, 2119, 2294, 2300, 2140	2192	± 132

Appendix 5.5.1

Tensile Strength Data

f = Fractional thickness of SF RTP layer

a) Test Pieces in the L direction

f	σ_y (MPa)	Mean σ_y (MPa)	95% Confidence Limit (MPa)
0	22.3, 21.7, 21.73, 21.61	21.83	± 0.5
0.1	23.62, 24.05, 23.21, 23.68	23.73	± 0.55
0.35	28.78, 25.7, 26.76, 26.7	26.98	± 2.05
0.52	32.28, 30.82, 30.85, 32.92	31.72	± 1.67
0.68	36.38, 35.83, 35.43, 36.11	35.94	± 0.64
1.0	41.05, 42.84, 40.5, 38.08	40.61	± 3.12

b) Test Pieces in the T Direction

f	σ_y (MPa)	Mean σ_y (MPa)	95% Confidence Limit (MPa)
0	21.78, 21.88, 21.79, 21.56	21.75	± 0.22
0.1	23.51, 23.66, 25.1, 25.0, 23.72	24.2	± 0.97
0.35	24.50, 23.18, 23.94, 24.10, 23.4	23.82	± 0.672
0.52	23.95, 23.69, 22.80, 22.95	23.35	± 0.89
0.68	25.5, 25.37, 24.77, 25.1, 24.56	25.06	± 0.49
1.0	24.87, 25.37, 27.5, 25.37, 25.94, 27.4	26.07	± 1.17

Appendix 5.5.2

Apparent Yield Strain Data

f = Fractional thickness of SF RTP layer

a) Test Pieces in the L direction

f	t_y (%)	Mean t_y (%)	95% Confidence Limit (%)
0	12.91, 12.15, 13.28, 13.71	13.01	± 1.05
0.1	10.97, 10.70, 10.22, 10.60	10.62	± 0.49
0.35	5.19, 6.63, 5.58, 6.54	5.98	± 1.13
0.52	4.85, 5.26, 5.18, 4.93	5.05	± 0.31
0.68	3.68, 3.89, 3.84, 3.67	3.77	± 0.18
1.0	3.56, 3.59, 3.62, 3.52	3.57	± 0.07

b) Test Pieces Taken in the T Direction

f	t_y (%)	Mean t_y (%)	95% Confidence Limit (%)
0	13.63, 13.08, 13.56, 13.23	13.38	± 0.42
0.1	12.19, 12.23, 11.02, 11.15, 11.4	11.59	± 0.72
0.35	8.02, 7.63, 7.74, 7.3, 7.78	7.7	± 0.32
0.52	7.11, 7.45, 7.48, 7.3	7.33	± 0.27
0.68	5.46, 5.48, 5.67, 5.39, 5.24	5.45	± 0.19
1.0	4.33, 4.41, 4.43, 4.29, 4.15, 4.22	4.3	± 0.11

Appendix 5.6

J - Integral Data

(a) High Density Unreinforced Resin (PE 2906 M6)

Test Piece	Dimensions		a_0 mm	J KJ/m ²	Δa mm
	B mm	D mm			
1	6.70	12.87	5.19	1.92	0.31
2	6.615	12.86	5.31	1.67	0.418
3	6.67	12.75	5.43	2.12	0.623
4	6.62	12.88	5.23	2.13	0.81
5	6.75	12.81	5.42	2.54	0.95
6	6.69	12.82	5.43	2.4	1.22
7*	6.735	12.78	5.99	3.3	1.658
8*	6.66	12.76	5.55	4.47	2.73

Regression Line Through the Data Points

$$J = 0.785 \Delta a + 1.56 \quad (A.5.6.1)$$

Blunting Line

$$J = 43.63 \Delta a \quad (A.5.6.2)$$

(J in KJ/m² and Δa in mm)

At the Point of Intersection,

$$J_{IC} = 1.59 \text{ KJ/m}^2$$

* Points Excluded in the Calculations.

J - Integral Data

(b) SF RTP (PE 2906 M6 + 30% Fibers)

Test Pieces in the L Direction.

Test Piece	Dimensions		a_0 mm	J KJ/m ²	Δa mm
	B mm	D mm			
1	6.8	12.98	5.73	3.94	0.207
2	6.9	12.31	5.58	4.41	0.495
3	6.77	13.01	5.61	6.36	0.869
4	6.6	13.28	5.69	6.3	0.89
5	6.75	12.98	5.69	6.23	1.1
6*	6.83	13.00	5.7	9.42	2.06

Regression Line Through the Data Points

$$J = 3.104 \Delta a + 3.24 \quad (\text{A.5.6.3})$$

Blunting Line

$$J = 81.24 \Delta a \quad (\text{A.5.6.4})$$

(J in KJ/m² and Δa in mm)

At the Point of Intersection,

$$J_{IC} = 3.36 \text{ KJ/m}^2$$

* Point Excluded in the Calculations.

J - Integral Data

(c) SF RTP (PE 2906 M6 + 30% Fibers)

Test Pieces in the T Direction.

Test Piece	Dimensions		a_0 mm	J KJ/m ²	Δa mm
	B mm	D mm			
1	6.81	13.41	5.83	1.74	0.485
2	6.68	12.86	5.69	1.45	0.494
3	6.54	13.31	5.55	1.57	0.58
4	6.46	13.11	5.63	2.86	0.818
5	6.44	12.79	5.66	3.38	0.938
6	6.71	12.97	5.65	3.7	0.943
7	5.78	12.9	5.41	5.2	1.35
8*	6.74	13.08	5.79	7.27	1.827

Regression Line Through the Data Points

$$J = 3.09 \Delta a + 0.12 \quad (\text{A.5.6.5})$$

Blunting Line

$$J = 50.25 \Delta a \quad (\text{A.5.6.6})$$

(J in KJ/m² and Δa in mm)

At the Point of Intersection,

$$J_{IC} = 0.125 \text{ KJ/m}^2$$

* Point Excluded in the Calculations.

J - Integral Data

d) Low Density Unreinforced Resin (PE 74 A)

Test Piece	Dimensions		a_0 mm	J KJ/m ²	Δa mm
	B mm	D mm			
1	6.50	12.76	5.73	10.37	0.45
2	6.66	12.74	5.79	15.61	0.67
3	6.81	12.66	5.76	23.70	0.9
4	6.60	12.85	5.81	26.18	0.952
5	6.47	12.84	5.64	34.47	1.11

Regression Line Through the Data Points

$$J = 35.79 \Delta a - 7.16 \quad (\text{A.5.6.7})$$

Blunting Line

$$J = 26.05 \Delta a \quad (\text{A.5.6.8})$$

(J in KJ/m² and Δa in mm)

No J_{IC} Value Could be Determined.

APPENDIX 5.7
BASIC PROGRAM FOR CALCULATING COMPLIANCE-CRACK LENGTH
RELATIONSHIP FOR SINGLE AND MULTI-LAYER TEST PIECES

```

140 DIM F1(50),R3(50)
150 REM CALCULATION OF COMPLIANCE AS A FUNCTION OF CRACK-LENGTH
160 REM (A/D=P) FOR A THREE POINT BEND SPECIMEN
170 REM R1 = RATIO OF THE STIFFNESS OF THE FILLED LAYER TO THAT OF
180 REM OF THE UNFILLED LAYER
190 REM R2 = THICKNESS OF THE FILLED LAYER AS A FRACTION OF THE TOTAL
200 REM THICKNESS D.
210 REM C0 = THE NON-DIMENSIONAL COMPLIANCE DEFINED AS
220 REM (1/SLOPE)*E(UNFILLED)*B(THICKNESS)
230 PRINT "SPECIMEN - "; \ INPUT M$
240 PRINT "C0 = "; \ INPUT C0
250 PRINT "R1 = "; \ INPUT R1
260 PRINT "R2 = "; \ INPUT R2
270 PRINT "D IN INCHES = "; \ INPUT D
280 P=0
290 PRINT "P = ";P,,, "C0 = ";C0
300 FOR I=1 TO 37
310 P=(I-1)/40
320 A=2*(1+2*P)*((1-P)^1.5)
330 B=1.99-P*(1-P)*(2.15-3.93*P+2.7*P*P)
340 C=3*B*SQR(P)
350 F=C/A
360 GO TO 560
370 F1(I)=F*F/R3(I)
380 NEXT I
390 REM INTEGRATION BY SIMPSON'S RULE
400 FOR I=5 TO 37 STEP 4
410 I1=I-1
420 P=(I-1)/40
430 S1=2*((2/D)^2)*F1(I)
440 S2=F1(1)
450 FOR J=2 TO I STEP 2
460 S2=S2+4*F1(J)
470 NEXT J
480 FOR J=3 TO I1 STEP 2
490 S2=S2+2*F1(J)
500 NEXT J
510 S2=(S2+F1(1))*0.25/3
520 C1=C0+2*((2/D)^2)*S2
530 PRINT "P=";P,"DCDA=";S1,,, "C1=";C1,"ER=";R3(I)
540 NEXT I
550 GO TO 710
560 IF R1=1 THEN 670
570 IF R2>(1-P) THEN 690
580 T1=(1-P-R2)^2+R1*R2*(2*(1-P)-R2)
590 T2=1-P+R2*(R1-1)
600 Y=.5*T1/T2
610 T3=(1-R2-P)^2+3*((2*Y-1+R2+P)^2)
620 T4=T3*(1-R2-P)/((1-P)^3)
630 T5=R2*R2+3*((2-R2-2*P-2*Y)^2)
640 T6=R1*R2*T5/((1-P)^3)
650 R3(I)=T4+T6
660 GO TO 700
670 R3(I)=1
680 GO TO 700
690 R3(I)=R1
700 GO TO 370
710 END

