DESIGN, CONSTRUCTION, AND TESTING OF A RAM EXTRUDER FOR HIGH VISCOSITY POLYMERS

BY

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ABSTRACT

DESIGN, CONSTRUCTION, AND TESTING OF A RAM EXTRUDER FOR HIGH VISCOSITY POLYMERS

by

Joseph S. Lubischer

Submitted to the Department of Mechanical Engineering on October 6, 1972 in partial fulfillment for the requirements for the Degree of Bachelor of Science in Mechanical Engineering and the Degree of Master of Science in Mechanical Engineering.

A ram extruder for the direct extrusion of high viscosity polymers from powder has been successfully designed and tested. The extrusions, in terms of density, surface finish, and dimensional accuracy, were of very high quality. The flow, heated by radial heat conduction, was found to exhibit a stick-slip phenomena. This has been satisfactorily explained by theoretical considerations of the high elastic state of polymers. The mathematical technique and experimental data needed for determining the maximum output of the extruder while retaining a high quality extrudate is explained.

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Title: Associate Professor of Mechanical Engineering
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Any thesis is first and foremost an educational experience. As such, suggestion and insights come to the writer from many different sources. It is impossible to acknowledge all, but I would like to thank a few. First, to Nam Suh, my thesis supervisor, for his help, criticisms, and encouragements. To Jack Leach, Fred Anderson, Ralph Whitemore, and Ralph Bowley, I express my thanks for their assistance and advice in constructing the experimental apparatus. To my fellow students, I thank you for many thought provoking discussions. Thanks must also go to the Shell Companies Foundation and the Department of Mechanical Engineering for financial support and to Mr. Allan Margolies, Allied Chemical Co., for providing the polyethylene used.
Among the many new polymers introduced in recent years are polyethylenes with high molecular weights. Especially interesting are those with weight average molecular weights in excess of 1,750,000. Termed ultra-high molecular weight polyethylene (UHMWPE) [1], these polymers are seeing increasing usage because of their unique physical properties. In particular, UHMWPE with a molecular weight of 8 million, which like other polyethylenes, is very tough and chemically inert, has a coefficient of friction comparable to that of polytetrafluoroethylene (teflon) and a very high abrasion resistance.*

However, these long chained polymers are not easily processable [2]. Because of the very high viscosity of UHMWPE melts, they cannot be extruded in conventional screw extruders. In practice, they simply "jam up" in the extruder. The processing modes known to be in use are counter-rotating twin-screw extruders, compression molding, and a "screwless injection machine". Twin screw extruders are undesirable because the large shearing forces created break up the molecular chains to the point where molecular weight, and thus physical properties, are significantly altered. Compression molding has the traditional drawbacks of low production rates and of limitation

* This UHMWPE has been kindly provided by Allied Chemical and is sold under the designation AC 260-100.
to single parts. A "screwless injection machine" has been introduced by Borg-Warner Machines which utilizes mating conical shear surfaces to heat the plastic, a powder metering screw, and two stuffer plungers for transporting the powder and melt, respectively (Figure 1). This machine is a significant improvement in processing high viscosity polymers. However, it is still complicated, expensive and, more importantly, utilizes shear work to melt the polymer.

The aim of this research work has been the design, construction, and testing of a ram extruder for high viscosity polymers. In contrast to all of the above processing methods, it is simple, inexpensive, and introduces an absolutely minimal amount of shear work, thereby minimizing physical deterioration of the polymer.
FIGURE 1. Schematic of Screwless Injection Machine made by Borg-Warner Machines.
II. LITERATURE REVIEW

This chapter reviews previous work which has been relevant to the design and analysis of the ram extruder. Not surprisingly, no work has been found dealing with the principles of ram extruder operation. The areas searched were powder flow, powder compression, polymer properties, and polymer flow.

II.1 COMPRESSION AND FLOW OF GRANULAR MEDIA

The only work found in the area of powder flow dealt with the free flow of powdered and granular materials through hoppers and flow along moving surfaces (such as blades). Adler [3] reviews and analyzes the factors which affect the gravity flow of powders. Little work has been done concerning pressure flow of powders. Platonov and Poltorak [4] investigated the shear of granular media along a surface at low normal stresses. They found that the resistance to shear (coefficient of external friction) depended considerably on the normal pressure and also the rate of increase of the shear stress. They also discovered a two-phase process of flow. The first phase was a stick-slip flow accompanied by an increase in the shearing force. The second phase was uniform movement under a constant shearing force. One of the materials used was granular polystyrene.

The compression of powdered materials has received signifi-
cant attention. Schwartz and Holland [5], working with iron powders, show that the stress state for initial yielding is given by the Mohr-Coulomb yield criterion

\[ \gamma = C + \sigma \tan \phi \]  

where \( \gamma \) is shear stress, \( \sigma \) is normal stress, \( C \) is the cohesion at zero normal stress, and \( \phi \) is the angle of internal friction. However, \( C \) increases and \( \phi \) decreases as densification proceeds. As a result, the failure envelope curves down, but can still be reasonably represented by the classical Mohr-Coulomb yield criterion. Lee and Schwartz [6] applied the modified Mohr-Coulomb criteria in an analysis of powder rolling. Several papers [7,8, and 9] have studied the failure of solid polymers under hydrostatic pressure. The data taken shows reasonable agreement with the Mohr-Coulomb yield criterion.

Kawakita and Ludde [10] approach the problem of powder compression via pressure-volume considerations. They give pressure-volume equations for piston, tapping, and vibrating compaction. For piston compression, the following expression is given.

\[ C = \frac{V_o - V}{V_o} = \frac{abP}{1 + bP} \]
where $C$ is the degree of volume reduction, $V_0$ is the initial apparent volume, $V$ is the volume under pressure $P$, and $a$ and $b$ are empirical constants. However, this equation is entirely empirical, has not been related to the state of stress, and the constants have not been related to any physical properties of the material. Thus the usefulness of this expression is extremely limited.

In summary, previous work indicates that the deformation of powder compaction can be idealized through the use of the Mohr-Coulomb yield criterion.

