

# A Mandrel-Rotating Die to Produce High-Hoop-Strength HDPE Pipe by Self-Reinforcement

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**ABSTRACT:** This article provides a method to obtain high-hoop-strength high-density polyethylene (HDPE) pipe by mandrel-rotating extrusion. With properly selected processing temperature, pressure, and mandrel-rotating speed, the hoop strength of 90 Mpa has been got. Differential scanning calorimetry and X-ray scattering found the great strength enhancement was owing to high degree of macromolecular orientation in circumferential direction and the shish-kebab structure. © 1998 John Wiley & Sons, Inc. *J Appl Polym Sci* 69: 323–328, 1998

**Key words:** high-density polyethylene; pipe; self-reinforcement; hoop strength; shish kebab

## INTRODUCTION

The axial stress in pipe wall is half of the hoop stress when an internal hydraulic pressure is applied to thin-walled pipe.<sup>1</sup> Normally, the axial strength is higher than hoop strength due to the macromolecular orientation during extrusion. So in order to improve the pressure endurance of the plastic pipe, we must increase the hoop strength at first. With the development of self-reinforcement technology, people can extrude high-density polyethylene (HDPE) rods whose axial strength is 6 times that of normal products because of the occurrence of a shish-kebab structure.<sup>2</sup> The self-reinforcement of polymer materials has been studied by researchers.<sup>3–8</sup> How to improve the hoop strength of plastic tubes is our keen interest.

This article will provide a method to improve the hoop strength and modulus greatly by self-reinforcement. In order to achieve good self-reinforcement result, it is the prerequisite to orient

the macromolecular chains along the hoop direction during extrusion. Then by imposing a properly controlled pressure and temperature field, the microfibril crystalline or shish-kebab structure, which is the crucial factor of strength enhancement, will be induced and locked in the products before they are expelled from the die. To satisfy above requirements, the extrusion die must have an elaborate temperature control system to provide satisfactory temperature gradient so as to accelerate crystallization, and a pressure transducer to indicate and regulate melt pressure, which can exert an even and appropriate shearing force in circumferential direction to make macromolecular chains orient along hoop direction.

## EXPERIMENTAL

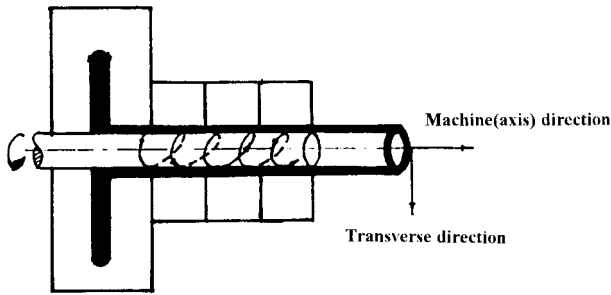
The extrusion system consisted of an extruder ( $D_{\text{screw}} = 45$  mm, produced by Shanghai Extruder Factory), die, temperature control unit, and mandrel activating unit. The die was divided into 3 sections, and the temperature of each section could be controlled separately by a mold-temper-

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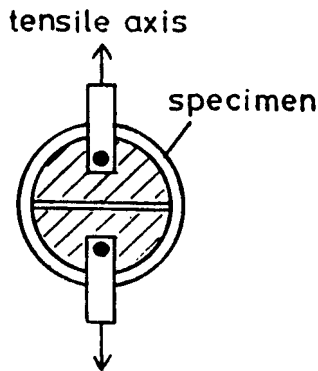
**Figure 1** Schematic diagram of molecular orientation in the die.

ature regulator. A temperature gradient of  $\sim 60^{\circ}\text{C}$  was applied between the die inlet and outlet. A motor and gear reduction system was used to turn the mandrel, with the rotating speed ranging from 0 to 40 rpm. The outer cylinder had a inside diameter of 63 mm, and the mandrel had a outside diameter of 55 mm, giving a gap of 4 mm. Melt from the extruder entered an adapter block, in which the melt stream was split into 2 equal channels, which led the melt to die orifice from the top and bottom of the block separately. The temperature of the adapter block was  $180^{\circ}\text{C}$ . The schematic diagram of the die is shown in Figure 1.

