SECTION 1

A STUDY OF THE CRITICAL PROCESS VARIABLES IN THE HYDROSTATIC EXTRUSION OF SEVERAL MATERIALS

SECTION I

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VI

SUMMARY SECTION 1

For this phase of the program, a range of materials was selected and the various parameters controlling their effective extrusion into round bar were examined. The materials ranged from relatively easy-to-work materials, such as 7075-0 aluminum, to high-strength alloys such as superalloys, and brittle materials such as beryllium. The aim with every material was to achieve a sound, high-quality product at minimum pressure levels and to determine the technology required to achieve these aims.

The critical process variables controlling effective extrusion were thoroughly evaluated with three basic materials:

(a) 7075-0 aluminum
(b) AISI 4340 steel
(c) Ti-6Al-AV titanium alloy.

The variables evaluated with these materials were:

(1) Extrusion ratio
(2) Billet lubricants and coatings and hydrostatic fluids
(3) Billet finish
(4) Die design
(5) Stem speed.

The important results from this study were applied in the hydrostatic extrusion of the more difficult to work materials such as superalloys, TZM molybdenum alloys, beryllium, and powder compacts. The main problem in producing sound, good quality extrusions with the relatively ductile materials was lubrication but in the cases of brittle materials, die design was important. Major strides have been made in the development of lubrication systems for the different alloys and a better understanding of their operational effectiveness has been achieved. Novel die designs have enabled the achievement of sound extrusions with TZM and beryllium. As a result of this work new frontiers in the potential cold working of brittle materials have been opened.

Empirical equations which permit estimations of the pressure requirements for a given extrusion ratio range for the wide variety of materials evaluated in this program are listed in Table V.

TABLE V. EMPIRICAL EQUATIONS RELATING PRESSURE AND EXTRUSION RATIO FOR COLD HYDROSTATIC EXTRUSION OF SEVERAL MATERIALS

Material	Extrusion Ratio Range	Fluid Extrusion Pressure P, 1000 psi
1100-0 Al(a)	20-200:1	$P = 23 \ln A/a$(b)
Dispersion-hardened sintered-aluminum product (SAP)	10-20:1	$P = 36 \ln A/a + 8$
7075-0 Al	2.5-20:1	$P = 44.6 \ln A/a$
	20:1-60:1	$P = 28.5 \ln A/a + 32$
AISI 4340	2.5-6:1	$P = 130 \ln A/a$
Ti-6Al-4V	2.5-4:1	$P = 160 \ln A/a + 7$
TZM (stress relieved)	2.5-5:1	$P = 116 \ln A/a + 23$
TZM (recrystallized)	4:1 only	$P = 116 \ln A/a + 4$
Be	2.5-4:1	$P = 116 \ln A/a + 23$
Alloy 718	3.3:1 only	$P = 182 \ln A/a$
A286	3.3-5:1	$P = 125 \ln A/a + 17$

(a) Data for 1100-0 aluminum obtained in previous program(1).

(b) A = billet cross-sectional area, a = extrusion cross-sectional area.

The extrusion pressure in hydrostatic extrusion is made up of three components.

(1) The work done in uniform plastic deformation per unit volume of material. This is the work of homogeneous deformation such as that which could be achieved if the billet were pulled in tension without necking to produce the same change in length. This is given by:

$$P = \overline{Y} \ln A/a \quad , \tag{1}$$

where

$\overline{Y}$ = mean yield stress in a true stress/true strain curve. It is seen that this equation is of the same basic form as the empirical equations obtained for extrusion pressure, P, in Table V.

(2) Redundant work

In assuming that the billet is reduced by homogeneous deformation, no account was taken of the internal shearing of individual elements. An element near the surface of the billet moves axially towards the die but on entering the die it is moved inward with a radial velocity component. On exit from the die, the element is sheared back to again move axially. Both shearing processes require energy which does not contribute to the external form of the product and is therefore called "redundant work". A large die angle requires a greater amount of shearing energy than does a long small die angle. However the small angle would give more frictional drag at the billet-die interface.

(3) The work done to overcome billet-die friction

In hydrostatic extrusion, the work in overcoming billet-die friction is considered to be a small component of the overall pressure requirements because of the good lubrication conditions which are obtained.

Pugh indicated that the billet hardness (before extrusion) gave a rough indication of extrusion pressure requirements(2). Figure 12 shows his mean line relating billet hardness with fluid breakthrough extrusion pressure per unit ln A/a and two lines bounding the scatter in his results. The points plotted in Figure 12 are Battelle data for fluid runout pressures given in this report and for 1100-0 aluminum in an earlier report(1). The fact that the Battelle data are for runout rather than breakthrough pressures accounts for the fact the points generally fall below the mean line for Pugh's data. The use of runout rather than breakthrough pressures is believed to be a more accurate basis for comparison and pressure prediction purposes because breakthrough pressures are very sensitive to variations in lubrication whereas runout pressures are almost insensitive for many lubricants as will be shown later in the report. It is of interest that the runout pressure data, as with Pugh's data, also tend to fall very roughly on a straight line, so that a rough estimate of extrusion runout pressures can be made from the billet hardness prior to extrusion.

If data are available on the true stress-true strain properties of the material, then of course a value of $\overline{Y}$, the mean yield stress at the equivalent strain can be determined and substituted in Equation (1). In most instances, the true stress-true strain curve for a material can be represented by the equation:

$$Y = A \ln \epsilon + B \quad , \tag{2}$$

where Y is the uniaxial yield stress at the one strain of ϵ and A and B are constants. Equation (2) can now be integrated to develop an expression for the mean yield stress between a strain of O and ϵ, as follows

$$\overline{Y} = \frac{\int_o^{\epsilon} Y \, d\epsilon}{\epsilon} = A \ln \epsilon + B - A = Y - A \quad . \tag{3}$$

The constants A and B were determined from tensile data in the published literature for four of the materials listed in Table V. The following data are given and used to predict $\overline{Y}$ and thence extrusion pressures at given ratios.

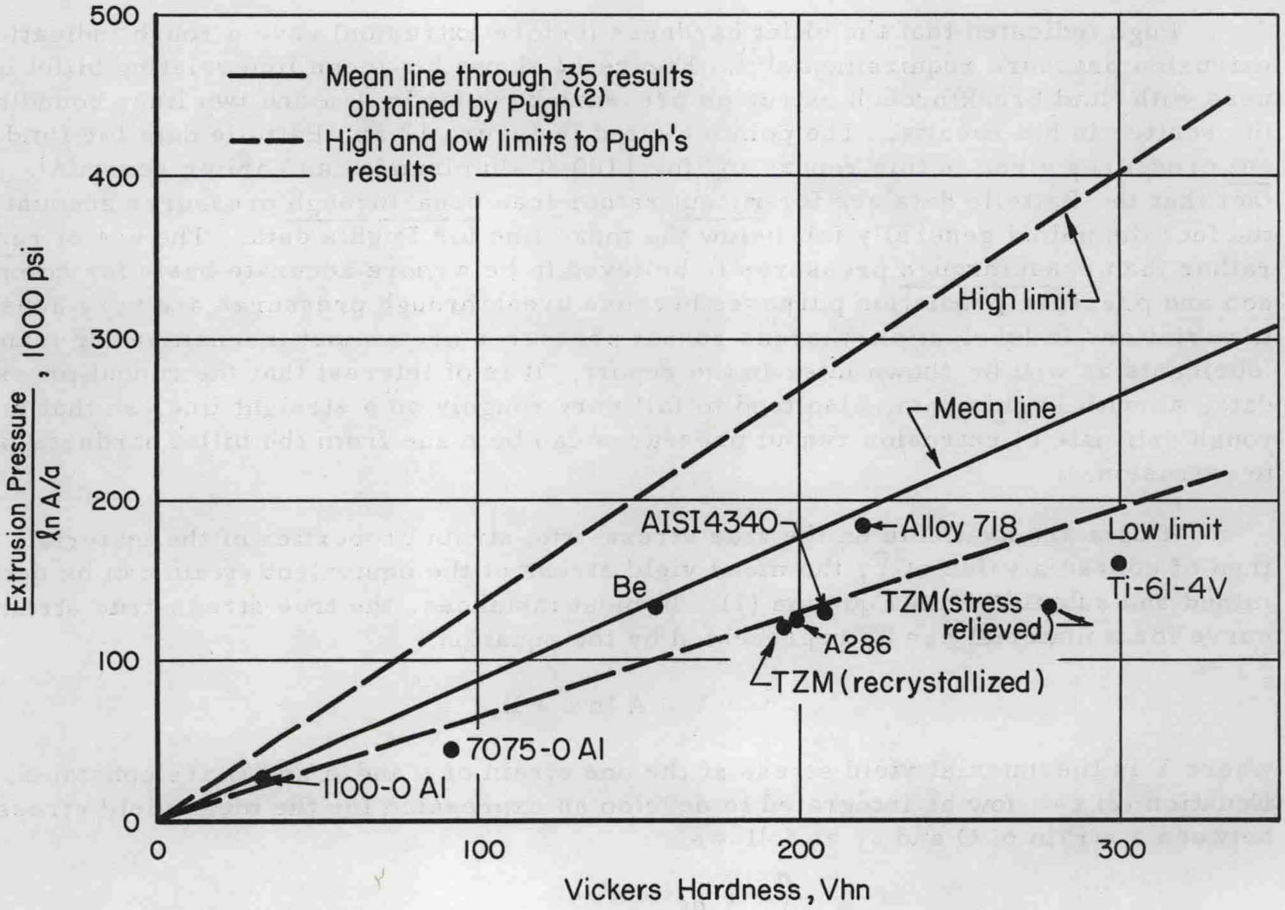


FIGURE 12. RELATIONSHIP BETWEEN EXTRUSION PRESSURE, EXTRUSION RATIO, AND BILLET HARDNESS

Material	Constants x 10^{-3} A	B	Reference	Extrusion Ratio	Mean Yield Stress At True Strain, $\bar{Y}$, 1000 psi	Extrusion Pressure, 1000 psi Actual	Predicted
1100-0 Al	1.16	14.1	7	2:1	13.7	14.6	9.5
				20:1	16.25	69.2	48.5
				200:1	19.0	122	101
AISI 4340	72.2	104	8	2.5:1	97.8	119	89.6
				6:1	161.8	233	290
Ti-6Al-4V	66.7	143	9	2.5:1	137.5	154	126
				4:1	168.8	229	234
A286	131.4	38.6	10	5:1	118.2	217	189