II.2 POLYMER PROPERTIES

Knowledge of the physical properties of polymers leaves a great deal to be desired. Because of the wide number of polymers, co-polymers, and polymer mixes, data must be collected from individual manufacturers. In addition, variations of many properties with temperature, state of stress, and stress rate are not well understood, let alone readily available. Also the inherent non-linearity of such properties as viscosity has made the synthesis of useful constituitive relations very difficult. As a result, almost all design and analysis related to polymers and polymer flows begins with very limiting assumptions regarding the nature of the properties.

Viscosity

In general, the temperature-shear-shear rate variation of the viscosity of a polymer melt has been very difficult to
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characterize, even though a great deal of work has been done. Recently, a computer simulation of flow in extruders [11, 12, and 13] has utilized several power law models to give a single viscosity curve. Viscosity relations which have been proposed for non-Newtonian fluids are given in Table 2-4, reference [14]. The most common, primarily because of its "usability", is the power law model given below:

\[ \eta = \eta_0 |\dot{\gamma}|^{n-1} \]  \hspace{1cm} (2-3)

A common modification is to include temperature dependance based on the Arrenhius equation:

\[ \eta = A \exp(-\Delta E/RT)|\dot{\gamma}|^{n-1} \]  \hspace{1cm} (2-4)

The major problem with this model is that it does yield the empirically determined Newtonian behavior at high and low shear rates. The actual viscosity data for the UHMWPE, AC 260-100, is shown in Figure 2. For comparison, the power law model is also plotted. This model, using equation (2-4), is

\[ \eta = (1.53 \times 10^7) \exp(-22.8/T)|\dot{\gamma}|^{-0.7} \]  \hspace{1cm} (2-5)

The value of \( \Delta E \) was taken from McKelvey [14].
FIGURE 2. Viscosity Data and Power Law Model for UHMWPE AC 260-100.
SPECIFIC HEAT

A common value of specific heat for polyethylene given in the literature [15 and 16] is

\[ c_{PE} = 0.55 \text{ Btu/lbm }^\circ F \quad (T=25^\circ C) \quad (2-6) \]

The data in reference [16] shows the temperature dependance of \( c_p \) for polyethylene up to 320\(^\circ\)K. Forsythe [17] gives the temperature effect as

\[ c_p = c_{p0}(1 + bT) \quad (2-7) \]

However, no data has been found to support this equation in the temperature range of the polymer melt. Also, no work has been done to the author's knowledge on the relationship between molecular weight and specific heat. No data is given by the manufacturer for the specific heat of AC 260-100.

DENSITY

The specific gravity of AC260-100 is given as .930, obtained by A.S.T.M. D792-62T. The density is thus

\[ \rho_{PE} = s.g.PE \cdot \rho_{H_2O} = .93 \cdot .9975 = .928 \text{ gr/cc} \quad (2-8) \]

Naturally, due to compressibility effects, the density has a strong temperature and pressure dependance. Though this phenomena has been studied, data is not generally generated by the
manufacturers. Forsythe [17] expressed the density change as a function of pressure and temperature:

\[ \rho = \left( \frac{\partial \rho}{\partial T} \right)_P dT + \left( \frac{\partial \rho}{\partial P} \right)_T dP \] (2-9)

Substituting the definitions of thermal expansion and volume compressibility he obtained

\[ d\rho = -\rho \cdot \varepsilon \cdot dT + \rho \cdot \beta \cdot dP \] (2-10)

Values of the constants \( \varepsilon \) and \( \beta \), not being generally available, were obtained from manipulation of the Spencer-Gilmore equation of state as will be noted below.

**THERMAL CONDUCTIVITY**

The thermal conductivity data, and its variation with temperature, has not been well established. Nor has the mode of heat transfer in polymers been well explained. Values for the thermal conductivity of high molecular weight polymers are not available except within an order of magnitude. The temperature dependances found [18] are in such disagreement as to be meaningless.

Data for thermal conductivity of polyethylene in reference [16] show a decrease in \( k \) with increasing temperature in the range -100°C to 50°C. However, work by Fuller and Fricke [18], Lohe [19], and Hennig [20] in the temperature range 100°C to 300°C is in serious disagreement. As a result, no conclusions
can be made concerning the temperature dependance of the thermal conductivity of polymer melts.

Fuller and Fricke also noted that thermal conductivity decreased with increasing complexity of the molecular chain. Hanson and Ho [21] propose a theory, and present limited data, to show that thermal conductivity increases with molecular weight. They also give data that shows thermal conductivity decreasing with temperature until the melt temperature is reached, at which point the thermal conductivity starts increasing very slowly. In general, however, data is insufficient for drawing any hard conclusions.

CONSTITUIVITIE RELATIONS

The author has found only one attempt to develop a basic constituitive relation. In 1949, Spencer and Gilmore [22 and 23] proposed equation (2-11), the Spencer-Gilmore equation of

\[(P + \pi) \cdot (V - b) = \frac{R_g T}{M_w}\]  (2-11)

state, where \(P\) is the external pressure, \(V\) is volume, \(R_g\) is the gas constant, \(T\) is absolute temperature, \(M_w\) is the molecular weight, \(\pi\) is internal pressure or cohesive energy density, and \(b\) is volume at absolute zero. Equation (2-11) appears to have been arrived at from considerations of the ideal gas law

\[PV = RT\]  (2-12)
and Van der Waale's equation of state,

\[(P + a/v^2)(v - b) = BT\]  \hspace{1cm} (2-13)

a modification of equation of (2-12) for higher pressures. Though limited, this equation can be used to derive expressions for the coefficients of thermal expansion and volume compressibility and the density. The derivation of the two coefficients is straightforward, involving only substitutions of their definitions, giving

\[\frac{1}{\beta} = (P + \pi) \cdot \left[1 + \frac{M \omega b}{R g T} (P + \pi)\right]\] \hspace{1cm} (2-14)

and

\[\frac{1}{\epsilon} = T + \frac{M \omega b}{R g} \cdot (P + \pi)\] \hspace{1cm} (2-15)

An expression for density can also be found (by combining equation (10), (14), and (15) and integrating). However, the resulting expression is much too complicated to be of any use. It should furthermore be noted that the Spencer-Gilmore equation of state has never been tested for polymers with ultra-high molecular weights.
II.3 Flow Behavior of Polymers

At the beginning of this work, the author reviewed many papers on the flow characteristics of polymer melts. However, later experimental work showed that the flow in the ram extruder exhibited a stick-slip phenomena. Since the work on viscous flow is not applicable, only a brief review of major work will be given.