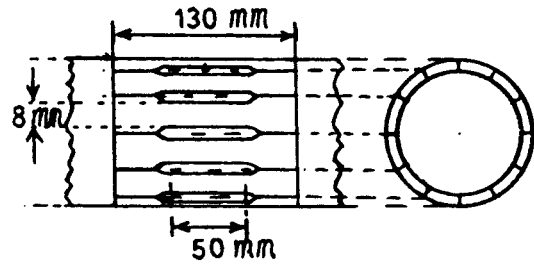
**Mechanical Testing**

**Hoop Strength and Modulus**

Five rings were cut from the pipe for each processing condition. The width of each ring was 20 mm. The testing was performed on a tensile machine (Shimadzu, AG-10TA) using a crosshead speed of 50 mm/min as shown in Figure 2.<sup>9</sup>



**Figure 2** Diagram of the method of testing the hoop strength of the tubes.



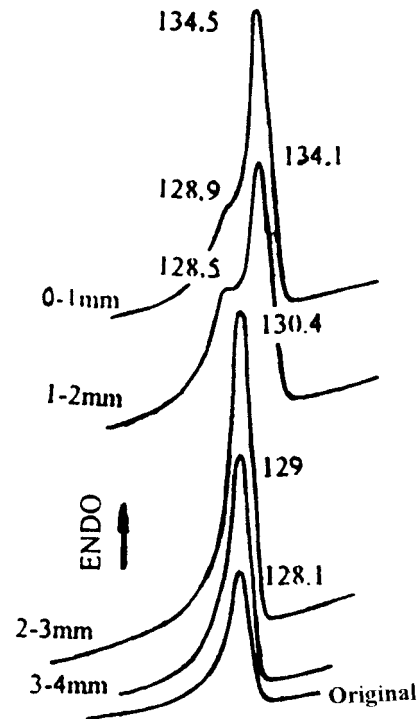
**Figure 3** Diagram showing the method of sectioning the tubes for tensile test samples in the axial direction.

**Axial Strength and Modulus**

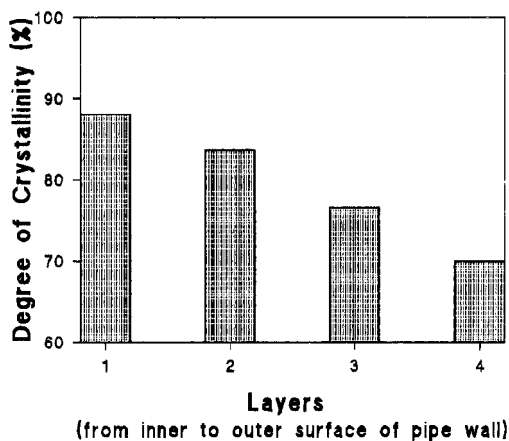
For each processing condition, 5 sections of the pipe were cut in the axial direction around the hoop and finished into samples, as shown in Figure 3.<sup>10</sup> Axial strength and modulus were tested on a tensile machine (Shimadzu, AG-10TA) with the crosshead speed of 50 mm/min.

**Short-Time Hydraulic Failure Pressure**

Short-time hydraulic failure pressure test was conducted according to ASTM D 1599-82.



**Figure 4** Melting endotherms of HDPE tube produced under the conditions: melt pressure is 10 Mpa and mandrel-rotating speed is 24 rpm. The distance of slices from the inner surface of tube is indicated above the curves.