```


Appendix 5.8

R - Curve Data for Single-Layer Test Pieces

(a) High Density Unreinforced Resin (PE 2906 M6)

Test Piece No. 1

B = 6.77 mm D = 12.74 mm
(a/D)₀ = 0.38 C₀EB = -30.196

Load Kg	CEB	(a/D) _{eff}	K _R MPa/m
6.0	31.275	0.388	0.59
8.0	32.622	0.4	0.811
10.0	33.86	0.405	1.03
11.0	34.9	0.42	1.18
11.4	35.38	0.424	1.24
11.8	36.74	0.434	1.32
11.95	38.08	0.448	1.39
11.96	39.3	0.452	1.41
11.8	40.76	0.458	1.41
11.4	43.89	0.48	1.46
11.0	46.67	0.49	1.45
10.6	49.85	0.508	1.48
10.0	54.78	0.522	1.46
9.4	60.35	0.542	1.47
9.0	64.61	0.557	1.48
8.6	68.97	0.572	1.49
8.0	76.57	0.59	1.49
7.0	93.36	0.623	1.48
6.0	116.83	0.662	1.5

Test Piece No. 2

B = 6.58 mm D = 12.86 mm
(a/D)₀ = 0.498 C₀EB = 45.0

Load Kg	CEB	(a/D) _{eff}	K _R MPa/m
5.0	46.04	0.504	0.7
6.0	47.09	0.509	0.855
7.0	48.14	0.513	1.01
8.0	49.7	0.52	1.18
9.0	51.86	0.527	1.36
10.0	54.41	0.533	1.54
10.6	56.27	0.54	1.67
10.0	62.37	0.56	1.69
9.0	72.78	0.59	1.7
8.0	86.07	0.615	1.7
7.0	104.34	0.648	1.66
6.0	131.15	0.685	1.67
5.0	172.03	0.72	1.69
4.0	243.82	0.759	1.69

Test Piece No. 3

B = 6.58 mm

D = 12.85 mm

$(a/D)_0 = 0.448$

$C_{0EB} = 41.89$

Load
Kg

CEB

$(a/D)_{eff}$

K_R
MPa/m

10.0	46.07	0.473	1.273
12.0	49.56	0.49	1.61
12.1	51.32	0.5	1.67
11.0	62.26	0.54	1.73
10.0	71.83	0.57	1.75
9.0	83.3	0.598	1.75
8.0	99.22	0.628	1.75
6.0	155.32	0.7	1.82
5.0	209.42	0.74	1.88
4.0	302.62	0.78	1.94
3.0	487.26	0.825	2.06

Test Piece No. 4

B = 6.55 mm

D = 12.68 mm

$(a/D)_0 = 0.18$

$C_{0EB} = 22.24$

Load
Kg

CEB

$(a/D)_{eff}$

K_R
MPa/m

17.5	23.17	0.20	1.09
20.0	23.98	0.22	1.32
22.5	24.25	0.23	1.52
24.6	24.47	0.23	1.52
22.5	29.55	0.31	1.87
20.0	33.57	0.353	1.86
17.5	39.33	0.404	1.87
15.0	46.71	0.453	1.84
12.5	57.92	0.51	1.83
10.0	76.32	0.572	1.81
7.5	110.11	0.64	1.77
5.0	190.2	0.726	1.78
3.0	358.7	0.797	1.69

(b) SF RTP (PE 2906 M6 + 30% Fibers)

Test Pieces in the L Direction.

Test Piece No. 1

B = 6.49 mm D = 12.73 mm
(a/D)₀ = 0.438 C₀EB = 60.265

Load Kg	CEB	(a/D) _{eff}	K _R MPa√m
15.0	61.17	0.445	1.80
17.5	62.84	0.457	2.18
20.0	64.76	0.468	2.58
22.5	67.76	0.484	3.05
25.0	73.13	0.51	3.67
26.5	83.0	0.543	4.35
26.9	94.8	0.578	4.99
26.82	105.14	0.602	5.45
26.5	118.63	0.625	5.9
26.0	129.22	0.643	6.25
25.0	147.88	0.67	6.77
22.5	193.39	0.71	7.41
20.0	229.7	0.734	7.51
17.5	284.11	0.763	7.82
15.0	361.59	0.792	8.17
10.0	661.8	0.845	8.49
7.5	996.83	0.873	8.61

Test Piece No. 2

B = 6.56 mm D = 12.64 mm
(a/D)₀ = 0.443 C₀EB = 60.0

Load Kg	CEB	(a/D) _{eff}	K _R MPa√m
15.0	60.46	0.445	1.81
20.0	63.75	0.46	2.52
22.5	66.67	0.474	2.96
25.0	74.73	0.51	3.68
25.75	86.32	0.555	4.40
25.5	106.69	0.605	5.25
25.0	121.10	0.63	5.69
24.0	141.77	0.663	6.29
22.5	166.08	0.69	6.7
20.0	206.61	0.72	6.95
17.5	254.83	0.748	7.13
15.0	320.48	0.776	7.3
12.5	418.94	0.808	7.7
10	583	0.835	7.73
7.5	901.9	0.868	8.12

(c) SFRTTP (PE 2906 M6 + 30% Fibers)

Test Pieces in the T Direction.

Test Piece No. 1 B = 6.61 mm D = 12.0 mm
(a/D)₀ = 0.4 C₀EB = 50.4

Load Kg	CEB	(a/D) _{eff}	K _R MPa√m
10.0	52.11	0.415	1.18
12.5	54.4	0.43	1.55
15.0	58.9	0.46	2.02
17.5	70.96	0.513	2.78
18.75	93.62	0.584	3.81
18.75	109.19	0.615	4.30
17.5	142.76	0.668	5.03
15.0	190.70	0.710	5.29
10.0	347.8	0.787	5.63
5.0	928.34	0.876	6.3

Test Piece No. 2 B = 6.37 mm D = 11.36 mm
(a/D)₀ = 0.4 C₀EB = 60.19

Load Kg	CEB	(a/D) _{eff}	K _R MPa√m
12.5	65.53	0.43	1.74
15.0	74.08	0.472	2.36
17.0	104.96	0.565	3.63
17.0	116.06	0.587	3.94
16.0	143.13	0.634	4.46
15.0	163.35	0.656	4.6
12.5	221.38	0.706	4.87
10.0	308.1	0.752	5.04
8.5	392.6	0.781	5.17
6.5	581.35	0.822	5.41
5.0	874.7	0.858	5.85

Test Piece No. 3

B = 5.82 mm D = 12.76 mm
 $(a/D)_0 = 0.414$ $C_{0EB} = 42.94$

Load Kg	CEB -	$(a/D)_{eff}$ -	K_R MPa $\sqrt{m}$
10.0	43.59	0.416	1.23
15.0	47.71	0.444	2.0
17.5	57.26	0.492	2.7
18.5	68.4	0.538	3.31
18.6	72.94	0.552	3.49
18.5	79.66	0.57	3.7
17.5	95.55	0.608	4.04
15.0	126.23	0.658	4.27
12.5	170.21	0.703	4.41
10.0	231.89	0.744	4.41
7.5	354.38	0.793	4.56
5.0	639.6	0.848	4.86