In 1951, Brinkman [24] solved the steady state flow problem for a Newtonian fluid with viscous heat generation. The governing equation

\[ \frac{\partial}{\partial x} \left( \frac{3}{r} \frac{\partial T}{\partial r} \right) + \frac{r^2}{4n} \left( \frac{dP}{dz} \right)^2 = 0 \]  \hspace{1cm} (2-16)

was solved by evaluating an eigenvalue series for boundary conditions (1) where the tube walls are at \( T_0 \) and the entering fluid is at \( T_0 \) and (2) where the walls are insulated. The results showed very significant temperature rises due to the viscous heat generation. Bird [25] extended Brinkman's work to the case of non-Newtonian flow characterized by a power law viscosity.

Toor [26] investigated the effects of compressibility on the temperature profiles of power law fluids. The governing energy equation used was

\[ 0 = \frac{k}{r} \frac{\partial}{\partial r} \left( \frac{3}{r} \frac{\partial T}{\partial r} \right) + \frac{n+2}{n} \left( \frac{2T_c + n}{2} \right) \left( \frac{r}{R} \right)^n - T_c \]  \hspace{1cm} (2-17)

where \( n \) is the power law index, \( \varepsilon \) is the coefficient of thermal
expansion, and $W$ is the average volume rate of energy dissipation across the tube. Toor [33] continues this work by examining the inlet region in order to determine the distance necessary to reach steady state flow. He solves equation (2-17), amended by the addition of the enthalpy transport term, for a fluid entering a tube with constant wall temperature equal to that of the entering flow. He shows that that the center temperature initially decreases due to expansion, but then increases with axial distance due to viscous heat generation. The Reynolds number is a measure of distance required to reach a steady velocity profile and the Peclet number is a similar measure of the development of the temperature profile. Toor develops an equation for the distance required to reach steady state profiles and notes basic theoretical agreement with the dimensionless group analysis. For high viscosity fluids, the velocity profile develops much more rapidly than the temperature profile. The Prandtl number is pointed out as a measure of the relative rates of development.

Lyche and Bird [27] obtained a semi-analytical solution for the Graetz-Nusselt problem extended to non-Newtonian flow. Reduced temperature profiles for heating and cooling for different power law indexes are given. A final comment pointed out the "urgent need for experimental data on thermal conductivities and heat capacities of non-Newtonian fluids." Fifteen years later this comment is still very applicable. In 1958, Toor [28] presented a heat transfer analysis of power law non-Newtonian fluids with viscous heat generation in a pipe. Gee
and Lyon [29] derive the basic equations governing the flow under discussion here and give a computer solution thereof for a particular material.

Christiansen et.al. [30,31, and 32] have worked on non-Newtonian flow problems but neglected the effects due to viscous heat generation and compressibility which have been shown to be important [24,25,26, and 33]. Gruntfest et.al. [34] present a transient analysis of a power law fluid with viscous heat generation. The work was based partly on that of Kearsley [35]. Forsythe [17 and 36] developed a numerical solution for steady-state flow of a temperature dependent power law fluid with viscous heat generation and compressibility effects. The results showed improvement over the methods of Bird [25] and Toor [26] for the cooling of polymers. These results, though agreeing well with experiments, are limited by the nature of a numerical solution--application to a particular material.
III. EXPERIMENTAL WORK

III.1 Basic Concept of the Ram Extruder

The ram extruder was designed for continuous processing of high viscosity polymers. The usefulness of such a machine was noted in the introduction. The basic concept may be illustrated through the use of Figure 3. A reciprocating ram (1), on every return stroke, uncovers a port (3) in the feed block (5) through which powder in the hopper (2) falls into that section of the chamber denoted as A. On the forward stroke, the ram pushes the powder forward. After a few strokes, the wall friction of the powder is sufficient to cause a pressure build-up in section B every time the ram pushes forward, thereby compacting the powder. This continuous slug of compacted powder is then forced through a heated tube (4) where the polymer is melted.

III.2 Design

Figure 4 shows the basic extruder design. Appendix A contains detail drawings of the parts and Appendix B is a list of standard equipment used. Figures 5 and 6 are photographs of the experimental apparatus, including instrumentation.

The extrusion tube was 6 inches long with a 1 inch OD and a $\frac{1}{4}$ inch ID. Four 125 watt band heaters were used. Two iron-constantan thermocouples with off-on controllers monitored
FIGURE 3. Schematic Conception of the Ram Extruder

- HOPPER (2)
- FEED BLOCK (5)
- RAM (1)
- HEATED TUBE (4)
- PORT (3)
- POWDER
FIGURE 5. Top View of Apparatus

FIGURE 6. Bottom View of Apparatus
the barrel temperature. Thermocouples A and B were mounted 1\(\frac{1}{4}\) inches from the exit and entrance ends of the tube, respectively. The tip of the thermocouple probe was \(\frac{3}{16}\) inch from the inside wall.

The thermal insulator between the feed block and the extrusion tube was included for two reasons. First, it was desired to maintain as sharp a temperature gradient as possible for later theoretical considerations. Second, melted polymer near the piston causes the piston to jam in the cylinder. The feed block was water cooled to prevent any temperature rise in this part. (During start up it was always necessary to turn on the water before powering up the heaters.)

Several designs and materials for the thermal insulator were tested. These included teflon, steel, machinable glass, water cooled epoxy, and water cooled steel sleeve with a teflon insert. Ceramic materials were judged to be unsuitable. Most of the materials were not acceptable because of low mechanical strength, high thermal conductivity, surface roughness, or brittleness. The only successful design was the water cooled steel-teflon composite. (See Appendix A.) However, the low strength teflon tended to flow out between the steel sleeve and the heated extrusion tube. Fortunately, the polyethylene filled this space without apparent effect on the flow. The design was thus suitable for testing purposes though in practice a more durable design would be necessary.