It is seen that the predicted extrusion pressures are of the same order as the measured extrusion pressures but that some rather large discrepancy exists. The predicted pressures are generally lower than the measured. The inaccuracies in predictions may be attributed to:

(1) Errors in extrapolation of the true stress-true strain curves. Except for 1100-0 aluminum most of these curves were obtained at true strains below unity, yet most of the extrusion ratios evaluated in Table VI represented strains beyond this level. To obtain true stress values at high levels of strain, a plane strain compression test would be necessary.

(2) Possible variations in strengths and work-hardening characteristics among materials of the same grade.

(3) Possible influence of effective temperature during extrusion on strengths of workpiece.

(4) Effect of neglecting die friction and non-uniform deformation.

In the remaining experimental work reported in Section 1 an investigation was conducted into hydrostatic compaction of Ti-6Al-4V alloy powder. At a pressure level of 225,000 psi, compacts having a 98 percent theoretical density were obtained.

This section also gives details of the effect of process variables on the mechanical properties of hydrostatic extrusions. The heavy cold work of hydrostatic extrusion gave high strength levels combined with good ductility. In some cases, the strength levels obtained were higher than could be achieved by other working processes.

VII

COLD HYDROSTATIC EXTRUSION OF 7075-0 ALUMINUM ROUNDS

The main process variables studied in the cold extrusion of 7075-0 aluminum were extrusion ratio, stem speed, and lubrication. Many trials were conducted at a ratio of 20:1 and a stem speed of 20 ipm with the aim of determining the best lubrication system for use at higher ratios and stem speeds, and for application to the extrusion of tubing and T-sections. These data are given in Table VI. Additional data obtained at higher ratios and/or stem speeds are presented in Table VII.

The development of a good lubrication system with 7075-0 aluminum was of primary concern because of the tendency of this alloy to stick-slip during extrusion. Stick-slip originated from momentary breakdown of the billet lubricant, most often at the point of breakthrough. The problem was overcome by the formulation of satisfactory billet lubricants and by billet nose design. Consequently, excellent surface finishes were obtained on extrusions made at ratios up to 60:1 and extrusion exit speeds as high as 250 fpm. In the earlier experiments with this alloy, surface cracking of the extrusion had resulted from bad lubrication.

7075-0 aluminum is known for its tendency to crack during conventional hot extrusion. To prevent cracking in this operation, the exit extrusion speeds are kept very low, in the order of 2-3 fpm. The exit speeds of 250 fpm obtained in hydrostatic extrusion offer significant potential advantages in a production operation.

The tensile and yield strengths of products extruded from annealed 7075-0 aluminum were doubled and tripled, respectively, without any sacrifice in ductility.

Extrusion Ratio

High-quality extrusions of 7075-0 aluminum were obtained over a range of extrusion ratios from 2.5 to 60. Figure 13 shows the relationship between fluid runout pressure and extrusion ratio. The upper curve in the figure depicts the data obtained with Lubricant L17. At ratios of 20:1 and above, stick-slip occurred with this billet lubricant. Later data obtained with Lubricant L53 at these ratios under good lubrication conditions are shown in the lower curve. Fluid-runout pressures were used in Figure 13 because they provide an accurate basis from which projections of pressure requirements can be made. The alternative measure, breakthrough pressure, is not so accurate because its level is sensitive to extrusion conditions such as stem speed and lubrication conditions and can vary from trial to trial under apparently identical conditions. The decrease in slope in the top curve at ratios above 20:1 may be associated with a decrease in the billet flow stress caused by adiabatic heating during deformation.

Extrapolation of the pressure requirements at the higher ratios indicate that ratios of 1000:1 are possible within the 250,000 psi pressure capacity of the hydrostatic extrusion container. The lubrication systems developed to date may not be capable of extension to such high ratios. In a single trial at 200:1, the lubricant (L53) broke down. However, other factors such as excessive adiabatic heating may be the limiting factor at these high ratios.

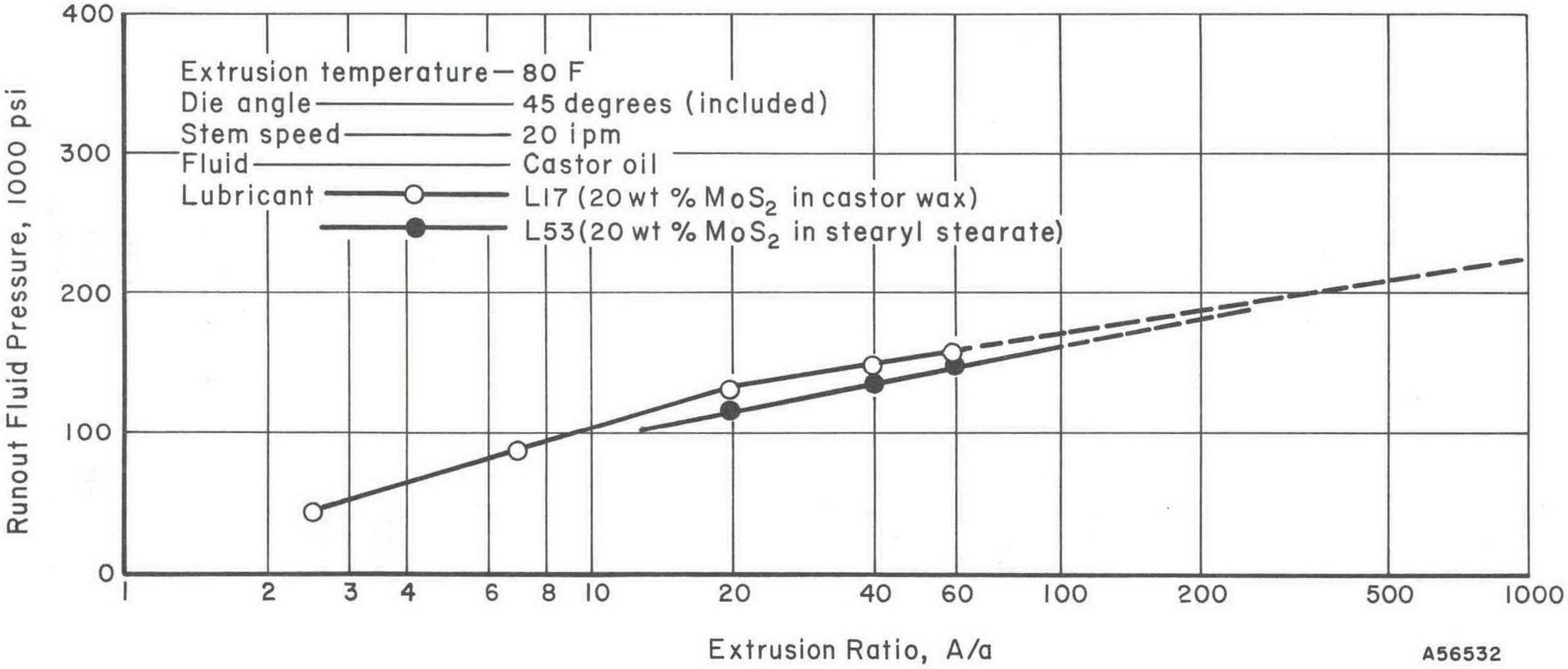


FIGURE 13. EFFECT OF EXTRUSION RATIO ON FLUID RUNOUT PRESSURES IN THE HYDROSTATIC EXTRUSION OF 7075-O ALUMINUM ROUNDS

Reliability of Data

Much of the discussion of the relative effects of the process variables is centered on comparisons of the breakthrough pressures, P_b and runout pressures, P_r. To determine the significance of pressure differences, the coefficients of variation in P_b and P_r were determined for five trials (463, 464, 453, 454, and 472) where good lubrication was obtained under nominally identical extrusion conditions. These pressure data are given in Table VI. The standard deviations were approximately 1,480 psi and 1,340 psi for P_b and P_r, respectively. The coefficients of variation were approximately 1.1 percent in each case. Consequently, it is suggested that the conclusions based on one trial are probably reliable.