Appendix 5.9

Derivation of the Equivalent Flexural Modulus $E(a/D)$

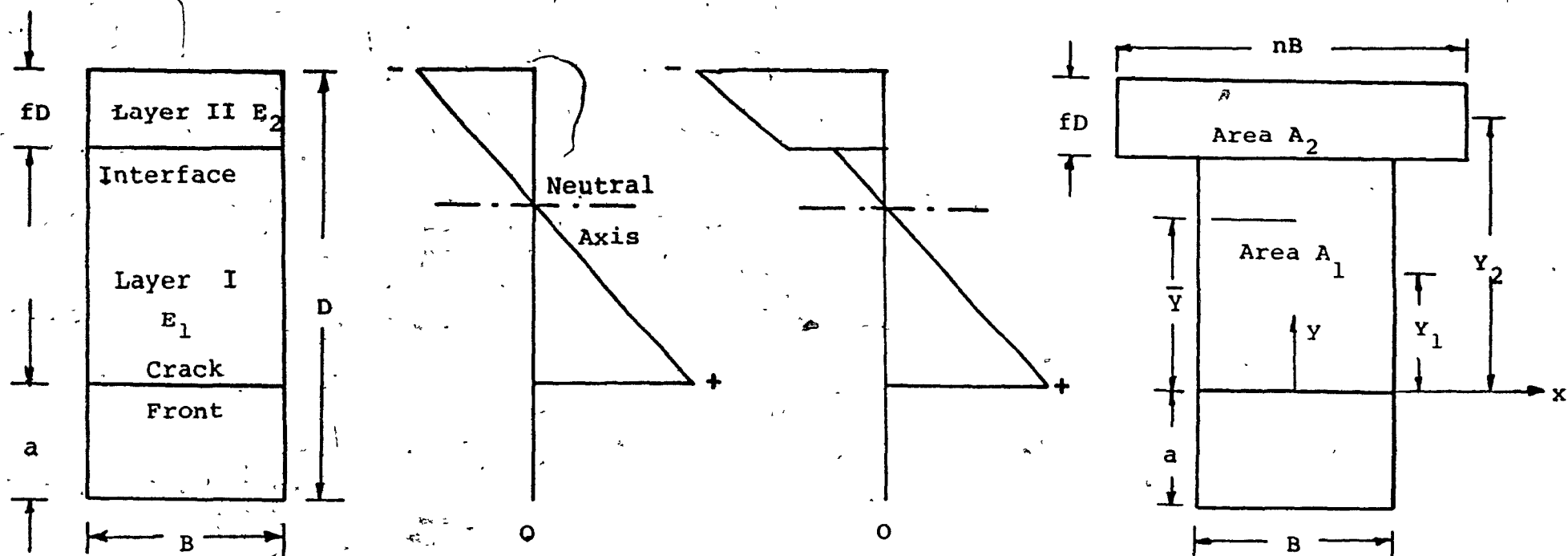
A cross section of the two-layer test piece used in the crack growth studies is shown in Figure A.5.9.1a. The test piece has a width B and a total depth D . A crack of length a is present in layer I having modulus E_1 . The fractional thickness of layer II is f , and its modulus is E_2 . It is desired to determine the flexural modulus $E(a/D)$ of a section of a homogeneous material of the same dimensions as the actual two-layer ligament (width B x depth $(D-a)$) and having an equivalent in flexural rigidity. The moment of inertia of such a ligament about its centroidal axis $I(a/D)$ is as follows.

$$I(a/D) = \frac{B (D - a)^3}{12} \quad (A.5.9.1)$$

The flexural rigidity of this equivalent homogeneous ligament, which is the product of its flexural modulus and the moment of inertia about its centroidal axis, is as follows.

$$\text{Flexural Rigidity} = E(a/D) \frac{B D^3 (1 - a/D)^3}{12} \quad (A.5.9.2)$$

It is desired to obtain an expression for $E(a/D)$ in terms of the layer thicknesses, test piece and crack dimensions, and layer moduli.



(a) Test Piece Cross-section

(b) Strain Distribution

(c) Stress Distribution

(d) Equivalent Section

Figure A.5.9.1 Equivalent Section of a Two Layer Test Piece.

The following derivation is based on classical theory of elastic bending of multi-layer or composite beams (150). According to this theory, bending strain is assumed to vary linearly with distance from the neutral axis and a perfect bonding is assumed between the adjacent layers so that the strains in the two layers at the interface are equal. The strain distribution is shown schematically in Figure A.5.9.1b.

Since the moduli of the two layers are different, the stresses cannot be equal in both of them at the interface. Assuming that the deflections involved are within the elastic limits for both layers, the stress distribution is as shown schematically in Figure A.5.9.1c. The stress in layer II at the interface is n times that in layer I, where n is the ratio of the modulus of layer II to that of layer I.

$$n = E_2/E_1 \quad (A.5.9.3)$$

In order to determine the flexural modulus $E(a/D)$ of the equivalent homogeneous section, it is convenient to replace the layer II of the actual ligament by a section consisting of the material of layer I such that the forces on the tensile and compressive sides of the neutral axis and their moments about the neutral axis are the same as those for the actual two-layer ligament. This can be done by substituting a section of the material of layer I of depth equal to that of layer II; but width n times that of layer II. The substituted section thus has dimensions of $nB \times fD$ and the overall replaced section of material I is shown in Figure A.5.9.1d.

In order to determine the flexural rigidity of this replaced section of the layer I material, it is necessary to locate its centroid. A cartesian frame of reference is chosen with x-axis along the crack front. It is convenient to divide the overall section into two rectangles representing the layer I and the substituted layer II, as shown in Figure A.5.9.1d. The centroids of these rectangles are located at distances Y_1 and Y_2 with respect to the x-axis and their areas are A_1 and A_2 respectively.

$$A_1 = B [(1-f)D - a]$$

$$A_2 = B f D (E_2/E_1)$$

$$Y_1 = [(1-f)D - a]/2$$

$$Y_2 = (1-f)D - a + (fD)/2 \quad (A.5.9.4)$$

The distance $\bar{Y}$ of the centroid of the overall section from the x-axis is given as follows.

$$\bar{Y} = \frac{A_1 Y_1 + A_2 Y_2}{A_1 + A_2} \quad (A.5.9.5)$$

Substituting values of A , A_1 , A_2 , Y_1 and Y_2 ,

$$\bar{Y} = \frac{D}{2} \cdot \frac{\{1-f-(a/D)\}^2 + f (E_2/E_1) \{2-f-2(a/D)\}}{\{1-(a/D)\} + f \{(E_2/E_1)-1\}} \quad (A.5.9.6)$$

The next step is to determine the moment of inertia of the entire section about its centroidal axis parallel to the x-axis. The moments of inertia I_1 and I_2 of the previously defined rectangles about their respective centroidal axes are given as follows.

$$I_1 = \frac{B D^3}{12} [1 - f - (a/D)]^3 \quad (A.5.9.7)$$

$$I_2 = \frac{B D^3}{12} f^3 (E_2/E_1) \quad (A.5.9.8)$$

By use of the parallel axis theorem, the moments of inertia I_1' and I_2' of these rectangles about the centroidal axis of the overall section of Figure A.5.9.1d are given by:

$$\begin{aligned} I_1' &= I_1 + A_1 (\bar{Y}_1 - \bar{Y})^2 \quad (A.5.9.9) \\ &= \frac{B D^3}{12} [1 - f - (a/D)]^3 + \frac{B D^3}{4} [1 - f - (a/D)] \times \\ &\quad [2(\bar{Y}/D) - 1 + f + a/D]^2 \end{aligned}$$

$$\begin{aligned} I_2' &= I_2 + A_2 (\bar{Y}_2 - \bar{Y})^2 \quad (A.5.9.10) \\ &= \frac{B D^3}{12} f^3 (E_2/E_1) + \frac{B D^3}{4} f (E_2/E_1) \times \\ &\quad [2 - f - 2(a/D) - 2(\bar{Y}/D)]^2 \end{aligned}$$

The moment of inertia $\bar{I}$ of the overall section about its centroidal axis is simply the sum of the moments of inertia I_1' and I_2' .

$$\begin{aligned} \bar{I} &= I_1' + I_2' \quad (A.5.9.11) \\ &= \frac{B D^3}{12} [1 - f - (a/D)]^3 + \frac{B D^3}{4} [1 - f - (a/D)] [2(\bar{Y}/D) - 1 + f + (a/D)]^2 + \\ &\quad \frac{B D^3}{12} f^3 (E_2/E_1) + \frac{B D^3}{4} f (E_2/E_1) [2 - f - 2(a/D) - 2(\bar{Y}/D)]^2 \end{aligned}$$

The flexural rigidity of the overall section is equal to the product of its modulus E_1 and its moment of inertia $\bar{I}$. It is also equal to the flexural rigidity of the equivalent ligament of the homogeneous material given by Equation A.5.9.2

$$E(a/D) I(a/D) = E_1 \bar{I} \quad (\text{A.5.9.12})$$

Hence,

$$E(a/D) = \frac{E_1}{[1-(a/D)]^3} \{ [1-f-(a/D)]^3 + 3[1-f-(a/D)] \{ 2(\bar{Y}/D) - 1 + f + (a/D) \}^2 \} \\ + \frac{E_2}{[1-(a/D)]^3} [f^3 + 3f \{ 2-f-2(a/D) - 2(\bar{Y}/D) \}^2] \quad (\text{A.5.9.13})$$

The apparent modulus $E(a/D)$ is thus given in terms of the crack length, test piece dimensions, and the moduli of the individual layers. It may be noted that when the crack reaches the interface ($a/D = 1-f$), the apparent modulus $E(a/D)$ becomes equal to E_2 , the modulus of the layer II, as expected. A representative plot of $E(a/D)$ is shown in Figure A.5.9.2 based on the parameters indicated on the plot.