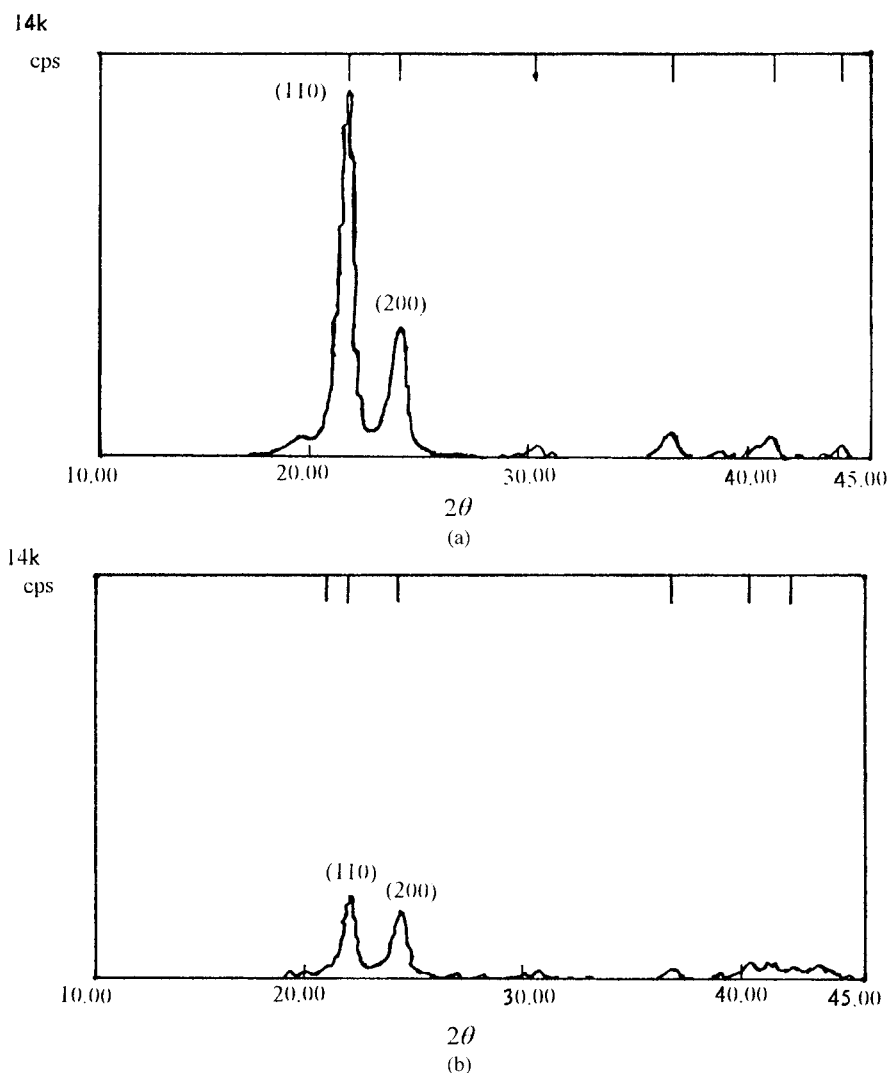
**Figure 5** The crystallinity of each layer in the pipe wall.

### Differential Scanning Calorimetry

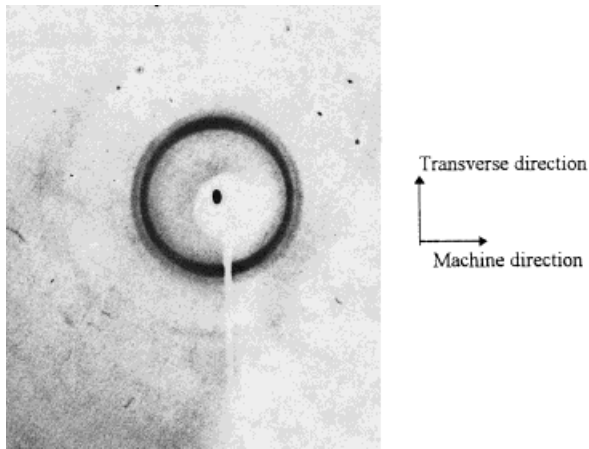
The pipe wall was divided into 4 layers. The structure of each layer was investigated by DSC using a Perkin-Elmer DSC-2. The heating rate was 5°C/min.