Billet Finish

A billet finish of 60-120 microinches obtained by machining was used as the basis for evaluating lubrication, extrusion ratio and stem speed. In the evaluation of billet finish, however, a range of 35-500 microinch obtained by machining or grit blasting was used in conjunction with two billet lubricants. Table VI gives the data obtained in this study.

Billet finishes in the order of 35 to 50 microinches, rms, resulted in the highest breakthrough pressure peak regardless of whether Lubricant L11 (Trial 251) or L17 (Trial 308) were used. Increasing the roughness to the 300-500 microinch range lowered these pressure peaks but generally did not succeed in preventing stick-slip.

In one trial (Trial 249) a low and gradual breakthrough pressure was followed by a smooth runout. This low breakthrough pressure, which was the main reason for the elimination of stick-slip, is attributed to the geometry of the rough machined surface on the billet. The billet-surface roughness peaks were sharper and a greater capacity for trapping lubricant in the valleys, than for the remaining trials, thus enhancing the squeeze lubrication potential. Other factors such as the method of lubricant application to the billet may have played an important part in the elimination of momentary seizure.

The grit-blast finishes lowered the breakthrough stick-slip peaks but did not succeed in preventing them. The matte finish produced by grit blasting smoothed out well on the extruded surface. The machined finish on billets, however, results in a helical grooved pattern on the extrusion which becomes more pronounced with increasing surface roughness and extrusion ratio. For billets machined to a rough finish (300-500 microinches), the helical grooves became sites for initiating surface cracks.

Lubrication Systems

Table VI gives data for the evaluation of 14 billet lubricants and 4 fluids (Trial 347 and below) at a ratio of 20:1 and stem speed of 20 ipm. Several good billet lubricants were developed and data obtained using these lubricants at higher ratios are given in Table VII.

TABLE VI. EXPERIMENTAL DATA FOR 80 F HYDROSTATIC EXTRUSION OF 7075-O ALUMINUM ROUNDS AT A RATIO OF 20:1 AND STEM SPEED OF 20 IPM

Die angle - 45 degrees
Exit speed - 61.5 ft/min

Fluid - Castor oil
Billet diameter - 1.75 inches

Objective or Variable	Trial	Billet Surface Finish(a), microinches, rms	Billet Lubricant (Details in Table 3)	Extrusion Pressure, 1000 psi				Type of Curve (Fig. 26)	Length of Extrusion, inches	Comments
				Breakthrough		Runout				
				Stem	Fluid	Stem	Fluid			
Billet finish	251	50	L11	195	175(h)	128	120	D2	61	
	250	110-130	L11	180	160(h)	125	117	D2	30	
	249	270	L11	149	135	142	125	B2	87	
	297	300	L11	172	157(h)	140	115	D2	49	
	298	350	L11	164	153(h)	141	118	D2	40	
	299	400	L11	168	153(h)	137	118	D2	44	
	256	Grit	L11	192	173(h)	--	--	D1	22	
	273	Grit	L11	178	180(h)	136	135	D1	26	
	283	Grit	L11	179	167(h)	147	126	D1	30	
Billet finish	255(b)	300	L17	162	159	--	--	--	0	P_b not reached
	271(b)	350	L17	239	225	--	--	--	0	Ditto
	272(b)	400	L17	274	248	--	--	--	0	"
Billet finish	308	35-50	L17	199	189	--	--	D2	17	
	309	100-250	L17	167	156	139	118	D2	50	
	329	350	L17	179	162(h)	143	116	D1	41	
	330	500	L17	154	153(h)	147	120	D1	51	
	281	Grit	L17	(c)	150	(c)	121	D2	43	
	282	Grit	L17	(c)	153	(c)	117	D2	46	
Billet lubricant and fluid	347	60-120	L17	162	152	144	130	C1	65	
	380	60-120	L8	180	172	--	--	--	--	P_b not reached
	343(d)	60-120	L22	234	215	--	--	--	--	Ditto
	344(d)	60-120	L46	195	186	--	--	--	--	"
	346	60-120	L46	168	144	--	--	--	--	Stopped prematurely due to false instrument reading
	345	60-120	L47	165	158	141	130	D1	44	
	365(e)	60-120	L47	165	154	143	136	C3	62	
	356	60-120	L51	202	187	--	--	--	--	P_b not reached
	424	60-120	L52	219	191	160	137	D1	75	
Billet lubricant, fluid, die material, and billet nose design	447	60-120	L31	168	148	136	125	C2	65	
	449(f)	60-120	L31	176	153	141	128	C2	65	
	440	60-120	L33	169	150	159	138	B3	40	
	436	60-120	L38	156	140	140	122	B2	39	
	444	60-120	L48 + L17	156	150	135	127	D1	80	
	431	60-120	L52	188	169	142	119	D2	74	
	432	60-120	L53	164	150	144	123	C2	79	Insufficient lubricant on nose
	433	60-120	L54	169	154	143	127	C2	57	Ditto
	463	60-120	L53	152	137	138	123	B2	66	Stepped billet nose
	464(g)	60-120	L53	149	135	133	119	B2	68	
	453	60-120	L53	155	138	142	122	B2	108	
	454(f)	60-120	L53	150	134	136	122	B2	108	
	472	60-120	L53	156	135	138	121	B2	65	

(a) The finish quoted was obtained by the turning operation except for the grit finish, which was obtained by vapor blasting.

(b) Trials 255, 271, and 272 were attempted with the 7075 billets in the T6 condition.

(c) Stem load-cell recorder did not function.

(d) Fluid was water.

(e) Fluid was polyethylene glycol.

(f) Flame coated die used.

(g) Fluid was silicate ester (SE).

(h) A small breakthrough pressure peak occurred at about 135,000 psi followed by seizure and stick-slip. For comparison purposes, the first large stick-slip peak is taken as the breakthrough pressure.

TABLE VII. ADDITIONAL EXPERIMENTAL DATA FOR 80 F HYDROSTATIC EXTRUSION OF 7075-O ROUNDS

Die angle - 45 degrees   Billet diameter - 1.75 inches   Fluid - Castor oil

t Surface nish, nches, rms	Extrusion Ratio	Stem Speed, ipm	Exit Speed, fpm	Billet Lubricant (Details in Table 3)	Extrusion Pressure, 1000 psi				Type of Curve (Fig. 26)	Length of Extrusion, inches	Comments
					Breakthrough		Runout				
					Stem	Fluid	Stem	Fluid			
5-65	40	20	123.0	L17	197	183	157	140	D1	90	
rit	40	20	123.0	L17	195	186	161	145	D1	80	
-120	40	20	123.0	L53	254	221	165	142	D2	58	
-120	40	20	123.0	L53	204	164	144	135	D3	137	
-120	40	20	123.0	L38	204	179	146	135	D1	119	
-120	40	20	123.0	L52	177	157	149	143	B2	120	Compound angle billet nose, A = 0
-120	40	20	123.0	L53	168	150	154	137	B2	140	Compound angle billet nose, A = 0
-120	60	6	55.4	L17	239	216	173	156	D1	60	
-120	60	20	184.5	L17	217	204	171	153	D1	81	
-120	60	20	184.5	L17	222	204	147	147	D1	90	
-120	60	20	184.5	L52	192	178	175	153	B2	200	Compound angle billet nose, A = 0
-120	200	6	282.5	L53	210	189	--	--	C4	49	Compound angle billet nose, A = 0
0-120	20	80	246.0	L17	167	155	139	129	B1	79	
rit	20	80	246.0	L17	167	154	141	130	B2	80	
on nose 0 on rest	20	80	246.0	L17	160	150	144	130	B1	64	
-120	20	80	246.0	L53	170	148	144	126	B2	88	
-120	20	80	246.0	L54	168	150	144	126	B2	50	
-120	20	80	246.0	L31	174	158	144	133	B2	44	
-120	25	20	7.7	L17	48	46	48	46	A1	8	
-120	7	20	21.4	L17	99	94	96	89	A2	18	

silicate ester.

neter was 1.414 inches.

Significant strides were made toward reducing the level of the breakthrough pressure peak and minimizing the runout pressure level. This can be seen quite clearly in Figure 14 which compares the pressure-displacement curves obtained with earlier lubrication system, L17 (20 wt percent MoS_2 in castor wax) to the best system developed in this program, L53 (20 wt percent MoS_2 in stearyl stearate). In addition, a further improvement is shown for L53 by replacing the fluid castor oil with silicate ester. A billet lubricant which was as equally effective in reducing pressures as L53, was L38 (PTFE) but the finish obtained was not as good. Other billet lubricants which showed marginal improvements over L17 were L31 (fluorocarbon telomer) and L33 (55 wt percent MoS_2 and 6 wt percent graphite in sodium silicate). Of particular significance was the excellent surface finish obtained with L53. While finishes were not measured, comparisons on a sensory basis showed L53 to give one of the best surfaces. The surface finishes obtained with the other lubricants mentioned above, however, were still quite good.