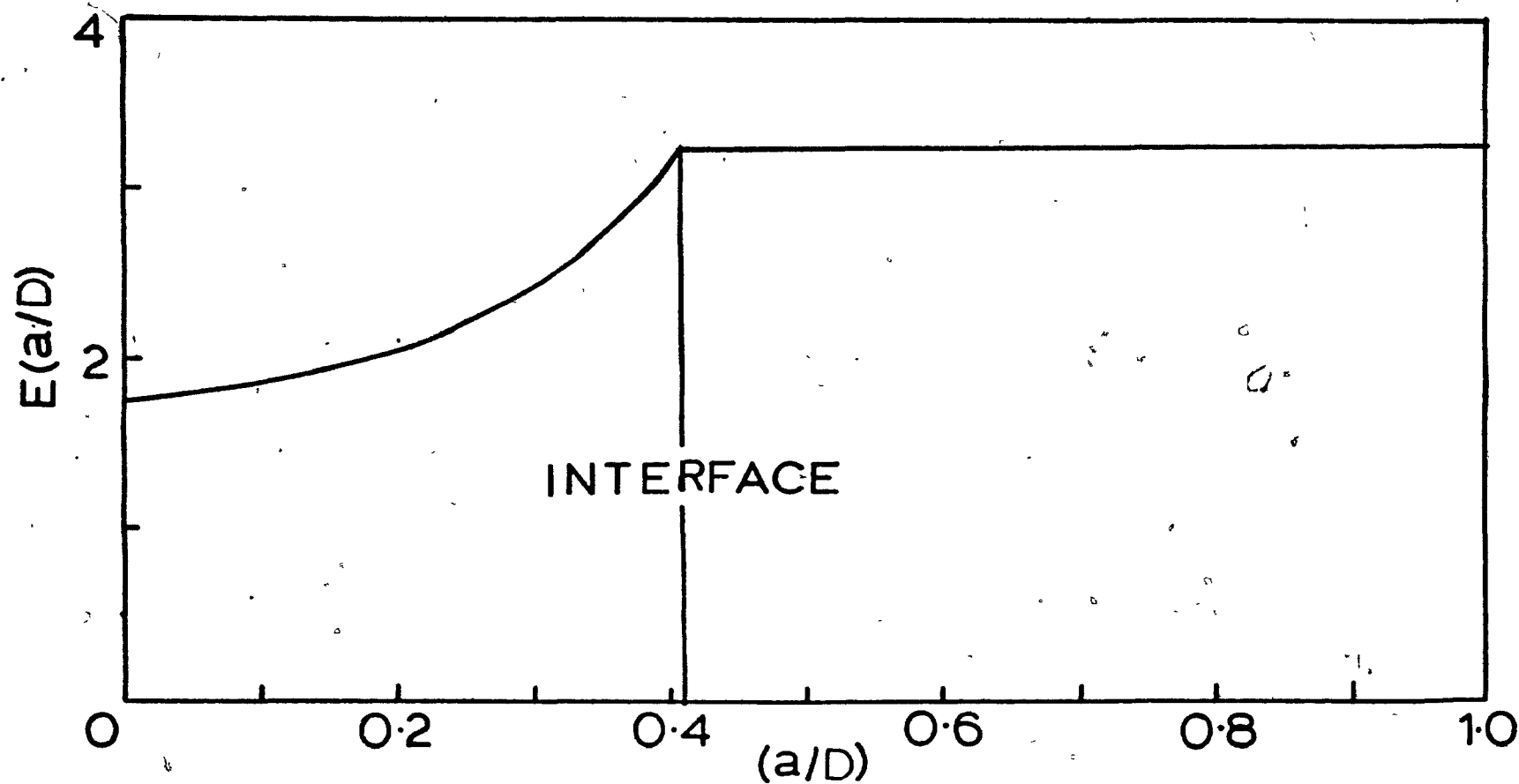


Figure A.5.9.2 Equivalent Flexural Modulus ($E(a/D)$) versus Dimensionless Crack Length (a/D).
($E_2/E_1 = 3.3$, $f = 0.58$)

APPENDIX 5.10

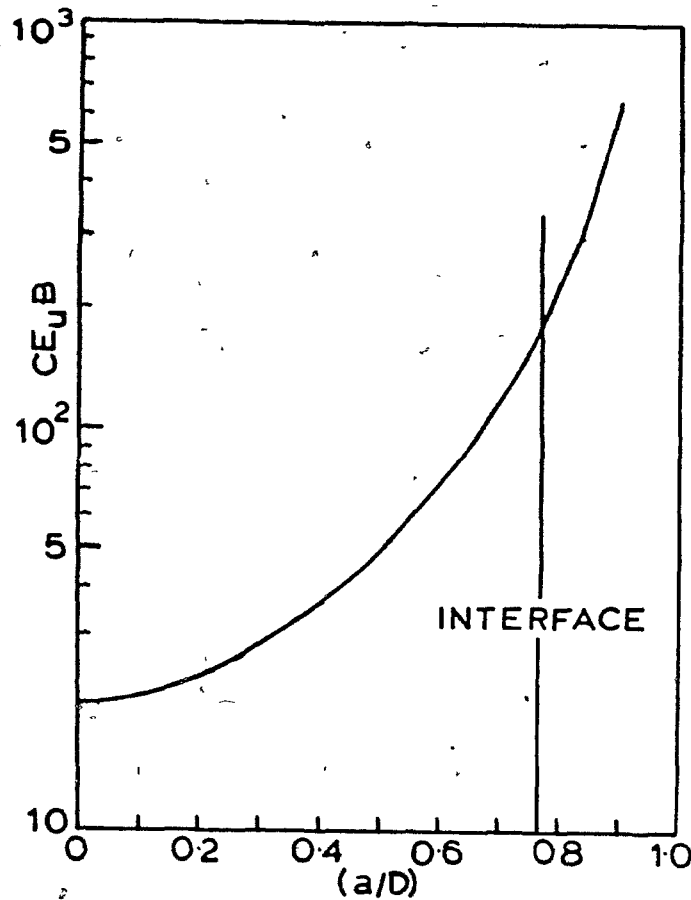


Figure A.5.10.1 CE_uB versus (a/D) Curve for Two Layer Test Pieces in the L Direction with $f = 0.23$. (Crack Propagation from Unreinforced to SF RTP Layer)

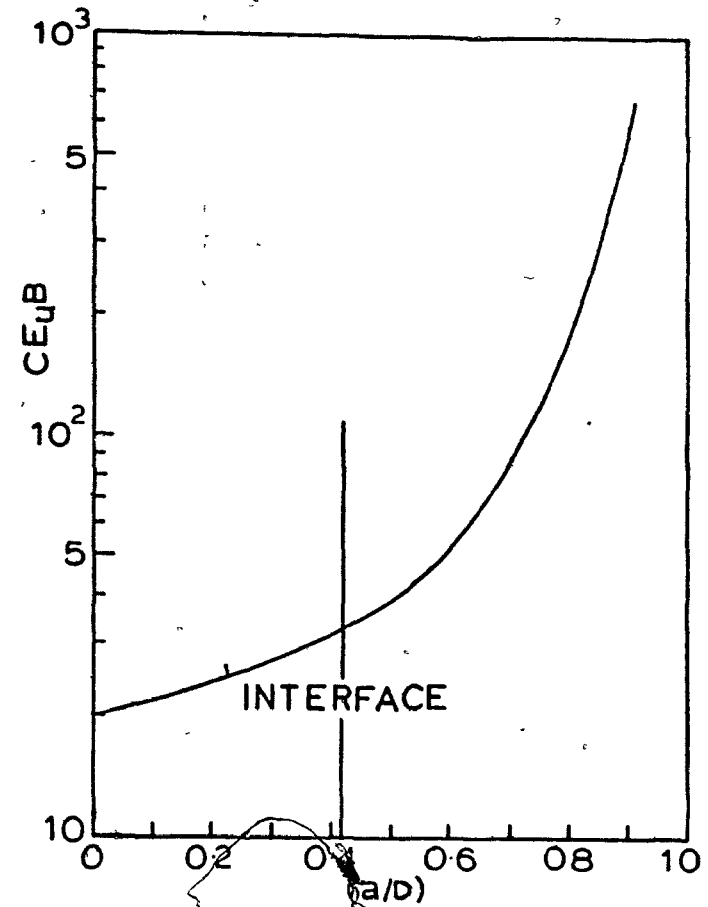


Figure A.5.10.2 CE_uB versus (a/D) Curve for Two Layer Test Pieces in the L Direction with $f = 0.58$. (Crack Propagation from Unreinforced to SF RTP Layer)

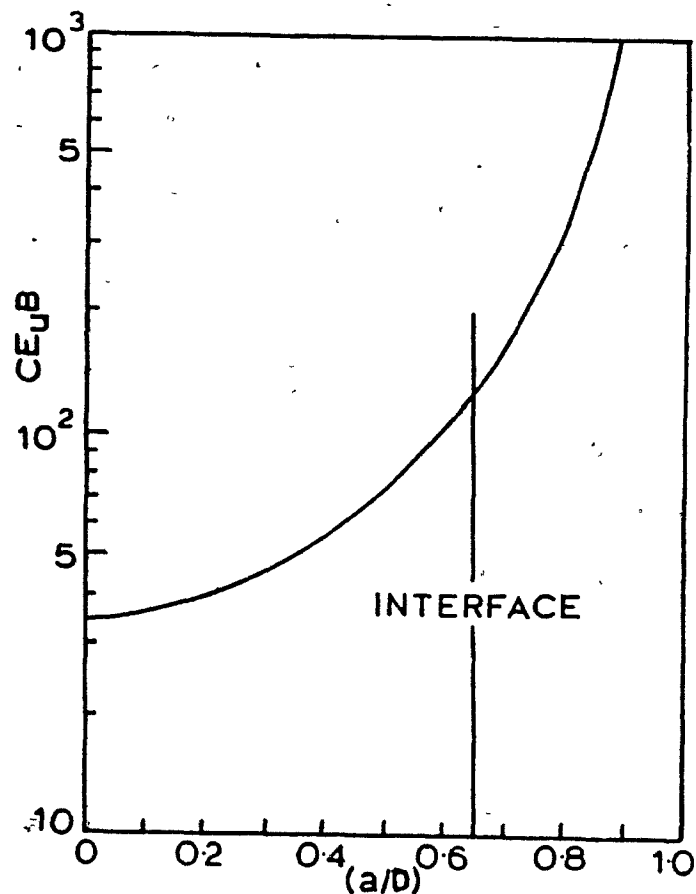


Figure A.5.10.3 CE_{uB} versus (a/D) Curve for Two Layer Test Pieces in the T Direction with $f = 0.35$. (Crack Propagation from Unreinforced to SF RTP Layer)