### X-ray Measurement

The orientations of crystal planes of samples were investigated by wide-angle X-ray scattering (WAXS) using a D/MAX-III A X-ray diffractometer. Wide-angle diffraction patterns of X-ray scattering were obtained with a VEM X-ray diffraction camera.



**Figure 6** (a) X-ray scattering curves for HDPE tubes prepared by mandrel-rotating extrusion. (b) X-ray scattering curves for HDPE tubes prepared by normal extrusion.

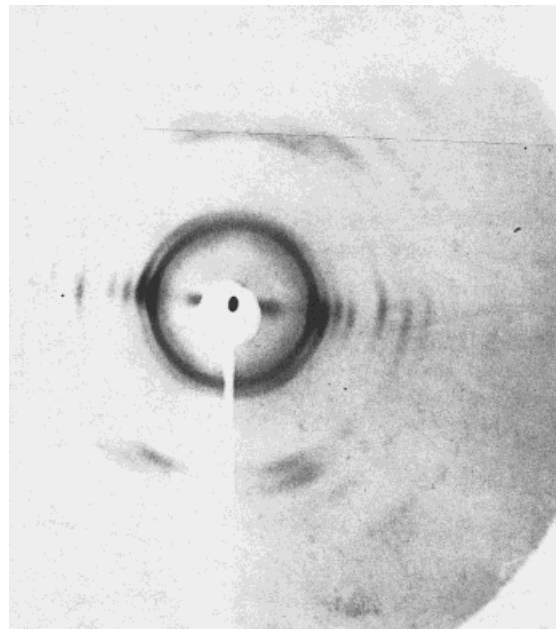


**Figure 7** XRD patterns of HDPE tube prepared by normal extrusion.

## RESULTS AND DISCUSSION

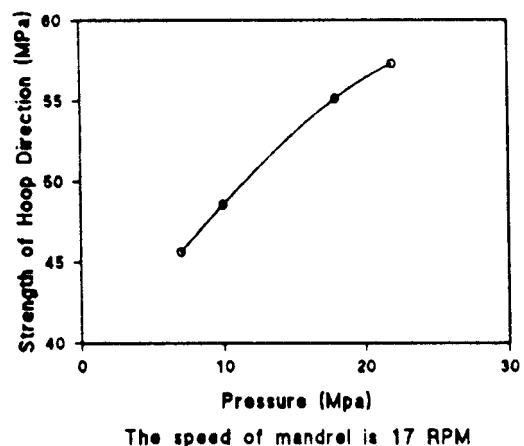
Differential scanning calorimetry (DSC) thermographs of 4 layers are shown in Figure 4. From the graphs, we can distinguish 2 melting peaks clearly: one at 128.9°C, and the other at 134.5°C, which represent 2 kinds of crystalline structures, respectively. The higher-temperature melting peak indicates a more thermostatically stable structure has been formed. The absence of the higher-temperature crystalline in original endotherm indicates that the higher-temperature crystalline is induced by the shear force exerted by mandrel rotation. With the increasing distance from inside surface of the pipe wall, the proportion of this kind of structure decreases because the shear force acted on the melt declines. Here, the peak at about 128°C represents the spherulites and lamellae in a shish-kebab structure, while the higher-temperature crystalline melting at about 134°C is stretched chain crystalline in the shish-kebab structure, which is induced by the shear forces and has a higher melting point.<sup>11</sup> Assuming the heat of fusion of perfect PE crystal is 69 cal/g,<sup>12</sup> we can calculate the crystallinity of each layer. The results indicates that the crystallinity decreases with the increasing distance from inside surface because the promotion of shear-induced crystallization decreases from inside to outside surface of the pipe wall (Fig. 5).

WAXS film patterns are a key qualitative indicator of molecular orientation. The two strongest reflection of HDPE are (110) and (200) planes.