Some problems were encountered in the free flow of powder through the port in the feed block, particularly after the
powder had absorbed humidity. All difficulties were eliminated by simply enlargening the port.

Significant problems resulted from the dry sliding friction between piston and cylinder. Initially, both parts were made of mild steel. However, after a break-in period piston seizures were repeated and rapid. Replacement with a brass piston and additional securement of the bushing prevented further problems. Periodic inspection of this arrangement showed signs of wear on the piston which indicated that the design was deficient. The problem is not overwhelming. Use of dual bushings to eliminate non-axial forces and use of hardened steel or low friction materials (such as teflon impregnated aluminum) should eliminate difficulties in the future.

The air motor was run off an 80 psig line through a regulator. Valving on the air motor is controlled by two micro-switches accuated by the lever arm. By adjusting the switch positions, the stroke distance can be controlled. Shaft speed is influenced by supply pressure, adjusting screws which restrict the flow passages, and load. As a result, fine adjustment of the piston speed was quite difficult. Future apparatus should employ a much less sensitive speed control.

The lever arm was designed to give a force magnification and stroke reduction factor of 3.
III.3 Instrumentation

Among the variables to be recorded were piston velocity, extrudate velocity, piston displacement, and piston force.

The velocities were measured with a self-generating, magnetic core type transducers (ISA#1-313 [37]). This type of transducer is suitable for uses where essentially no loading of the measured parameter can be tolerated. The instrument specifics are given in Appendix B. Due to the set-up of the apparatus, it was necessary to mount the transducers in parallel with the moving piston and extrudate. A bent arm, rather than straight shaft, coupling was thus employed. (Figures 5 and 7.) Initial problems were encountered due to the spring effect of these arms. The arms were strengthened at the bend and a teflon guide was inserted in the transducer to prevent lateral motion. This eliminated all problems except that of piston velocity overshoot at the beginning of the return stroke. Since the unloaded piston velocity is a constant, this overshoot was obvious and could simply be ignored. The transducer outputs were fed into an oscilloscope with camera attachment. 60 cycle pickup was 12 mv_p-p for piston velocity and 4 mv_p-p for extrusion velocity.

An LVDT (ISA#1-034) was used to measure piston displacement. Output was fed into a Sanborn recorder and calibrated with depth micrometer measurements of piston position.

Piston force was to be calculated from measurements of the differential pressure across the air motor piston. Pressure taps were located in the cylinder walls at each end of the
motor and connected via $\frac{1}{8}$ "Poly-Flo" tubing to a differential pressure transducer. (See Figure 6.) Output was fed to the Sanborn recorder and calibrated by comparison to a calibrated Bourdon tube gage at 20 psid. The transducer was mounted separately from all other apparatus to prevent vibration pick-up.

### III.4 Tests and Results

A series of tests were run to determine the operating characteristics of the ram extruder. The factors varied were the stroke length, the barrel temperature, and the speed or cycling rate. Stroke length was adjusted with the extruder operating by moving the valve controlling micro-switches. The travel was read directly from the LVDT output. The extruder tube temperature was controlled by setting the temperature controllers. The speed of operation, as noted above, was difficult to adjust. This was accentuated by the fact that if the machine began producing a partially melted extrudate, the process usually, even when slowed down, went to a new steady state situation where only a thin outer layer of the extrusion was melted. The author attempted to set the air motor speed adjusting screws and the regulator pressure to yield a maximum output condition for a given stroke and temperature. Measurement of the success in doing so is discussed below.

Variation of differential pressure and piston position were continuously recorded versus time. Pictures of piston
and extrudate velocities were taken during the test period of each specimen. The specimens were marked, during the piston return stroke, with a knife. A notation was made on the pressure trace to indicate at which cycle the specimen was marked. Appendix C contains the specimen notation, temperature conditions, and readings taken directly from the output recordings and the specimens. These later include time, cycles, stroke, pressure, and specimen length, diameter, and weight. Calculations included are frequency, mass flow, average extrusion velocity, and residence time.

Typical velocity traces under different conditions are shown in Figures 8, 9, and 10. The upper trace is piston velocity, the lower is extrudate velocity.

Measurements of the density of samples from each specimen were made. The method and results are included in Appendix D.

Also, at the end of tests for a given stroke the partially melted polymer was pulled out of the tube. Only about $\frac{1}{4}$ inch of the specimen, at the tube entrance, did not pull out. By slicing the extrudate axially, the axial point at which the polymer first becomes completely melted was found. Presumably, in the ideal situation the polymer would first become completely melted at the tube exit. On this basis, the speed of operation was judged to be about 30-40% below the maximum speed.

The specimens themselves were of very high quality. Dimensional tolerance was within .001 inch. Surface quality was excellent. A few small bubbles were noticed in the hot extrudate for a short (5/16) stroke. With the longer strokes,
no bubbles were noticed at all. Density measurements, however, showed no dependance on stroke. A large amount of iron oxide did appear on the inside wall of the steel extrusion tube. However, none of the specimens were contaminated in any way.

III.5 LIMITATIONS

There are two primary limitations in this experimental work. First, difficulty in controlling the speed of operation, and therefore the residence time of the polymer in the heated section, prevented the extruder from attaining the maximum output steady state condition. Second, the four 125 watt heaters were insufficient to keep the barrel temperature much above 450°F. As a result, higher temperature ranges (at which, for short times, the polyethylene will not decompose) were not studied.
FIGURE 3a.
Test No. 203
Vertical Scale: .667 in/sec/cm
Sweep Speed: .5 sec/cm
Temperature:
  Thermocouple A: 400°F
  Thermocouple B: 400°F
Stroke: .311 in

FIGURE 3b.
Test No. 221
Vertical Scale: .667 in/sec/cm
Sweep Speed: .5 sec/cm
Temperature:
  Thermocouple A: 450°F
  Thermocouple B: 450°F
Stroke: .315 in

FIGURE 3c.
Test No. 242
Vertical Scale: 1.33 in/sec/cm
Sweep Speed: .2 sec/cm
Temperature:
  Thermocouple A: 475°F
  Thermocouple B: 460°F
Stroke: .320 in
FIGURE 9a.
Test No. 303
Vertical Scale: .667 in/sec/cm
Sweep Speed: .5 sec/cm
Temperature:
Thermocouple A: 400°F
Thermocouple B: 490°F
Stroke: .488 in