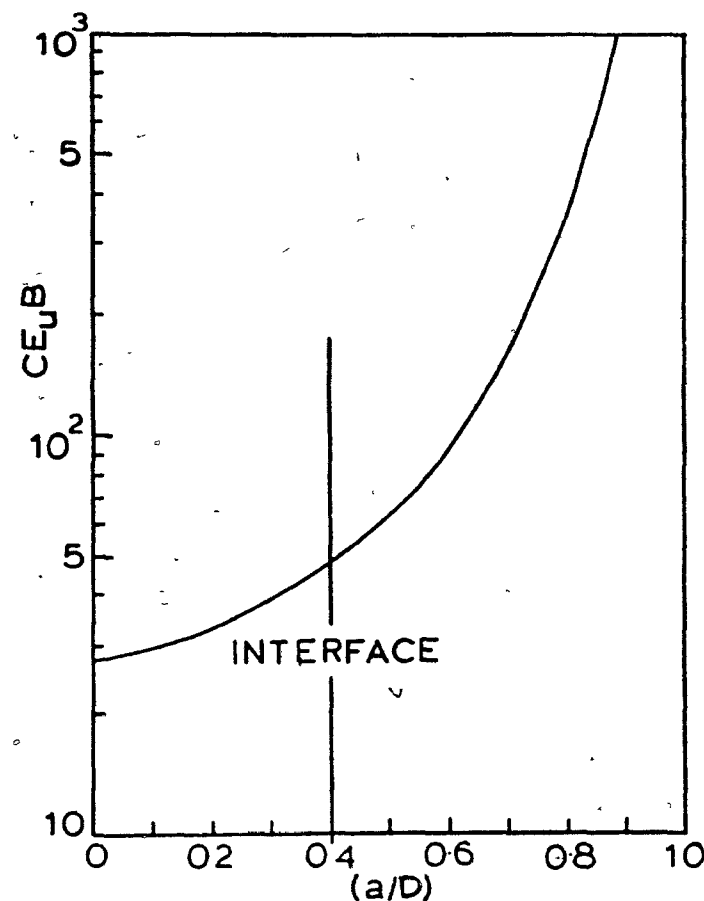


Figure A.5.10.4 CE_{uB} versus (a/D) Curve for Two Layer Test Pieces in the T Direction with $f = 0.60$. (Crack Propagation from Unreinforced to SF RTP Layer)

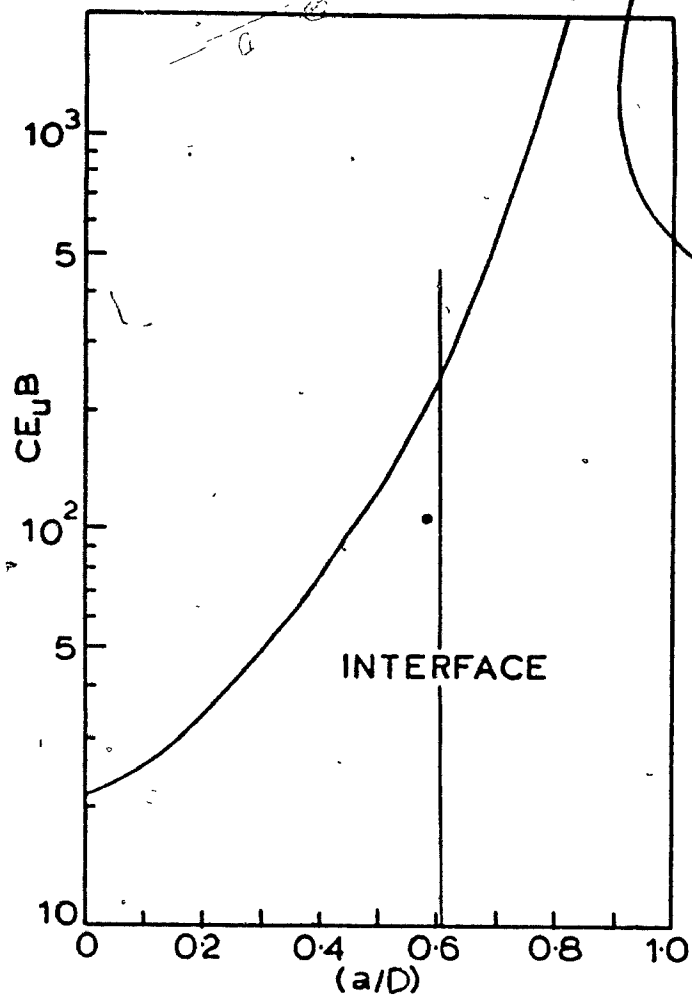


Figure A.5.10.5 CE_uB versus (a/D) Curve for Two Layer Test Pieces in the L Direction with $f=0.61$. (Crack Propagation from SFRTF to Unreinforced Layer)

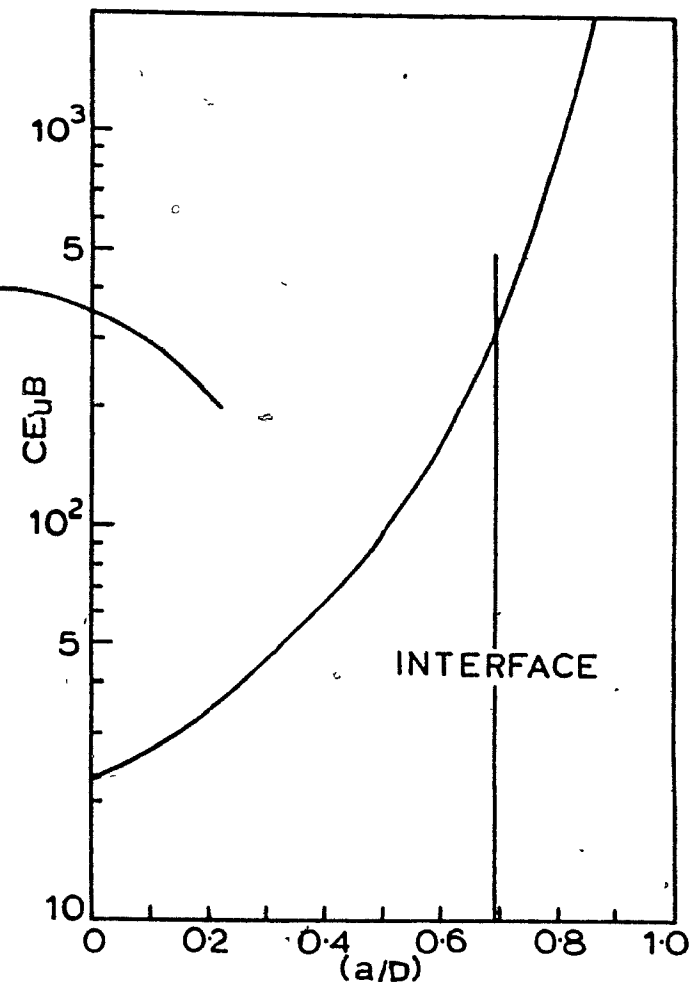


Figure A.5.10.6 CE_uB versus (a/D) Curve for Two Layer Test Pieces in the T Direction with $f=0.69$. (Crack Propagation from SFRTF to Unreinforced Layer)

Appendix 5.11

R - Curve Data for Crack Extension from Unreinforced to SF RTP Layer

(a) Test Pieces in the L Direction

Fractional Thickness of SF RTP Layer = 0.23

Test Piece No. 1

$B_c = 7.31 \text{ mm}$ $D = 11.20 \text{ mm}$
 $(a/D)_0 = 0.346$ $C_{0EUB} = 31.4$

Load Kg	CE_{UB}	$(a/D)_{eff}$	K_R MPa/m
10.0	31.4	0.346	0.98
12.0	31.8	0.348	1.19
14.0	32.23	0.354	1.41
16.0	33.43	0.37	1.68
16.8	34.2	0.38	1.81
16.0	35.9	0.40	1.82
14.0	41.1	0.445	1.81
12.0	48.1	0.494	1.80
10.0	57.92	0.545	1.77
8.0	73.85	0.603	1.75
6.0	100.4	0.663	1.69
4.2	172.23	0.765	2.04
4.2	269.15	0.83	3.33
4.0	351.8	0.86	4.26
3.6	462.6	0.882	4.96
3.0	677.6	0.90	5.32
2.4	995.34	0.904	4.52

Test Piece No. 2

$B_c = 6.58 \text{ mm}$ $D = 11.24 \text{ mm}$
 $(a/D)_0 = 0.54$ $C_{0EB} = 57.24$

Load Kg	CE_{UB}	$(a/D)_{eff}$	K_R MPa/m
6.0	57.94	0.545	1.45
7.0	61.63	0.56	1.56
7.14	65.74	0.575	1.63
7.0	71.2	0.593	1.73
6.0	91.45	0.646	1.81
5.0	117.28	0.695	2.08
4.0	165.97	0.76	2.34
3.8	194.0	0.785	3.19
3.8	260.0	0.825	4.33
3.6	364.75	0.862	4.8
3.4	431.2	0.876	5.3
3.0	568.2	0.893	5.0