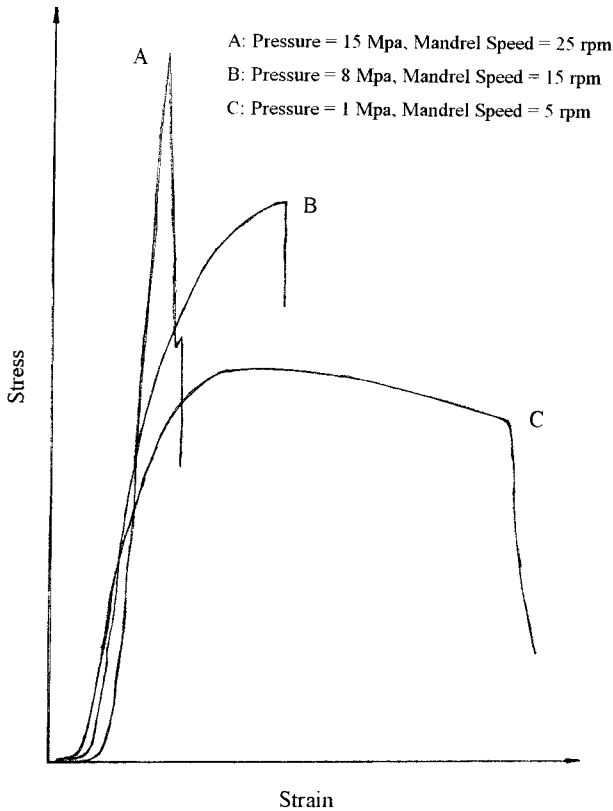


**Figure 8** XRD patterns of HDPE tube prepared by mandrel-rotating extrusion.

Figure 6(b) shows the orientation degree of (110) and (200) planes was increased considerably in reinforced extrudates. From the film pattern shown in Figures 7 and 8, the c-axis has been rotated to predominantly the transverse direction by mandrel rotation;<sup>13-16</sup> that is, the macromolecule chains tend to orient along transverse direction in mandrel-rotating extrudates, which is the prerequisite to increase the hoop strength and modulus of HDPE pipe.



**Figure 9** The hoop strength as a function of melt pressure.



**Figure 10** The strain–stress curves of specimens produced in different conditions.

The hoop strength of normal extruded pipe is about 26 Ma. By the mandrel-rotating extrusion, the hoop strength varied notably with different extrusion pressure, rotating speed of mandrel and melt temperature, but all the values are much higher than those of normal extrudates. The highest strength we obtained was 90 Mpa, almost 4 times of normal strength.

The dependence of the hoop strength on the extrusion pressure is shown in Figure 9. With increasing pressure, the hoop strength increases. According to Van Krevelen,<sup>17</sup>  $V_k \propto \exp[-1/(T_m - T_x)]$ , where  $T_m$  is the melting point and  $T_x$  is the crystallization temperature. Since  $T_m$  is raised, and  $T_x$  is almost kept constant when increasing extrusion pressure, so  $V_k$  is increased. Therefore, the growth of crystalline or shish-kebab structure is accelerated and results in higher strength and modulus.

The increase of mandrel rotating speed and extrusion pressure also results in the enhancement of hoop modulus. Our measure

equipment shown in Figure 2 cannot determine the accurate value of the hoop modulus, but the strain–stress curves of the specimens clearly proved this enhancement (Fig. 10). The greater the slope of the curve, the higher the hoop modulus.

In the short-time hydraulic failure pressure testing, the normal extrudates were broken along axial direction at the pressure of 3 Mpa, while the reinforced products tended to crack along hoop direction when the pressure was above 5 Mpa. This result indicates that the axial strength is higher than hoop strength in the normal extrudates, while in the reinforced products, the hoop strength was greatly enhanced and exceeded the axial strength.

## CONCLUSION

The rotating annular die has been successfully used to control the macromolecular orientation in HDPE pipe extrusion. By properly selecting the processing parameters, the hoop strength and modulus of HDPE pipe could be greatly enhanced. The DSC test found new high-temperature crystalline: shish-kebab structure, whose quantity varied across the cross section of the pipe wall. WAXS patterns verified that the macromolecular chains oriented along transverse direction.

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