FIGURE 9b.
Test No. 321
Vertical Scale: .667 in/sec/cm
Sweep Speed: .5 sec/cm
Temperature:
Thermocouple A: 450°F
Thermocouple B: 450°F
Stroke: .488 in

FIGURE 9c.
Test No. 343
Vertical Scale: .667 in/sec/cm
Sweep Speed: .2 sec/cm
Temperature:
Thermocouple A: 475°F
Thermocouple B: 460°F
Stroke: .488 in
FIGURE 10a.
Test No. 402
Vertical Scale: .667 in/sec/cm
Sweep Speed: .5 sec/cm
Temperature-
Thermocouple A: 400°F
Thermocouple B: 400°F
Stroke: .582 in

FIGURE 10b.
Test No. 422
Vertical Scale: .667 in/sec/cm
Sweep Speed: .5 sec/cm
Temperature-
Thermocouple A: 450°F
Thermocouple B: 450°F
Stroke: .582 in

FIGURE 10c.
Test No. 443
Vertical Scale: .667 in/sec/cm
Sweep Speed: .5 sec/cm
Temperature-
Thermocouple A: 475°F
Thermocouple B: 460°F
Stroke: .574 in
IV. ANALYSIS

IV.1 INAPPLICABILITY OF VISCOUS FLOW ANALYSIS

It was initially supposed that the flow of polymer out the tube would behave as standard non-Newtonian fluid flow. Therefore, the governing equations were developed [38] assuming axi-symmetrical conditions, negligible axial heat conduction, negligible radial velocity and steady state. Expressions for the effects of compressibility and thermal expansion were developed following Toor [26]. The equations of continuity, momentum, and energy then reduced to,

\[ \frac{\partial}{\partial z}(\rho z) = 0 \]  
\[ \rho v_z \frac{\partial v_z}{\partial z} = \frac{1}{r} \frac{\partial}{\partial r}(r \tau_{rz}) - \frac{\partial P}{\partial z} \]  
\[ \rho c_v v_z \frac{\partial T}{\partial z} = \frac{1}{r} \frac{\partial}{\partial r}(kr \frac{\partial T}{\partial r}) - T \cdot \frac{\partial}{\partial z} \tau_{rz} + \tau_{rz} \frac{\partial v_z}{\partial r} \]

respectively, where \( \tau_{rz} = n \cdot \frac{\partial v_z}{\partial r} \), \( n \) is the viscosity, \( \varepsilon \) is the coefficient of thermal expansion, and \( \beta \) is the compressibility.

An order of magnitude analysis was then performed for UHMWPE under the approximate test conditions. The continuity equation was rewritten as
following Forsythe [17]. The analysis showed that $\frac{\partial v_z}{\partial z}$ was negligible in both the energy and momentum equations and hence that the temperature changes due to compression and expansion were negligible. The resulting momentum and energy equations reduced to:

$$
\frac{\partial p}{\partial z} = \frac{1}{r} \frac{\partial}{\partial r} (r \cdot \tau_r) \tag{4-5}
$$

$$
\rho c_v v_z \frac{\partial T}{\partial z} = \frac{1}{r} \frac{\partial}{\partial r} \left( k r \frac{\partial T}{\partial r} \right) + \tau_r \frac{\partial v_z}{\partial r} \tag{4-6}
$$

However, over the temperature range (300-450°F) for which the extruder was tested, the flow conditions were not describable by equations (4-5) and (4-6). At test temperatures of 400° and above the flow exhibited a definite stick-slip phenomena. This can be seen by comparing the piston and extrusion velocities (upper and lower traces, respectively) in Figures 8, 9, and 10. In the range of 300-325°F the flow appeared to be viscous flow. But inspection of the extrusion tube showed a heavy iron oxide coating on the interior wall. Since the extruded polyethylene rod was clean and showed no signs of contamination, it must be assumed that the UHMWPE was not adhering to the tube walls. The flow was thus not sticking to the wall and application of a viscous flow analysis to this problem is impossible.
IV.2 The High Elastic State and Stick-Slip Flow

The purpose of this section is to explain the nature of and reasons for the stick-slip flow observed in the experiments. The phenomena of stick-slip flow has received almost no attention in the literature. However, a very recent paper by Vinogradov, et. al. [39] sheds a great deal of light on the subject. In particular, it is shown that under certain conditions a polymer in the high elastic state can exhibit stick-slip flow.

Linear uncured polymers, with molecular weight above a critical value and at temperatures above the glass transition temperature, can exist in the fluid and high elastic states. The high elastic state is characterized by the rubbery nature of the polymer and by the reversible nature of deformations. In the fluid state, deformations are of course irreversible.

The critical molecular weight for transition to the high elastic state is given as

\[ \frac{M}{M_c} = \frac{M}{2M_e} > 10 \] (4-7)

where \( M \) is the molecular weight, \( M_c \) is the critical molecular weight corresponding to the change in the nature of the viscosity power dependance on molecular weight, and \( M_e \) is the molecular weight of a chain section between nodes (entanglements). The values of \( M_c \) and \( M_e \) for polyethylene are extremely low, about 2,000 and 4,000 respectively. Thus flow instability has
has been widely noted for polyethylene. For AC260-100, we find that since

\[ \frac{M}{M_c} = 4,000 \gg 10 \]  \hspace{1cm} (4-8)

this UHMWPE easily meets the necessary criterion for possible transition to the high elastic state.