Several trials were conducted with the stearyl-stearate based billet lubricants. Three of these trials (453, 454 and 472) were conducted to evaluate tandem billet design and are presented here for comparison purposes. Details of the tandem billet design are given later. As discussed earlier, the repeatability of these results was good, only small variations in breakthrough pressure being observed. The initial stick-slip encountered with L53 in Trial 432 was due to improper lubrication on the billet nose. The remaining trials with this lubricant demonstrated the importance of careful billet lubrication.

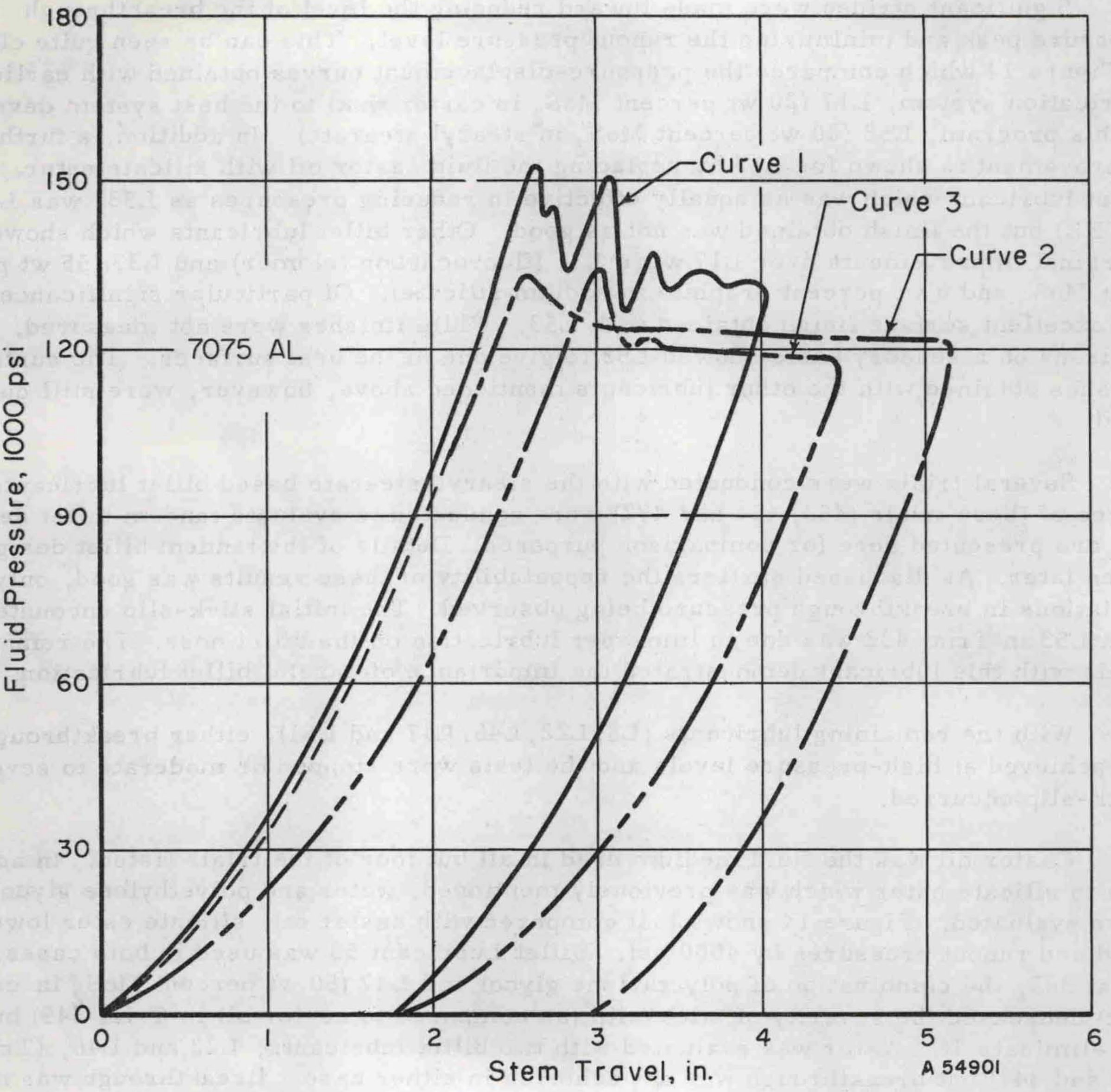
With the remaining lubricants (L8, L22, L46, L47 and L51), either breakthrough was not achieved at high-pressure levels and the tests were stopped or moderate to severe stick-slip occurred.

Castor oil was the fluid medium used in all but four of the trials listed. In addition to silicate ester which was previously mentioned, water and polyethylene glycol were evaluated. Figure 14 shows that compared with castor oil, silicate ester lowered fluid and runout pressures by 4000 psi. Billet Lubricant 53 was used in both cases. In Trial 365, the combination of polyethylene glycol and L47 (50 wt percent MoS_2 in carbowax) decreased the severity of stick-slip (as compared to castor oil in Trial 345) but did not eliminate it. Water was evaluated with two billet lubricants, L22 and L46, (Trials 343 and 344) but breakthrough was not achieved in either case. Breakthrough was not however achieved when castor oil was used with L46 (Trial 346).

At an extrusion ratio of 40:1 and a stem speed of 20 ipm, severe stick-slip always occurred when the standard billet nose configuration was used. Lubricants L53 and L38 which proved to be efficient lubricants at a ratio of 20:1 failed to eliminate stick-slip though L38 caused some lowering of pressure levels. In spite of the stick-slip, however, the extruded surface finish obtained with these lubricants was very good. Again, silicate ester (Trial 468) resulted in marginal reductions in pressure obtained with castor oil (Trial 435), but stick-slip was not eliminated.

Billet Nose Design

In an attempt to reduce the high breakthrough pressure peaks, billet nose design was changed. In Trial 463 conducted at a ratio of 20:1, a stepped billet nose consisting of a 1.25-inch diameter step about 1/4 inch long located at the juncture between the



Extrusion Conditions: Extrusion ratio 20:1
Stem speed 20 ipm
Die angle 45 degrees included

Curve	Trial	Fluid	Billet Lubricant
1	347	Castor oil	20 wt. % MoS_2 in castor wax (L17)
2	454	Castor oil	20 wt. % MoS_2 in stearyl stearate (L53)
3	464	Silicate ester	20 wt. % MoS_2 in stearyl stearate (L53)

FIGURE 14. EFFECT OF FLUID AND BILLET LUBRICANT ON PRESSURE-DISPLACEMENT CURVES OBTAINED IN THE HYDROSTATIC EXTRUSION OF 7075-0 ALUMINUM AT A RATIO OF 20:1

conical nose and cylindrical portion was used. The stepped nose did not reduce the P_b peak but it effected a multistepped, more gradual transition from the maximum pressure to the runout and pressure.

These results led to the design of the compound angle billet nose shown in Figure 15. The compound-angle design was initially evaluated at the higher ratio of 40:1 where stick-slip was more of a problem. In Figure 16 the extrusion pressure-ram travel characteristics obtained with the compound-angle design (Trial 470) is compared with those for standard nose design. Clearly the compound-angle nose design not only reduced the P_b peak by about 70,000 psi but severe stick-slip was completely eliminated. These results were obtained with Lubricant L53 which was effective with the standard nose at a ratio of 20:1. Lubricant L52, which was not so good at 20:1, was also evaluated with the compound-angle nose at 40:1 and 60:1 (Trials 473 and 474 in Table VII.) At both ratios, low P_b peaks were obtained followed by smooth runouts resulting in products having an excellent finish.

The success of the compound angle nose in extending the range in extrusion ratio, for which a given lubrication system is capable, can be explained as follows:

(1) The second or upper angle on the compound-angle nose provides for more efficient "thick-film" lubrication at breakthrough, thereby, reducing the coefficient of static friction, μ, and thus, the P_b peak. This assistance in lubrication is clearly promoted by the presence of the pressurized fluid at the billet-die interface at the critical point of breakthrough.

(2) Elimination of a high P_b prevents the initiation of stick-slip during runout. This is partly because lubrication breakdown may occur during the arrest period of a stick-slip cycle due to excessive extrusion exit speeds that can occur during slip.

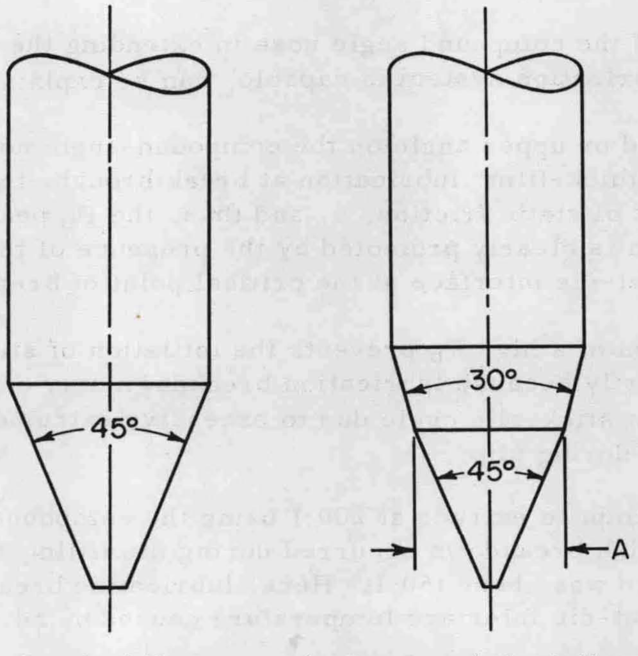
In a single attempt to extrude at 200:1 using the compound angle nose design (Trial 504), lubrication breakdown occurred during die filling at the point where the extrusion ratio achieved was about 150:1. Here, lubrication breakthrough was probably due to excessive billet-die interface temperature caused by adiabatic heating.