Test Piece No. 3

B = 6.36 mm

D = 11.19 mm

$(a/D)_0 = 0.395$ $C_{0EB} = 35.4$

Load Kg	CE_{UB}	$(a/D)_{eff}$	K_R MPa $\sqrt{m}$
10.0	36.0	0.40	1.31
12.0	37.08	0.41	1.62
13.0	38.12	0.42	1.80
14.0	39.3	0.43	2.0
14.5	40.45	0.442	2.14
14.0	42.04	0.453	2.13
12.0	49.05	0.5	2.11
10.0	59.27	0.55	2.08
8.0	74.33	0.6	2.0
6.0	100.8	0.663	1.94
4.0	156.76	0.748	2.01
3.2	252.21	0.82	2.68
3.2	355.87	0.86	3.92
3.0	445.0	0.88	4.64
2.4	726.5	0.904	5.2

(b) Test Pieces in the L Direction

Fractional Thickness of SF RTP Layer = 0.58

Test Piece No. 1

B = 6.46 mm D = 10.68 mm
(a/D)₀ = 0.2 C₀E_uB = 19.95

Load Kg	C _{E_u} B	(a/D) _{eff}	K _R MPa√m
15.0	21.8	0.26	1.44
20.0	23.66	0.3	2.12
22.5	24.87	0.33	2.58
24.8	25.82	0.35	2.94
23.0	27.46	0.385	3.05
21.0	30.17	0.434	3.2
20.0	31.99	0.455	3.24
19.5	33.76	0.476	3.36
19.75	38.23	0.52	3.91
19.9	42.18	0.553	4.41
19.0	54.89	0.625	5.52
17.5	66.77	0.665	6.04
15.0	86.4	0.71	6.45
10.0	150.78	0.79	7.0
5.0	391.26	0.879	8.06

Test Piece No. 2

B = 6.6 mm D = 10.67 mm
(a/D)₀ = 0.223 C₀E_uB = 22.99

Fractional Thickness of SF RTP = 0.61

Load Kg	C _{E_u} B	(a/D) _{eff}	K _R MPa√m
15.0	25.05	0.275	1.46
20.0	27.19	0.325	2.22
24.5	29.39	0.375	3.1
23.0	31.31	0.41	3.21
22.0	32.83	0.433	3.28
20.6	35.77	0.47	3.42
20.5	37.18	0.485	3.56
21.5	41.89	0.53	4.32
21.75	46.62	0.568	4.99
20.0	58.68	0.63	5.82
17.5	74.98	0.682	6.42
15.0	93.36	0.72	6.67
10.0	161.66	0.795	7.13
5.0	403.1	0.88	8.01

(c) Test Pieces in the T Direction

Fractional Thickness of SF RTP Layer = 0.35

Test Piece No. 1

B = 5.88 mm D = 10.66 mm
(a/D)₀ = 0.36 C₀E_UB = 44.0

Load Kg	CE _U B	(a/D) eff	K _R MPa√m
8.0	44.89	0.37	1.13
9.0	46.03	0.376	1.29
10.0	47.29	0.385	1.46
11.0	48.65	0.4	1.68
11.04	49.44	0.404	1.76
10.0	56.54	0.45	1.76
8.0	71.34	0.525	1.78
6.0	98.08	0.6	1.74
5.0	124.45	0.647	1.76
4.6	160.4	0.7	2.07
4.5	221.16	0.75	2.67
4.0	316.92	0.797	3.25
3.0	542.27	0.845	3.67

Test Piece No. 2

B = 6.22 mm D = 10.85 mm
(a/D)₀ = 0.36 C₀E_UB = 50.69

Load Kg.	CE _U B	(a/D) eff	K _R MPa√m
8.0	51.43	0.368	1.03
9.0	51.87	0.37	1.17
10.0	52.62	0.387	1.3
11.0	53.23	0.384	1.48
12.0	54.56	0.39	1.64
13.0	56.6	0.406	1.86
12.0	61.49	0.444	1.91
11.0	67.25	0.476	1.93
10.0	74.18	0.509	1.94
9.0	82.64	0.542	1.95
8.0	93.46	0.573	1.94
7.0	108.23	0.608	1.93
6.0	133.85	0.653	1.99
5.4	191.21	0.72	2.49
5.0	255.57	0.765	3.0
4.4	340.77	0.803	3.45
4.0	412.92	0.82	3.6
3.4	548.28	0.845	3.83

Test Piece No. 3

$B = 6.19 \text{ mm}$ $D = 10.5 \text{ mm}$
 $(a/D)_0 = 0.383$ $C_{0EuB} = 0.35$

Load Kg	CE_{uB}	$(a/D)_{eff}$	K_{R-} MPa/m
8.0	53.7	0.4	1.19
10.0	55.77	0.415	1.55
11.0	57.15	0.422	1.73
11.72	58.69	0.43	1.89
11.0	62.71	0.455	1.91
10.0	69.14	0.494	1.95
9.0	76.95	0.527	1.96
8.0	86.72	0.555	1.91
7.0	99.67	0.588	1.89
6.0	117.26	0.628	1.89
5.0	164.76	0.693	2.11

(d) Test Pieces in the T Direction

Fractional Thickness of SFRTF Layer = 0.6

Test Piece No. 1

B = 6.67 mm D = 10.23 mm
(a/D)₀ = 0.18 C₀E_uB = 33.02

Load Kg	C _{E_u} B	(a/D) _{eff}	K _R MPa/m
12.0	35	0.22	1.1
14.0	36.28	0.245	1.37
16.0	37.75	0.285	1.65
16.74	38.69	0.285	1.81
16.0	40.76	0.31	1.85
14.0	46.73	0.375	1.92
12.0	54.69	0.407	1.8
12.0	60.61	0.437	1.92
12.4	70.79	0.525	2.65
12.0	84.3	0.572	3.02
10.0	120.8	0.655	3.5
8.0	170.34	0.717	3.79
6.0	264.73	0.774	3.99
5.0	345.69	0.8	4.0
4.0	475.48	0.823	3.85
3.0	706.42	0.851	3.75

Test Piece No. 2

B = 6.96 mm D = 10.14 mm
(a/D)₀ = 0.16 C₀E_uB = 28.55

Load Kg	CE _u B	(a/D) _{eff}	K _R MPa√m
12.0	30.62	0.205	1.0
14.0	31.93	0.234	1.26
16.0	33.34	0.255	1.52
18.0	34.67	0.275	1.80
20.0	36.52	0.302	2.15
20.4	37.32	0.315	2.26
20.0	38.07	0.324	2.27
18.0	42.32	0.37	2.31
16.0	47.64	0.413	2.31
13.82	57.01	0.475	2.31
13.82	63.42	0.51	2.67
13.8	69.6	0.535	2.89
13.0	83.76	0.582	3.21
12.0	98.3	0.618	3.4
10.0	131.91	0.678	3.69
8.0	183.15	0.73	3.85
6.0	276.66	0.78	3.94
4.0	497.43	0.828	3.91

Test Piece No. 3

B = 6.45 mm D = 10.26 mm
(a/D)₀ = 0.16 C₀E_uB = 30.35

Load Kg	CE _u B	(a/D) _{eff}	K _R MPa√m
15.0	31.99	0.2	1.31
17.5	33.28	0.225	1.63
20.0	35.27	0.26	2.04
21.2	36.37	0.274	2.24
20.0	38.55	0.303	2.28
17.5	44.25	0.37	2.38
15.0	51.68	0.43	2.4
14.0	61.52	0.49	2.68
12.5	87.6	0.59	3.37
10.0	123.05	0.66	3.6
7.5	186.49	0.73	3.83
5.0	335.5	0.8	4.02
4.0	461.4	0.822	3.83

Appendix 5.12

R Curve Data for Crack Extension from SF RTP to Unreinforced Layer

(a) Test Pieces in the L Direction

Fractional Thickness of SF RTP Layer = 0.61

Test Piece No. 1

B = 5.96 mm D = 10.43 mm
(a/D)₀ = 0.297 C₀E_uB = 32.02

Load Kg	CE _u B -	(a/D) _{eff} -	K _R MPa/√m
8.0	33.68	0.3	0.95
10.0	34.91	0.308	1.22
12.0	37.32	0.319	1.5
14.0	41.34	0.332	1.81
15.0	44.9	0.347	2.02
16.0	53.37	0.378	2.34
16.05	56.5	0.39	2.43
16.0	59.29	0.395	2.45
15.0	74.63	0.434	2.57
14.0	87.28	0.463	2.61
12.0	116.85	0.51	2.59
10.0	156.54	0.55	2.46
8.0	213.46	0.592	2.30
6.0	289.04	0.63	2.0
4.0	438.79	0.696	1.8

Test Piece No. 2

B = 6.07 mm D = 10.44 mm
(a/D)₀ = 0.3 C₀E_uB = 32.35

Load Kg	CE _u B -	(a/D) _{eff} -	K _R MPa/√m
8.0	33.56	0.304	0.95
10.0	34.96	0.31	1.2
12.0	36.86	0.318	1.47
14.0	39.87	0.33	1.77
16.0	47.08	0.357	2.17
17.0	55.45	0.385	2.49
16.6	70.28	0.427	2.73
16.0	78.95	0.446	2.78
15.0	92.2	0.47	2.8
14.0	105.69	0.49	2.78
12.0	136.34	0.53	2.71
10.0	176.55	0.568	2.58
8.0	223.73	0.6	2.32
6.0	301.0	0.637	2.02

Test Piece No. 3

B = 6.76 mm D = 10.44 mm
 $(a/D)_0 = 0.29$ $C_0 E_u B = 30.13$

Load Kg	$CE_u B$	$(a/D)_{eff}$	K_R MPa $\sqrt{m}$
10.0	31.86	0.3	1.05
12.0	33.18	0.304	1.27
14.0	~~35.21~~	0.31	1.51
16.0	38.2	0.325	1.79
18.0	43.77	0.347	2.14
19.0	49.96	0.368	2.38
19.12	55.72	0.388	2.53
19.0	60.72	0.4	2.6
18.0	72.7	0.433	2.7
17.0	84.45	0.455	2.72
16.0	95.78	0.48	2.76
14.0	121.46	0.514	2.69
12.0	152.82	0.545	2.56
11.0	172.78	0.562	2.49
10.0	192.86	0.58	2.41
8.0	243.76	0.61	2.17
6.0	327.89	0.648	1.9
4.0	499.9	0.69	1.54

b) Test Pieces in the T Direction.