Transition of a polymer system from the fluid state to the high elastic state is dependent on the shear stress and the shear strain rate. The critical values of these parameters for transition to occur are denoted \( \tau_c \) and \( \dot{\gamma}_c \). Vinogradov and co-workers state that the critical shear stress, \( \tau_c \), does not increase with molecular weight and is, in fact, independent of the molecular weight. It is also suggested that \( \tau_c \) should be relatively insensitive to temperature. The critical shear strain rate, \( \dot{\gamma}_c \), is however dependent on molecular weight. The expression

\[ \dot{\gamma}_c = M^{-\alpha} \]  \hspace{1cm} (4-9)

is given. An effect of this relationship is that a narrow molecular weight distribution (MWD) implies a sharp change from the fluid state to the high elastic state. Since the initial viscosity dependance on molecular weight is

\[ \eta_0 = M^\alpha \]  \hspace{1cm} (4-10)
(where $a$ is the power dependance of initial viscosity on the molecular weight), it is also found that the critical shear strain rate is inversely proportional to viscosity and is therefore proportional to temperature, equation (4-11).

$$\dot{\gamma}_c = \eta_0^{-1} \propto T \quad (4-11)$$

Transition to the high elastic state also causes a change in the nature of deformation. The primary changes are non-adhesion to solid surfaces and extrudate distortion. The latter has been referred to in [39] as elastic turbulence. In the literature, extrudate distortion has been generally referred to as flow instability (such as the so-called melt-fracture).

The experimental work by [39] utilized a constant pressure drop apparatus to push a narrow MWD polybutadiene through a rectangular duct. The flow rate was measured and the nature of the flow and extrudate distortion was observed. From the data, a flow rate--pressure drop curve was obtained and is reproduced here as Figure 11. The branch of that curve denoted I is the region of viscous flow. Branch II is the spurt region, with stick-slip flow progressing to continuous slippage as the pressure drop increases. Branch III is characterized by very strong extrudate distortions. The orthogonal arrows on the curve indicate the beginning of small scale distortions in the extrudate during viscous flow. Deformation in branch II, however, is confined to the rounding off of the corners of the
FIGURE 11. Flow rate dependance on pressure drop for polybutadiene [39].
rectangular extrusion. Due to the major change in the nature of deformation going from branch II to branch III, it is convenient to define a second set of critical parameters, \( \tau_C \) and \( \dot{\gamma}_C \), for transition between the two regions.

IV.3 The Theory of the High Elastic State Applied to the Ram Extruder

In this section, the nature of the high elastic state as explained by [39] will be compared to the results from this experimental work. Similar to the experimental work of [39], this work uses a device which applies a relatively constant force to the polymer throughout the extrusion stroke. Also in this section, the possible effects of the wall-polymer friction factor on the pressure gradient and the iron oxide coating on slippage at the wall will be considered.

Two situations from this experimental work are noted. First, at temperatures of 400°F and up, the flow was stick-slip. Second, at temperatures of 300-325°F both continuous slippage at the exit and some surface ruptures were observed. There are two possible explanations which account for these observations. The first explanation takes into account only the nature of the high elastic state, while the second includes considerations of the polymer-metal coefficient of friction and the pressure gradient.
EXPLANATION ONE

In both cases (high and low temperature), the first set of critical parameters for transition to the high elastic state are obviously passed. At the higher temperature, stick-slip flow is observed and the polymer is therefore in region II of Figure 11. At the lower temperature, continuous slippage and surface distortion are noted. This suggests that the polymer is in the area where regions II and III meet.

These observations may be explained as follows. Since $T_c$ is not temperature dependent, we might well assume that $T_c$ (the critical shear stress for ruptured extrudate) is also independent of temperature. And since pressures for both cases are roughly equal, we can assume that the shear stresses are not very different. Thus the important parameter is $\dot{\gamma}_c$ (the critical shear strain rate for a ruptured extrudate).

From section IV.2, equation (4-11), we note that the critical shear strain rate, $\dot{\gamma}_c$, is temperature dependent. Let us assume this is also true for $\dot{\gamma}_c$. Then since piston velocities were roughly the same, the shear strain rates induced would not be very different. Therefore, raising the barrel temperature could have raised the second critical shear strain rate, $\dot{\gamma}_c$, such that the polymer was in region II--spurts without serious distortion.
EXPLANATION TWO

In this explanation, the reasoning above is accepted with the exception that the strong temperature dependance of $\gamma_C$ assumed above is taken to be insufficient in explaining the observed phenomena. Now the coefficient of friction between metals and polymers decreases with temperature after the polymer melting temperature is exceeded. Then for a given applied pressure, the pressure gradient will become steeper as the temperature decreases. For low temperature, therefore, the state of stress would be very high near the tube entrance and low elsewhere. As the temperature is increased, the pressure gradient and the maximum stress decrease and a larger portion of the polymer is subjected to higher stresses.

Observations indicate that for temperatures around 400°F, the state of stress is sufficient to put a large part of the polymer in region II. For temperatures around 300°F, a smaller portion of the polymer would be in a much higher stress state which could account for the distortions observed. The continuous slippage noted at this temperature can be simply attributed to a combination of the non-adhesiveness of the polyethylene and the iron oxide acting as a dry lubricant. It should be pointed out that the non-adhesiveness of the polymer may become pronounced well below $\tau_C$ and $\gamma_C$ due to the MWD.

The author feels that the second explanation is more reasonable. However, the data is really insufficient for drawing a conclusion.
IV.4 HEATING TIME

Of utmost importance in the design of this extruder is the time necessary to melt the polymer. This is referred to as the residence time (of an infinitely thin disk in the heated tube).

Comments regarding the nature of gradients in the extruder tube are in order here [40]. First, using a maximum heat flux into the polymer (1.6 Btu/hr) the radial temperature gradient was found to be completely negligible. Second, the gradient at the tube entrance was found to be significant, as follows. Temperature conditions and physical dimensions are shown in Figure 12. If the radial gradients at the thermal insulator--extruder tube interface are small, 98% of the heat flux in region A will go through the steel sleeve. We can now equate

![Diagram](image)

FIGURE 12. Calculation of Tube Entrance Temperature Gradients
expressions for heat flux in the two regions provided we select an equivalent area for region B. Averaging the interface area with the area of the extruder tube, we obtain $A_B = .56$ sq. in. $T$ is then given by

$$T = \frac{A}{\Delta x} A \frac{B}{70 + A \frac{400}{\Delta x B}}$$  \hspace{1cm} (4-12)

which yields $T = 215^\circ F$. Taking into account the fact that the sleeve is cooled by assuming $\Delta x_A = \frac{1}{2} \Delta x_A$, we find $T = 160^\circ F$. Thus it seems that "shortening" of the heated section by about 10% would be reasonable.