It is of interest to note that the compound-angle nose was evaluated in the previous program(1) on 1100-0 aluminum at a ratio of 10:1. No pressure reduction was obtained, however, because the lubrication system used was entirely adequate for the less severe extrusion conditions.

Stem Speed

Several trials were conducted at a stem speed of 80 ipm and a ratio of 20:1. Details are given in Table VII. Under these conditions the extrusion leaves the die at 250 ft/min which was the fastest exit speed accomplished in the program.

The pressure-displacement curve for each trial was characterized by a sharp breakthrough pressure peak indicating a stick-slip situation only at breakthrough followed by a smooth runout. Stick-slip during runout was prevented because the high stem speeds did not allow the billet to stop at the end of fluid decompression during slip after breakthrough, thus maintaining kinetic friction conditions. The breakthrough and runout pressure levels were approximately 150,000 psi and 130,000 psi respectively for each



a. Standard Nose b. Compound - Angle Nose

FIGURE 15. TWO BILLET NOSE DESIGNS EVALUATED IN HYDROSTATIC EXTRUSION

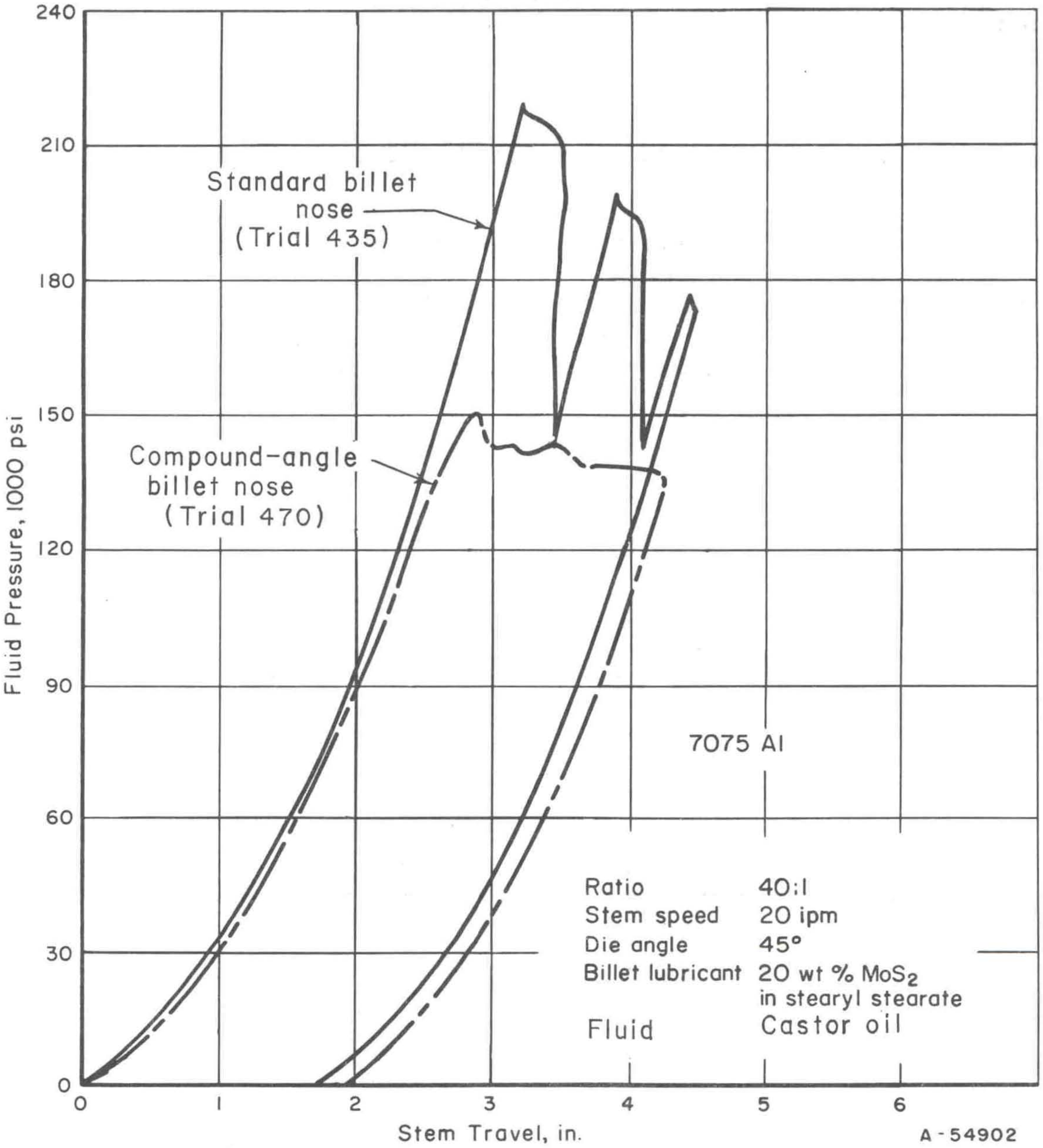


FIGURE 16. EFFECT OF BILLET NOSE SHAPE ON PRESSURE-DISPLACEMENT CURVES OBTAINED IN THE HYDROSTATIC EXTRUSION OF 7075-0 ALUMINUM AT A RATIO OF 40:1

billet lubricant. Of the four lubricants evaluated, Lubricant L53 again provided the lowest pressures. The various billet surface finishes evaluated at this stem speed did not appear to effect the pressure levels appreciably.

In every case, the surface finish on the runout part of the extrusion was excellent. However, shallow cracks formed at the leading end of the extrusions these were probably due to the high exit speed during the slip portion of stick-slip where adiabatic heating may have been excessive.

Tensile Properties of 7075-0 Aluminum Hydrostatic Extrusions

Tensile data for 7075-0 aluminum extrusions produced at extrusion ratios of 20:1, 40:1, and 60:1 are listed in Table VIII. The tensile and yield strengths of the material in the annealed condition were almost doubled and tripled, respectively, by extrusion at ratios up to 60:1 without any appreciable sacrifice in ductility. Exit speed does not appear to have influenced mechanical properties at an extrusion ratio of 20:1.

TABLE VIII. ROOM-TEMPERATURE TENSILE PROPERTIES OF 7075 ALUMINUM ROUNDS PRODUCED BY HYDROSTATIC EXTRUSION

Extrusion, Ratio	Reduction in Area of Extrusion, percent	Trial	Speed, ipm Stem	Speed, ipm Exit	Ultimate Tensile Strength, 1000 psi	Yield Strength (0.2 percent Offset), 1000 psi	Reduction in Area, percent	Elongation in 2 Inches, percent
20	95.0	311	20	740	56.3	40.9	20.8	21.0
40	97.5	318	20	1480	60.2	41.4	39.5	26.0
60	98.3	324	20	2220	61.3	35.0	38.7	24.0
20	95.0	310	80	2960	55.2	40.6	22.9	21.0
1	0	As-annealed bar stock			33.8	15.5	45.2	23.3

VIII

HYDROSTATIC EXTRUSION OF AISI 4340 STEEL ROUNDS

A good foundation was laid in an earlier research program(1) for the study of the critical process variables for AISI 4340 steel.

AISI 4340 proved to be the least difficult of the materials in this program to lubricate and, consequently, extrusions of excellent quality were obtained at ratios of up to 6:1. Extrusion pressure requirements for ratios above 6:1 were beyond the capacity of the tooling. Lubrication systems were thoroughly evaluated at both room temperature and temperatures up to 500 F in terms of:

(1) hydrostatic fluids

(2) billet coatings

(3) billet lubricants

(4) billet surface finish (in a few cases).

Table IX gives data obtained in the evaluation of lubrication systems at room temperature and under constant extrusion conditions.

Data obtained in the investigation of the effect of other process variables such as extrusion ratio, stem speed, and die angle on extrusion pressures are given in Table X. For reasons of clarity, some duplication of data occurs in Tables IX and X.

Several lubrication systems were evaluated at three elevated temperature levels. Details are given in Table XI. Here also, AISI 4340 steel was satisfactorily lubricated by most of the systems evaluated. The choice of a lubrication system in a production operation would appear to depend on the relative costs of these lubricants and their ease of application.

Extrusion Ratio

The range in extrusion ratio covered in the hydrostatic extrusion of AISI 4340 steel in this program was from 3.3:1 to 6.0:1. Figure 17 shows the pressure requirements within this extrusion ratio range for both room temperature (80 F) and 400 F. Extrapolation of each line enables an estimate to be made of the extrusion ratios possible with containers having various pressure capabilities. A container design with a pressure capacity of 450,000 psi is currently being considered at Battelle. With such a container, the limiting reduction ratios would be about 35:1 at 80 F and 50:1 at 400 F. In view of the advancements made in lubrication for AISI 4340 in this program, those predicted ratios are a realistic possibility and would represent sizable reductions not hitherto possible with steel at these low temperatures.