Fractional Thickness of SERTP Layer = 0.69

Test Piece No. 1

$B = 7.26 \text{ mm}$ $D = 10.44 \text{ mm}$
 $(a/D)_0 = 0.294$ $C_0 E_U B = 37.27$

Load Kg	$CE_U B$	$(a/D)_{eff}$	K_R MPa $\sqrt{m}$
12.0	40.26	0.31	1.2
14.0	42.43	0.323	1.45
16.0	46.23	0.347	1.77
18.0	54.83	0.388	2.22
18.2	59.75	0.407	2.36
18.0	64.59	0.422	2.44
16.0	85.1	0.482	2.59
14.0	106.0	0.525	2.6
12.0	133.87	0.57	2.6
10.0	175.21	0.615	2.57
8.0	237.5	0.66	2.49
6.0	324.76	0.7	2.26
4.0	501.0	0.747	1.95

Test Piece No. 2

$B = 5.9 \text{ mm}$ $D = 10.42 \text{ mm}$
 $(a/D)_0 = 0.302$ $C_0 E_U B = 35.9$

Load Kg	$CE_U B$	$(a/D)_{eff}$	K_R MPa $\sqrt{m}$
10.0	41.3	0.333	1.32
12.0	45.84	0.355	1.67
13.0	49.82	0.374	1.9
14.0	57.39	0.407	2.25
14.0	70.8	0.45	2.54
12.0	97.93	0.515	2.66
10.0	130.66	0.57	2.68
8.0	182.33	0.621	2.6
6.0	287.84	0.684	2.57
4.0	456.17	0.74	2.31
3.0	630.76	0.77	1.2

Test Piece No. 3

B = 6.00 mm D = 10.43 mm
 $(a/D)_0 = 0.37$ $C_0 E_u B = 42.5$

Load Kg	$CE_u B$	$(a/D)_{eff}$	$K_R \sqrt{m}$ MPa
10.0	46.79	0.39	1.5
12.0	52.84	0.412	1.92
13.36	66.48	0.456	2.42
12.0	92.48	0.518	2.64
10.0	128.54	0.575	2.68
8.0	184.79	0.63	2.65
6.0	291.6	0.69	2.6
4.0	462.2	0.74	2.27
3.0	630.3	0.772	2.07
2.0	987.5	0.81	1.82

Appendix A.5.13

Crystallinity of SF RTP and the High Density Unreinforced Resins

In order to assess the crystallinity in SF RTP and the high density, unreinforced resins, differential scanning calorimetric (DSC) experiments were carried out using the slowly cooled samples. The test records in terms of the rate of heat input vs. the temperature are shown in Figures A.5.13.1 and A.5.13.2. The area under the peak in these plots represents the heat of fusion per unit sample weight (cal/gm). The heat of fusion is expected to be directly proportional to the degree of crystallinity for a partially crystalline polymer such as PE. As can be seen; the heat of fusion of the unreinforced sample is 51.68 cal/gm, while that of SF RTP sample is 35.45 cal/gm. Thus the heat of fusion of SF RTP sample and, hence, its degree of crystallinity is about 70% of that of the unreinforced sample. This appears to be mainly due to the fact that SF RTP resin contains 30% (by weight) inert material in the form of fibers that does not undergo a phase transition. Thus, in a multi-layer plaque, there is less contraction of SF RTP layer upon solidification as compared to the unreinforced layer. This differential contraction of the two layers can give rise to residual stresses in the plaque.

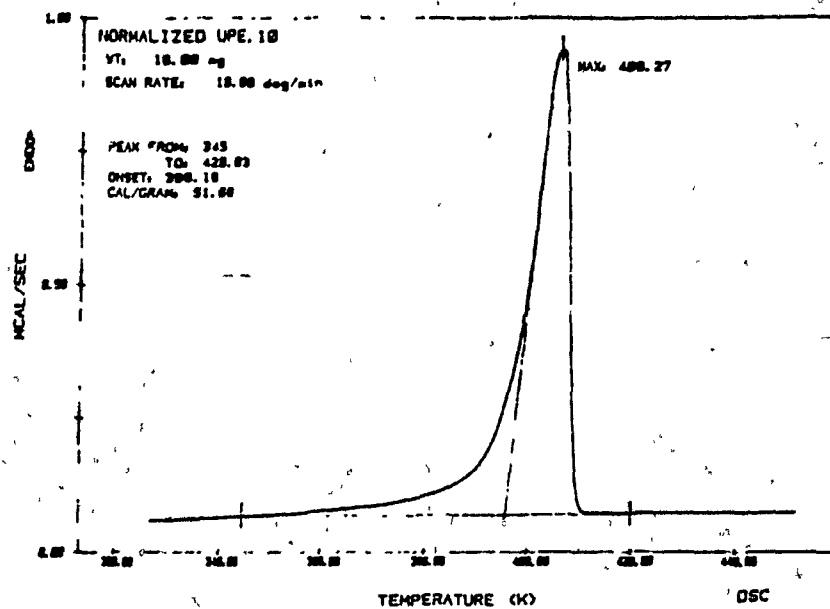


Figure A.5.13.1 DSC Test Record for High Density Unreinforced PE (PE 2906 M6) Sample.

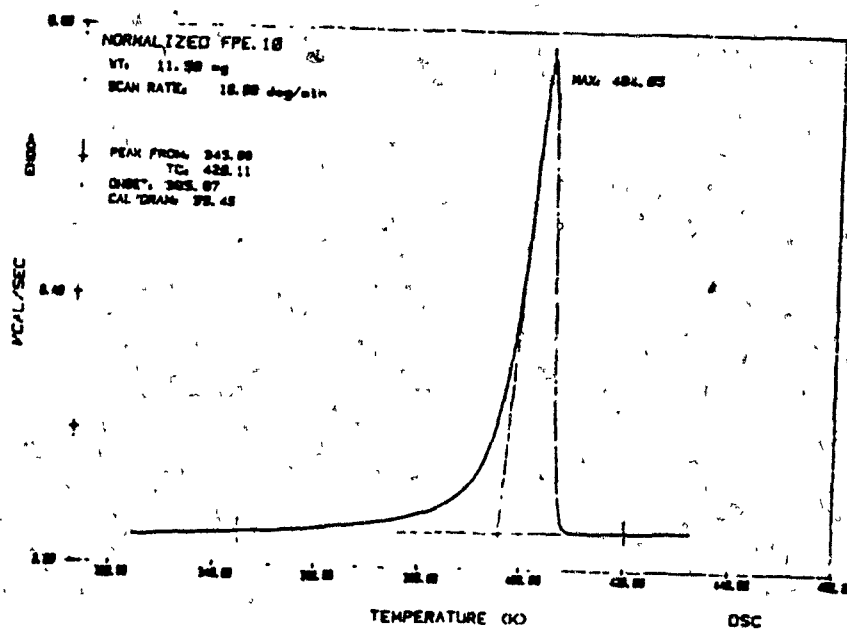


Figure A.5.13.2 DSC Test Record for SFRTTP Sample.

Appendix 6.1

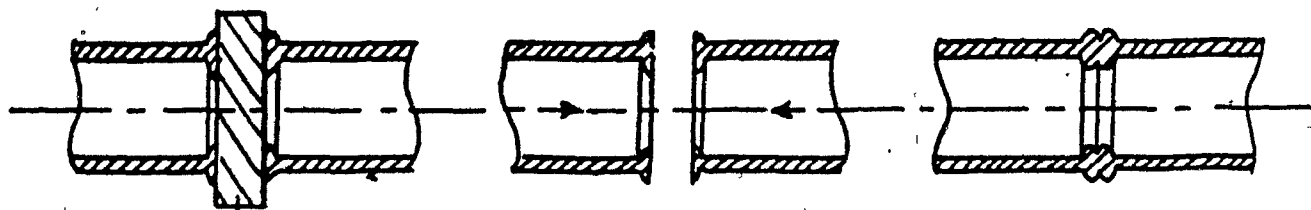
- Methods of Joining Plastic Pipe

There are two basic methods of joining plastic pipe, viz., fusion and mechanical methods. Fusion methods are useful for joining ends of plastic pipe of the same or similar materials, while mechanical methods are necessary for joining components of dissimilar plastic materials or plastic and metal (152-154).