As noted in Chapter III, the specimens were run 30-40% too slow. Thus another correction factor, of about 35%, is necessary to obtain the effective residence time:

$$t_{\text{corrected}} = t_{\text{measured}} \cdot (.9)(.65) \hspace{1cm} (4-13)$$

We can now treat the problem as a cylinder at constant temperature subjected to a sudden increase in the wall temperature. Carslaw and Jaeger [41] have solved this problem. The solution for average and center temperature is plotted with $\frac{V}{V}$ versus $S$ as the coordinate axes where
\[ \frac{V}{V} = \frac{T - T_i}{T_w - T_i} \]  
\[ S = \frac{\alpha \cdot t}{r^2} \]

T is taken to be either \( T_{ctr} \) or \( T_{ave} \), \( \alpha \) is the thermal diffusivity, \( t \) is the corrected residence time, and \( r \) is the cylinder radius.

Residence times for the tests at 400°F and 450°F were averaged. At 400°F the average residence time was 82.0 sec and at 450°F it was 63.8 sec. Applying equation (4-13), the effective residence times are found to be 48.0 sec and 37.3 sec. The thermal diffusivity of polyethylene is approximately \( 0.01 \text{ ft}^2/\text{hr} \). Calculating \( S \), we find \( (S \text{ at } 400° \text{ is } 1.23) \) and \( (S \text{ at } 450° \text{ is } 0.96) \). These values of \( S \) indicate that a flat temperature distribution exists in the polymer [41].

A flat temperature distribution could be explained in several ways. First, the melting temperature, \( T_m \), is time dependent. Thus the melting temperature for a given residence time could be very close to the wall temperature. The actual value of \( T_m \), at this time, can only be stated as being above 300°F and below the wall temperature (400° or 450°). Second, the non-adhesion of the polyethylene and the presence of the oxide layer indicate the possible existence of a significant contact resistance. Third, the thermal conductivity, and thus the thermal diffusivity, could be in error by as much as a factor of 2. In all probability, all three explanations probably
contribute to obtaining the calculated results. Modification of the experimental apparatus and further testing is definitely necessary. However, it is still believed that the polymer temperature increase is caused solely by radial heat conduction and therefore the idealization of the problem made above is valid.

IV.5 Powder Compression and Air Expulsion

During all the tests, no problem was ever encountered due to inadequate powder compression or entrapped air. As mentioned in the literature review, the powder compression can be analyzed by use of the Mohr-Coulomb yield criterion. Application of this criterion to powder compaction in a cylinder shows that the pressure drop is exponential.

The expulsion of air from the powder is explained as follows. Initially the powder is compressed to near its theoretical density. (Preliminary data indicate that the compaction results in an initial density of 90-95% of the theoretical density.) As the temperature increase in the powder, the air diffuses back through the powder to the tube entrance and escapes past the piston. This temperature activated diffusion process completely degases the polymer.

IV.6 Maximum Output Conditions

The residence time necessary for melting of the powder is proportional to the radius squared and is also related to the
values of the melting temperature and the wall temperature via the solution to the heating problem of an infinite cylinder. If the melting temperature--time and degradation temperature--time relationships are found, then an iterative graphical solution technique may be used to find the minimum residence time. The only other necessary consideration is to make sure that the shear stress and shear strain rate do not move the polymer into range of extrudate distortion--branch III in Figure 11. The theoretical background for this is simply

\[ t_m = f(T_m, T_d, \alpha) = \frac{V}{L} \quad (4-16) \]

where \( t_m \) is the minimum residence time, \( v \) is the average extrusion velocity, \( L \) is the barrel length, \( T_m \) is the melting temperature, \( T_d \) is the degradation temperature, and \( \alpha \) is the thermal diffusivity. Now, since \( \dot{\gamma}^c \) is inversely proportional to viscosity, even a low extrusion velocity will cause of shear strain rate exceeding \( \dot{\gamma}^c \) for the case of high viscosity polymers. Therefore the shear stress will be the primary factor controlling whether the polymer is in the region of stick-slip flow or in the region of extrudate distortion. The shear stress is primarily affected by the pressure drop which depends on the barrel length. Increasing the length of the extrusion tube would raise the pressure drop and therefore the shear stress would increase. For a given barrel temperature, the output mass flow is limited by that length of the extrusion tube
which causes a pressure drop corresponding to the critical shear stress. Much more experimental work will be necessary to establish all of the functions needed to determine the maximum flow rate.
V. CONCLUSIONS

The ram extruder has been shown to be feasible for the extrusion of high viscosity UHMWPE in cylindrical form. It is expected that other high viscosity polymers can also be extruded. The nature of the flow has been found to be stick-slip due to the fact that the polyethylene is in the high elastic state.

Heating of the polymer is by means of radial heat conduction. Thus the time required to heat the polymer is proportional to the square of the radius. As a result, the extruder mass flow is greatest for thin extrusions.

The piston driving source should have separate velocity controls for extrusion and return strokes and a constant force should be applied by the piston for maximum output. The return stroke speed is limited by the time required for the powder to fall through the port.

Maximum output may be calculated, if the melting temperature--time and degradation temperature--time relations are known, through use of the solution to heating of an infinite circular cylinder. Extrudate quality can be kept high (i.e. the polymer stays in the region of stick-slip flow) by appropriately limiting the barrel length.
## APPENDIX A

### DETAIL DRAWINGS

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<tr>
<td>Displacement Transducer Mount</td>
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</table>
Extrusion Tube

Material: Steel
Make (1)
Tolerances: Fractions ± 1/64
Decimals ± 0.01
Piston
Mat: Brass
Make (1)
Tolerances: Fractions ± 1/64
Decimals ± 0.001

Air Motor Adapter
Mat: AL
Make (1)
Tolerances: Fractions ± 1/32
Decimals ± 0.001
LEVER
MAT: 3/8 AL. PLATE
MAKE (2)
TOLERANCES: FRACTIONS ± 1/16
DECIMALS ± .01
UNLESS OTHERWISE INDICATED
Radius \( \frac{3}{8} \), minimum