The runout pressure levels shown in Figure 17 are only slightly lower than those obtained in the previous program(1).

IX. INVESTIGATION OF LUBRICATION SYSTEMS UNDER CONSTANT EXTRUSION CONDITIONS FOR 80 F HYDROSTATIC EXTRUSION OF AISI 4340 R

Die angle - 45 degrees (included)
Billet diameter - 1-3/4 inches

Stem speed - 20 ipm
Billet surface finish - 60 to 120 microinches

					Extrusion Pressure, 1000 psi						
			Billet Lubrication(a)		Breakthrough		Runout		Type of Curve	Length of Extrusion,	
	Trial	Fluid	Coating	Lubricant	Stem	Fluid	Stem	Fluid	(Fig. 26)	inches	Com
					Extrusion Ratio 5:1						
	215	Castor oil	C1	L11	249	212	248	212	A1	17	
	216	Ditto	C1	L11	253	214	251	214	A2	17	
	230	"	None	L11	248	209	248	235	B4	13	Die scored
	209	"	C1	L17	256	217	253	212	B1	11	
	210	"	C1	L17	254	215	252	212	B1	16	
	211	"	C1	L17	253	214	252	212	B1	18	
	212	"	C1	L17	256	216	254	213	B1	17	
	217	"	None	L17	254	218	252	215	B1	17	
	218	"	None	L17	255	216	254	215	B1	17	
	219	"	C1	None	272	228	259	216	B1	16	
	220	"	C1	"	272	230	261	215	C1	17	
	221	"	None	"	265	225	256	229	D3	14	
ant	222	Castor oil	C1	L18	245	213	244	213	A1	17	
ıg,	223	Ditto	C1	L18	250	214	250	214	A1	17	
	231	"	C1	L19	254	215	248	209	B1	17	
	232	"	C1	L19	252	212	246	207	B1	18	
	233	"	C1	L20	245	208	245	209	A1	18	
	234	"	C1	L20	245	206	245	207	A1	17	
	235	"	C1	L21	262	218	249	208	B1	17	
	236	"	C1	L21	262	219	250	208	B1	19	
ınt	277	Castor oil	None	L17	240	223	240	216	B1	13	
	257	Ditto	"	L17	255	218	251	215	B1	17	
l	258	"	"	L17	296	238	--	--	--	--	P_b not achi
	315(b)	"	"	L17	240	221	241	217	B1	15	
	429	"	"	L38	267	230	264	218	B1	13	
	430	"	"	L31	266	230	262	217	B1	10	
	462	"	"	L53	260	225	255	221	B3	11	

Coating	225	Castor oil	C3	None	--	255	--	--	--	0	P_b not achieved
	224	Ditto	C4	"	261	223	258	229	B3	16	
	237	"	C4	L11	244	204	243	213	A4	16	
Fluid with coating, C1	238	Ethylene glycol	C1	L17	252	212	250	210	A1	12	
	240	Ditto	C1	L17	248	210	246	209	A1	16	
	241	"	C1	L17	247	211	246	208	B1	16	
	242	Polyethylene glycol	C1	L17	250	212	248	210	B3	18	
	243	Ditto	C1	L17	243	208	242	208	A1	17	
	252	"	C1	L19	240	207	240	211	A4	15	
	253	"	C1	L20	240	207	240	212	A4	15	
	254	"	C1	L21	248	211	248	213	A3	16	
Fluid without coating, C1	274	Polyethylene glycol	None	L22	249	229	249	226	A1	15	Stem seal broke at P_b
	276	Ditto	"	L22	278	224	248	213	B1	1	
	275	"	"	L23	247	230	245	228	B1	13	
	465	Silicate ester	"	L17	255	219	255	219	A1	14	
					Extrusion Ratio 4:1						
Fluids and lubricants	289	Castor oil	None	L17	208	186	208	186	A1	11	
	306(b)	Castor oil	"	L17	204	192	200	190	A1	13	
	303	Polyethylene glycol	"	L22	205	184	208	188	A1	14	
	293	Polyethylene glycol	"	L23	204	189	201	185	B3	15	
	294	Water	"	L17	192	178	205	188	A3	9	
	295	"	"	L17	204	189	212	189	A1	13	
	301	"	"	L17	206	186	212	186	A1	15	
	302(b)	"	"	L17	202	191	204	189	A1	13	

(a) Billet lubricants listed in Table III, coatings in Table IV.

(b) Billet surface finish was obtained by grit blasting followed by vapor blasting.

TABLE X. INVESTIGATION OF EXTRUSION RATIO, STEM SPEED, AND DIE ANGLE FOR 80 F HYDROSTATIC EXTRUSION OF AISI 4340 ROUNDS

Billet diameter - 1-3/4 inches
Fluid - Castor oil
Billet surface finish - 60 to 120 microinches
Billet lubricant - L17 (20 wt% MoS_2 in Castor Wax)

Trial	Extrusion Ratio	Stem Speed, ipm	Die Angle (Included), degrees	Billet(b) Coating	Extrusion Pressure, 1000 psi				Type of Curve (Fig. 26)	Length of Extrusion, inches	Comment
					Breakthrough		Runout				
					Stem	Fluid	Stem	Fluid			
285	3.3	80	45	None	262	243	--	--	D2	8	
287	3.3	80	45	"	177	167	180	167	A3	12	
312	3.3	80	45	"	171	160	168	157	A1	14	
289	4	20	45	None	208	186	208	186	A1	11	
306(a)	4	20	45	"	204	192	200	120	A1	13	
323(a)	4	80	45	"	206	189	206	188	A1	17	
261	5	20	30	None	273	250	--	--	--	--	P_b not achieved
257	5	20	45	"	255	218	251	215	B1	17	
277	5	20	45	"	240	223	240	216	B1	13	
280	5	20	45	"	248	227	248	220	B1	13	
259	5	20	60	"	256	228	254	223	B1	19	
265	5	20	60	"	259	233	257	231	A1	14	
213	5	20	60	C1	261	219	260	216	B1	19	
214	5	20	60	C1	259	219	258	216	B1	17	
260A	5	20	90	None	296	260	--	--	--	2	P_b not achieved
260B	5	20	90	"	270	240	--	--	--	--	P_b not achieved
262	5	1	45	None	266	244	263	234	D1	6	
288	5	1	45	"	260	229	256.5	217	D2	11	
263	5	6	45	"	255	235	256	229	B1	13	
206	5	6	45	C1	266	224	254	216	B1	15	
207	5	6	45	"	260	218	256	216	B2	15	
208	5	6	45	"	256	218	250	215	B1	16	
328(a)	5	80	45	None	240	220	240	219	A2	4	
339	5	80	45	"	240	219	240	218	A1	5	
340	5	80	45	"	237	217	243	217	A1	14	
246(c)	6	20	45	C1	281	235	280	235	B3	17	
247	6	20	45	None	280	233	278	233	B1	16	
451	6	20	45	"	285	246	--	--	--	--	P_b not achieved
248(d)	6	20	45	C1	279	231	278	231	C1	16	
244	6	20	60	"	284	237	282	234	B1	16	
245	6	20	60	"	284	236	283	233	B1	16	

(a) Billet surface finish was obtained by grit blasting followed by vapor blasting.

(b) C1 = Phosphated coating.

(c) Billet lubricant was L18.

(d) Billet lubricant was L11.

TABLE XI. EXPERIMENTAL DATA FOR HYDROSTATIC EXTRUSION OF AISI 4340 ROUNDS AT ELEVATED TEMPERATURES

Billet diameter - 1-3/4 inches
Die angle - 45 degrees (included)
Billet surface finish - 60 to 120 microinches
Stem speed - 20 ipm

Trial	Extrusion Ratio	Fluid	Billet Lubricant	Type of Stem Seal(c)	Extrusion Pressure, 1000 psi				Type of Curve (Fig. 26)	Length of Extrusion, inches	Comments
					Breakthrough		Runout				
					Stem	Fluid	Stem	Fluid			
					Temperature 140 F						
226	5	Castor oil	L17(a)	1r	251	211	250	207	B1	16	
227	5	Ditto	L17(a)	1r	249	210	248	206	B1	16	
228(b)	5	"	L17(a)	1r	252	211	250	207	B1	16	
229	5	"	None(a)	1r	266	222	254	210	C1	15	
					Temperature 400 F						
413	4	Silicate ester	L31	2t	188	182	189	180	B1	10	
423	4	Ditto	L33	2t	198	173	192	170	B1	12	
414	5	"	L31	2t	223	216	--	--	--	3	Stem-seal leak occurred at breakthrough
422	5	"	L31	2t	223	196	214	193	B1	13	
					Temperature 500 F						
394	4	Polyphenyl ether	L31	1t	198	196	197	194	B1	13	
418	5	Ditto	L31	2t	243	213	233	206	B3	8	
420	5	"	L31	2t	230	200	222	197	B1	15	
393	4	Polyphenyl ether	L33	1t	195	195	--	--	--	1	P_b not reached
397	4	Ditto	L34	1t	187	199	185	195	A2	14	
409	4	"	L35	2t	194	190	190	186	B1	12	
399	4	"	L38	1t	199	204	197	203	B1	5	
401	4	"	L38	1t	193	200	189	195	B2	13	
407	4	"	L40	1t+1r	195	189	186	183	B2	10	
406	4	"	L43	1t+1r	202	198	199	194	B2	10	
408	4	"	L44	1t+1r	199	192	198	191	A4	9	
410	4	Tricresyl phosphate	L31	2t	200	187	200	185	A2	11	
411	4	Triaryl phosphate	L31	2t	202	192	201	191	A2	12	
412	4	Chlorinated biphenyl	L31	2t	196	186	191	181	B1	10	

(a) C1 coating was applied to billet; billet lubricants listed in Table III.
(b) Temperature was 120 F.
(c) 1t = 1 PTFE O-ring on stem; 2t = 2 PTFE O-rings on stem; 1t+1r = 1 PTFE O-ring and 1 rubber ring on stem.