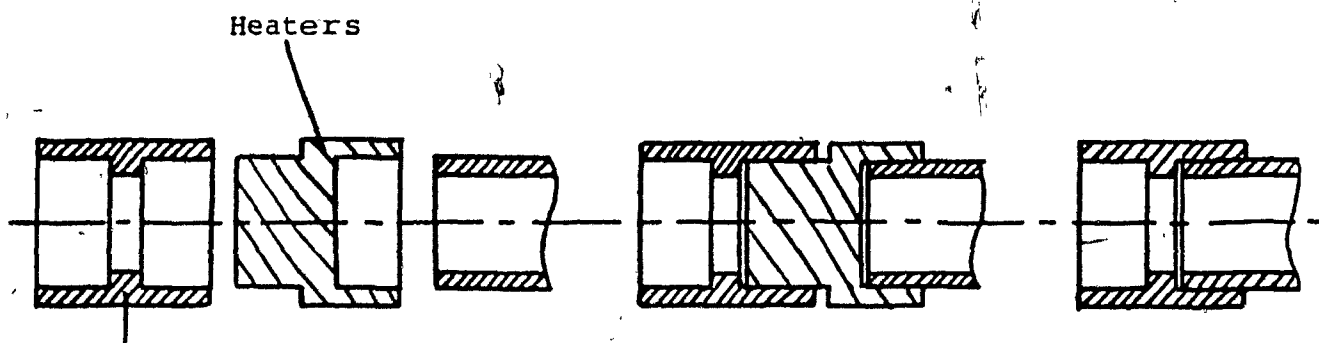
A.6.1 Fusion Methods

Most of the fusion methods described below are developed for polyethylene pressure pipe, although some may also be used for polyvinyl chloride pipe. There are four common fusion techniques, viz., butt fusion, socket fusion, electrofusion and hot gas welding.

- (a) **Butt Fusion :-** Butt fusion is used for pipe in the diameter range of 50 mm to 1200 mm, and requires a minimum wall thickness of about 5 mm to form a satisfactory joint. The method is illustrated in Figure A.6.1.1a. The two pipe ends to be joined are heated using a specially designed plate heater and melted. The heater is then removed, and the ends are brought together, and fused under appropriate pressure.
- (b) **Socket Fusion :-** Socket fusion is used for small diameter pipes (20 mm to 100 mm). It involves joining

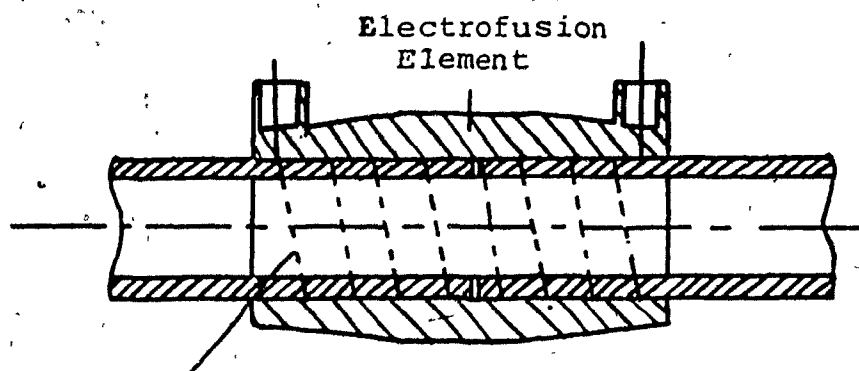


(a) Butt Fusion



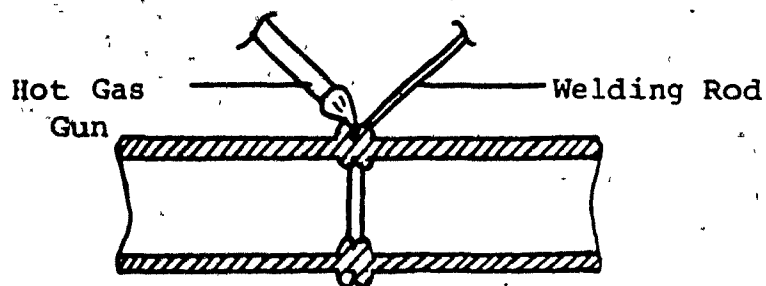
Socket Fitting

(b) Socket Fusion



Heating Wire Coil

(c) Electrofusion



(d) Hot Gas Welding

Figure A.6.1.1 Fusion Methods of Joining Plastic Pipe.

of two pipe ends using a socket fitting made of the same resin as the pipe, as shown schematically in Figure A.6.1.1b. The outside surface of the pipe end and the inside surface of the socket fitting are heated and melted using a specially designed heater. The heater is subsequently withdrawn, and the socket is fused over the pipe end under moderate pressure.

(c) Electrofusion :- Electrofusion is used in the pipe diameter range of 20 mm to 200 mm. As shown schematically in Figure A.6.1.1c, it involves fusing a sleeve type electrofusion element over the outer surface of the pipe end. The element is made of the same resin as the pipe, and incorporates a resistance wire heating coil. Heating and melting is thus carried out electrically, and fusion is obtained under slight pressure.

(d) Hot Gas Welding :- Hot gas welding technique is used for shop fabricated polyethylene pipe joints. The technique involves the use of a hot gas gun for heating and melting and a welding rod of the same polyethylene resin as the pipe. The pipe ends to be joined are chamfered and aligned. The torch is played simultaneously over the area to be fused and the end of the welding rod. Pressure is applied to the rod to force the melted material into the joint being made, as shown schematically in Figure A.6.1.1d.

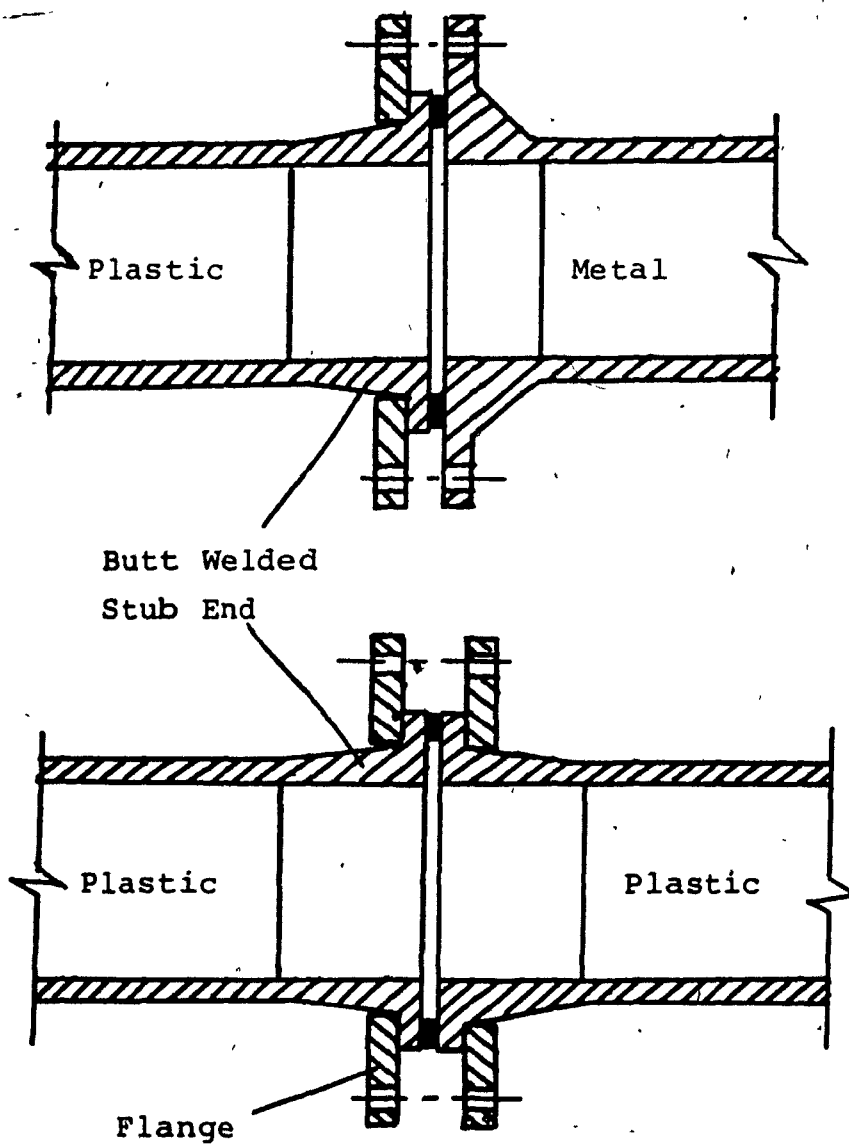


Figure A.6.1.2 Mechanical Joining of Plastic Pipe.

A.6.2 Mechanical Joining Methods

Mechanical joining methods for large diameter pipe usually involve a flanged connection. Flanges of aluminum or cast iron are slipped onto the pipe, and stub ends made of the same resin as the pipe are butt fused to the ends. The two ends can then be joined by bolts, as shown schematically in Figure A.6.1.2. A gasket between the ends is usually necessary to ensure a leak proof joint.