\( \frac{3}{4} \) Ream

Connecting Rod

MAT: 1/4 AL. PLATE
MAKE (x)

TOLERANCES: FRACTIONS \( \pm \frac{1}{32} \) DECIMALS \( \pm .01 \)

Bushing

MAT: BRONZE
MAKE (1)

TOLERANCES: FRACTIONS \( \pm \frac{1}{64} \) DECIMALS \( \pm .001 \)

6

5.00

\( \frac{1}{2} \) Ream

\( \frac{3}{8} \)
Extruder Tube Mount

Material: 3/16 Steel Plate

Make: 1

Tolerances: Fractions ± 1/6
Decimals ± 0.01

Lever Mount

Material: Steel

Make: 1

Tolerances: Fractions ± 1/32
Decimals ± 0.001
Feed Block

Mat: steel

Make (1)

Tolerances: fractions ± 1/32, decimals ± .01

Note: ■ indicates brass plug

1/4 ream, polish
1/8 NPT (2), depth not 1/2 (this view only)
1/64 drill (4)
\textbf{Thermal Insulator}

\textbf{Mat: Steel \[\text{\textbullet}\]}

\textbf{Teflon \[\text{\textbullet\textbullet}\]}

\textbf{Epoxy (High Temperature and High Thermal Conductivity) \[\text{\textbullet}\]}

\textbf{Make (1)}

\textbf{Tolerances: Fractions \(\pm\) 1/32. Decimals \(\pm\) 0.001}
VELCITY TRANSDUCER MOUNT
MAT: 3/8 AL. PLATE
MAKE (4)
TOLERANCES: FRACTIONS ± 1/32
DECIMALS ± .01

DISPLACEMENT TRANSDUCER MOUNT
MAT: AL.
MAKE (1)
TOLERANCES:
FRACTIONS ± 1/64
DECIMALS ± .01
APPENDIX B

EQUIPMENT LIST

UHMWPE

AC 260-100, Allied Chemical Co.
Weight Average Molecular Weight of 8,000,000

Air Motor

Piston type, Bellows-Valvair Inc.
Bore: $\frac{3}{8}$ in.
Stroke: 2 in.
Force: up to 1500 lbs at 150 psig.
Valving: electrically initiated.
Speed: wide, variable range.

Heaters

Band type, Watlow Inc.
Model: 11344G
Rating: 125 watts at 120 volts.
Dimensions: 1 in. ID, 1 in. width.

Thermocouples

Iron-constantan.

Temperature Controllers

Off-on control type.
West Model JP and
Barber-Colman Model 297C.

Pressure Transducer

Differential type, Fredric Flader Inc.
Model: PSB
Range: 0-160 psi.
Displacement Transducer
LVDT, Hewlett-Packard Inc.
Model: LinearSYN 585DT-1000.
Stroke: 1 in.
Non-linearity: less than 1%.

Velocity Transducers
Magnetic core, Hewlett-Packard Inc.
Model: LVSYN 7LV6-NB6.
Stroke: 6 in.
Non-linearity: less than 1%.
Sensitivity: 150 mv/in/sec within 5% over full stroke.

Recorder
Sanborn 321 Dual Channel Carrier Amplifier-Recorder.

Oscilloscope
Tektronix Type 564 Storage Scope with Camera C-27.
APPENDIX C

TEST CONDITIONS, RESULTS, AND CALCULATIONS

Specimen Nomenclature:

Test specimens were labeled with three digit numbers. The first digit (2,3, or 4) indicates the nominal stroke and the second digit (0,2, or 4) indicates temperature. The last number specifies a particular specimen in a test set.

Test conditions, results and measurements are on pages C-2 through C-4. Specimens 202, 342, and 402 were incompletely melted.
<table>
<thead>
<tr>
<th>Test#</th>
<th>Stroke (in.)</th>
<th>Thermocouple Temperature(°F)</th>
<th>Total Number of Cycles</th>
<th>Total Time of Test (sec)</th>
<th>Average Piston Frequency (Hz)</th>
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APPENDIX D

DENSITY MEASUREMENTS

Two 1.25 inch (nominal) samples were cut from each specimen. The samples were taken approximately one-third of the way in from the ends of the specimens. The density was then found by the hydrostatic weighing technique.* The specimen is first weighed in a liquid, then in air. Knowing the liquid density, one may then calculate the specimen density. The equation is

\[ \rho_s = \frac{\rho_l \cdot m_s}{m_b + m_s - m_{\text{net}}} \]  

(D-1)

where \( \rho_s \) = density of specimen,
\( \rho_l \) = density of liquid,
\( m_s \) = mass of specimen,
\( m_b \) = mass of basket (specimen holder) in liquid, and
\( m_{\text{net}} \) = mass of specimen and basket in liquid.

The apparatus has been used to measure densities to an accuracy of \( 10^{-5} \) gr/cc. The author, due to the large number of specimens, attempted to achieve a final accuracy of \( 10^{-4} \) gr/cc. Specimen mass was corrected for the bouyancy effect of air using

*Apparatus at the Materials Science Center, MIT, was used. The assistance of Dr. J.A. Kalnajs is gratefully acknowledged. For a description of the apparatus see A.Smakula and V.Sils, "Precision Density Determination of Large Single Crystals by Hydrostatic Weighing", Phys.Rev., 99, #6 (1955).
where \( \rho_w \) is the density of the weights used. A Mettler H16 balance (±0.02 mg), with stainless steel weights, was used for these measurements. The buoyancy effect of air in the hydrostatic weighings was less than the error involved and was therefore ignored. Ethanol was used for the liquid and a Wang calculator for calculations. All masses are in grams; all densities in grams per cubic centimeter. Constants for all calculations are:

\[
\begin{align*}
\rho_l &= 0.78505 \\
\rho_{air} &= 0.001197 \text{ (for test conditions)} \\
\rho_w &= 7.77 \\
m_b &= 2.17920
\end{align*}
\]

The results are tabulated on the following page.
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(Theoretical density is .928 gr/cc.)
REFERENCES


21. Hanson, D., and Ho, C. C., J. Poly Sci, A3 (1965)