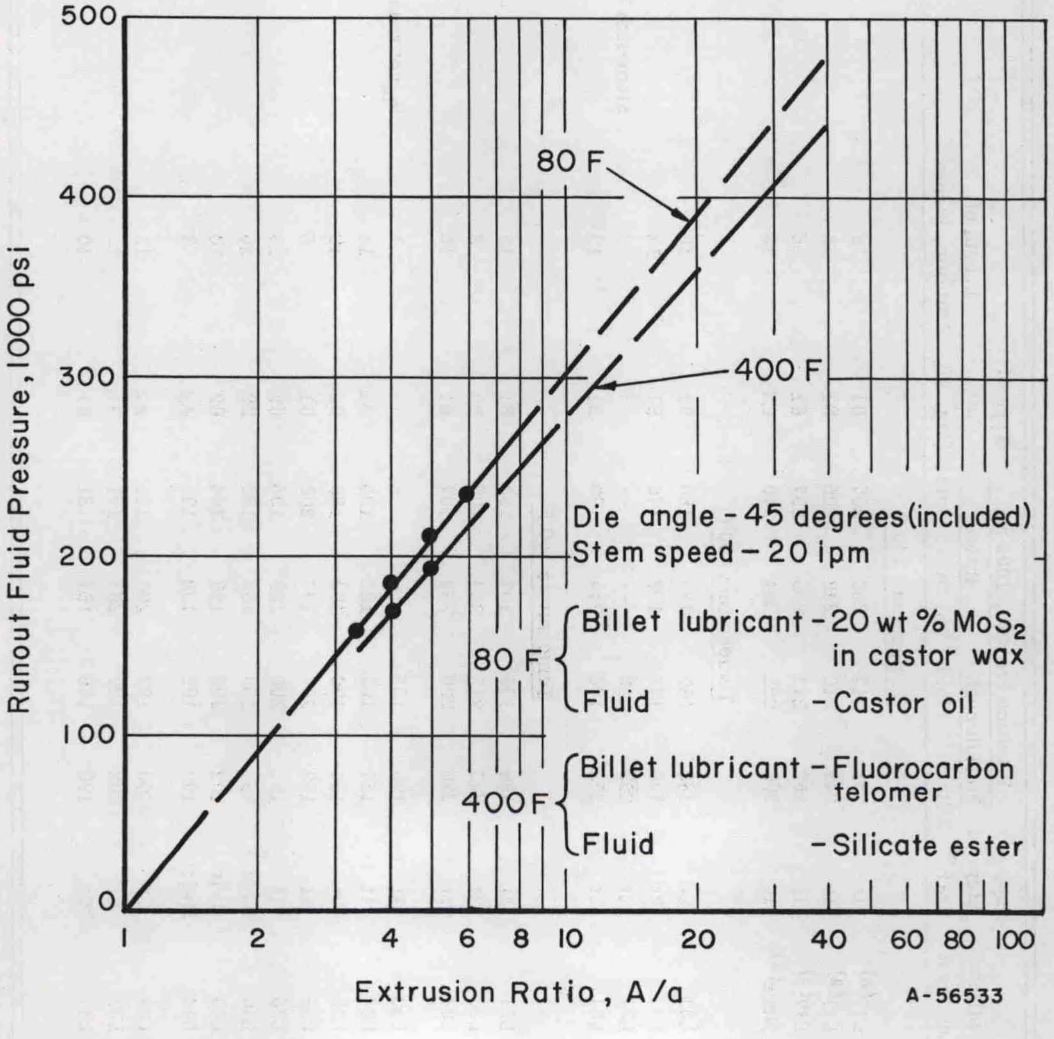


FIGURE 17. EFFECT OF EXTRUSION RATIO ON PRESSURES REQUIRED FOR HYDROSTATIC EXTRUSION OF AISI 4340 STEEL

Fluids and Billet Lubrication at 80 F

Several fluids and billet lubricants were investigated at room temperature with the aims of minimizing breakthrough and runout pressures and obtaining good finishes on the extruded product. In addition, it was hoped that lubricants would be developed which would negate the need for billet conversion coatings. The main billet conversion coating involved in this study was a zinc phosphate coating (Cl) which was used extensively with success in the previous program(1). Zinc phosphate coatings on steel are commonly used in conventional cold extrusion to assist in lubrication. Billet surface finish, which affects the performance of lubricants at the die billet interface, was also investigated to a limited extent.

Further study of lubrication systems for AISI 4340 was also necessary because, in the past program(1), it was found that the effectiveness of some fluids and billet lubricants diminished as the fluid pressure level was increased. This was believed to be due possibly to (1) excessive viscosity of the fluid and (2) incompatibility of the fluid and billet lubricant.

The conditions under which the study of lubricants was made were essentially constant (see Table IX). Most of the trials were conducted at an extrusion ratio of 5:1 where the pressure level required to effect extrusion was sufficiently high (215,000 psi approximately) to permit judgement of the effectiveness of the lubrication systems. Some trials were conducted at an extrusion ratio of 4:1. For ease of reporting, the components of the lubrication system will be dealt with separately in the following discussion.

The repeatability of data from Tables IX and X were checked to determine the significance of the comparisons made later in the discussion. Three examples are given below where the data from three or more trials were obtained under nominally identical extrusion conditions.

Trials	Standard Deviation, psi		Coefficient of Variation, percent	
	P_b	P_r	P_b	P_r
209 through 212	1,120	435	0.52	0.2
238 through 241	810	810	0.31	0.31
257, 277, 280	3,500	2,160	1.65	1.0

It is seen that except for the third example, the repeatability of the data is very good. In the several cases where trials were repeated only once, the agreement in results is excellent; differences in the order of about 1000 psi are recorded. On this basis it is felt that reliable conclusions can be made on the results obtained in a single trial.

Billet Lubricant and Coatings

The effects of the individual billet lubricants in conjunction with castor oil as the fluid medium, on fluid pressures and an assessment of billet finish are identified in Table XII. The table condenses the data presented in Table IX for direct comparison. A measure of the effectiveness of the lubricants is given by comparison of breakthrough and runout pressures. The reference lubricant chosen was L17 (20 wt percent MoS_2 in castor wax) without a billet coating. This lubricant resulted in a low breakthrough pressure peak, smooth runout conditions, and an excellent surface finish on the extrusion.

The remaining lubricants used without a billet coating generally resulted in higher pressure levels than those obtained with L17 and with two of the systems some lubrication breakdown occurred on runout, which caused a poor finish. Stick-slip and scoring was encountered with L11 (castor wax). This result suggests that the solid film additive (MoS_2) to the castor wax, which comprises L17 was responsible for the prevention of lubrication breakdown obtained in the reference conditions. Lubrication breakdown did not occur on runout with Lubricants L31 and L38. Here the runout pressure levels were very close to that obtained with L17 though the breakthrough pressures were somewhat higher.

When billet coating Cl was used in conjunction with billet lubricants (Items 6 through 11), it invariably produced extrusions which had excellent finishes and which required lower pressure levels than those required for the reference lubrication system. Except for two lubricants, the pressure curves were of the Al type which exhibited no breakthrough pressure peak. Three lubricants, L19, L20, and L21 used with coating Cl gave the lowest runout pressure level achieved at 5:1. This pressure level, 208,000 psi, was nearly 4 percent lower than the runout pressure for the reference system.

The relative effectiveness of Lubricants L18, L19, L20 and L21 in comparison with Lubricants L11 and L17 appears to be associated with the melting points of the constitient waxes. The lowest melting point lubricant (L20) required the least P_b to effect extrusion whereas the highest melting point lubricant (L21) required the highest. The lubricants with similar melting points (L18, L19, L11 and L17) required about the same P_b.

Coating Cl without a separate billet lubricant (Item 12) performed satisfactorily. Even though the breakthrough pressure peak was high and small amplitude stick-slip occurred, smooth runout conditions were achieved and an extrusion having an excellent finish was produced. Apparently, the lubrication provided by the castor oil, the hydrostatic fluid, was fairly adequate. However, castor oil without both a billet coating and lubricant could not prevent momentary seizure at the die billet interface and the product was scored. Pressures were increasing rapidly during runout and complete seizure might have occurred had the extrusion stroke continued further.

Two other coatings were evaluated. Coating C3 (metal-free phthalocyanine) was not as effective as Cl. However, experimental data from friction tests on C3 suggests that its effectiveness may be better at elevated temperatures. Coating C4 (lead) was not effective by itself, but in conjunction with Lubricant L11 it provided extremely low friction conditions at breakthrough. However, the pressures rose continually on runout. The lead coating apparently broke down by melting as the extrusion temperatures rose. Consequently, the extruded surface was discontinuous and spotty. The surface temperature of the product, as measured by a contact pyrometer about 30 seconds after extrusion, was found to be around 600 F.

TABLE XII. EFFECT OF BILLET LUBRICATION IN HYDROSTATIC EXTRUSION OF AISI 4340 WITH CASTOR OIL AS THE FLUID MEDIUM

Extrusion ratio – 5:1 Die angle – 45 degrees (included) Stem speed – 20 ipm

	Billet Lubrication(a)		Average Fluid Pressure, 1000 psi						
Item	Coating	Lubricant	Breakthrough	Percent Reduction	Runout	Percent Reduction	Number of Trials	Lubrication Breakdown or Stick-Slip	Surface finish
1(b)	None	L17	220.5	–	216.3	–	6	No	Good
2	None	L11	209	5.2	235	-8.6(c)	1	Yes	Scored
3	None	L38	230	-4.5	218	-0.8	1	No	Good
4	None	L31	230	-4.5	217	-0.3	1	No	Good
5	None	L53	225	-2.0	221	-2.2	1	Yes	Fair
6	C1	L11	213	3.2	213	1.5	2	No	Good
7	C1	L17	215.5	2.3	212.3	1.9	4	No	Good
8	C1	L18	213.5	3.2	213.5	1.3	2	No	Good
9	C1	L19	213.5	3.2	208	3.8	2	No	Good
10	C1	L20	207	5.7	208	3.8	2	No	Good
11	C1	L21	218.5	0.9	208	3.8	2	No	Good
12	C1	None	229	-3.85	215.5	0.4	2	Yes	Good
13	None	None	225	-2.04	229	-5.9	1	Yes	Scored
14	C3	None	P_b not achieved at 255,000 psi				1	Yes	–
15	C4	None	223	-1.1	229	-5.9	1	Yes	Fair
16	C4	L11	204	7.2	213	1.5	1	Yes	Fair

(a) Billet lubricants listed in Table 3.

(b) Reference conditions to which remainder are compared.

(c) Negative means higher pressures than reference system.

TABLE XIII. EFFECT OF FLUID MEDIUM AND BILLET LUBRICATION IN HYDROSTATIC EXTRUSION OF AISI 4340 AT 80 F

Die angle - 45 degrees (included) Stem speed - 20 ipm

		Billet Lubrication[(c)]		Average Fluid Pressure, 1000 psi				Number of	Lubrication Breakdown	Surface
Item	Hydrostatic Fluid	Coating	Lubricant	Breakthrough	Percent Reduction	Runout	Percent Reduction	Trials	or Stick-Slip	Finish
					Extrusion Ratio 5:1					
1	Castor oil[(a)]	None	L17	220.5	--	216.3	--	6	No	Good
	Polyethylene glycol	"	L22	226.5	-2.7[(b)]	219.5	-1.5	2	"	"
	Polyethylene glycol	"	L23	230	-4.3	228	-5.4	1	"	"
	Silicate ester	"	L17	219	0.7	219	1.2	1	"	"
2	Ethylene glycol	C1	L17	211	4.3	209	3.4	3	"	"
	Polyethylene glycol	C1	L17	210	4.7	209	3.4	2	"	"
	Ditto	C1	L19	207	5.7	211	2.5	1	Yes	"
	"	C1	L20	207	5.7	212	2.0	1	"	"
	"	C1	L21	211	4.3	213	1.5	1	"	"
3	Castor oil[(a)]	C1	L17	215.5	--	212.3	--	4	No	"
	Ethylene glycol	C1	L17	211	2.1	209	1.6	3	"	"
	Polyethylene glycol	C1	L17	210	2.6	209	1.6	2	"	"
4	Castor oil[(a)]	C1	L19	213.5	--	208	--	2	"	"
	Polyethylene glycol	C1	L19	207	3.0	211	1.4	1	Yes	"
5	Castor oil[(a)]	C1	L20	207	--	208	--	2	No	"
	Polyethylene glycol	C1	L20	207	0.0	212	1.9	1	Yes	"
6	Castor oil[(a)]	C1	L21	218.5	--	208	--	2	No	"
	Polyethylene glycol	C1	L20	211	3.4	213	2.3	1	Yes	"
					Extrusion Ratio 4:1					
7	Castor oil[(a)]	None	L17	189	--	188	--	2	No	"
	Water	"	L17	186	1.6	188	0.0	4	"	"
8	Polyethylene glycol	"	L22	184	2.4	188	0.0	1	"	"
	Polyethylene glycol	"	L23	189	0.0	185	1.6	1	Yes	"

(a) Castor oil used as the reference fluid in each section.

(b) Negative sign means higher pressures than the reference system.

(c) C1 - phosphate coating; billet lubricants listed in Table 3.

Table X shows that, except at the extrusion ratio of 6:1, Coating C1 with Lubricant L17 was generally a more effective lubrication system than L17 alone at both low stem speeds (6 ipm) and various die angles. At a ratio of 6:1, there was little difference between pressure levels for these two systems. In the evaluation of Lubricants L18 and L11 in conjunction with C1 at this ratio, moderate lubrication breakdown occurred on runout in both cases.

In summary, using Coating C1 with several good billet lubricants appears to be slightly more effective than the lubricants alone or with other coatings. However, it would not be necessary to use a billet coating if some small sacrifice in pressure level of about 4 percent could be tolerated. Billet Lubricant L17 alone was satisfactory at all ratios up to the maximum of 6:1 and economically would be the better choice for a production operation.

Hydrostatic Fluids

The results of trials with several hydrostatic fluids are summarized in Table XIII. The breakthrough and runout pressures are compared with the same reference system that was used for comparing billet lubricants, i.e., castor oil with Lubricant L17 alone. In addition, comparisons are made between data obtained when castor oil and other liquids were used as pressurizing fluids in the cases where billet lubricants and coatings were common.

The data in Table XIII indicate that the hydrostatic fluids tried had no significant difference in effect on the surface finish of the extruded product.

With coated billets, using ethylene glycol and polyethylene glycol as the pressurizing fluid gave virtually similar results. Both glycols usually resulted in lower extrusion pressures than castor oil. The results on uncoated billets, Item 1, for Lubricants L22 and L23 are exceptions to that statement. In those cases, the higher extrusion pressures with polyethylene glycol are attributed to effects of the billet lubricants rather than the hydrostatic fluid. Those lubricants appear to be pressure sensitive and less effective at higher pressures because they performed quite well under the less severe conditions indicated in Item 8.

Although the decrease in extrusion pressures attributed to the glycol fluids, indicated in Item 3, are small they appear to be real. The differences of about 2 percent indicate that the ethylene glycol type fluids provide lower friction conditions at the die/billet interface than does castor oil. However, polyethylene glycol and Coating C1 with Lubricants, L19, L20 and L21 (Item 4, 5, and 6) was not so effective in maintaining low friction conditions because during runout these systems broke down and stick-slip occurred. In spite of the stick-slip, breakthrough and initial runout pressures were less than obtained with castor oil. These apparent discrepancies in the performance of polyethylene glycol may be connected with the compatibility of the fluids with the billet lubricants.

Silicate ester was a particularly good fluid at 400 F, but in a single trial at room temperature (Item 1) it showed only marginal improvement over the reference system. Its high cost in comparison to castor oil prevents this fluid from being competitive in cold hydrostatic extrusion. Water, on the other hand might well prove to be an economical and practical replacement for castor oil. Four trials with water at a ratio of 4:1 (Item 7) proved it to be equally as effective as castor oil. Water is not known to be a good lubricant, therefore, its ability to lower breakthrough pressures by about 2 percent is attributed to its lower viscosity at that pressure level. The practical objection that water causes corrosion might be overcome by using an emulsion of soluble oil and water typical to that used in machining.

Billet Surface Finish

The effect of billet surface finish on extrusion pressure and surface quality was investigated for AISI 4340 at ratios of 4:1 and 5:1 (Tables IX and X). A comparison was made between standard machined surfaces (60-120 microinch) and relatively rough surfaces obtained by grit blasting followed by vapor blasting. The latter step was used to remove superficial grit and any sharp points and edges caused by grit blasting. Stem speeds of 20 and 80 ipm were used. Castor oil and water were used as the fluid media and L17 as the billet lubricant.

The extrusion pressures and extruded surface finishes were found by visual examination to be about the same for either the machined or grit-blast finish. This is an indication that the billet lubricant used was quite effective by itself and that a rough billet-surface finish in this case does not cause any significant pressure change.

Stem Speed

The influence of stem speed on extrusion pressures and surface quality was evaluated for uncoated AISI 4340 rounds at a ratio of 3.3, 4.0 and 5.0:1. Stem speeds up to 80 ipm, the maximum speed of the hydraulic press used, were investigated. The data for comparison are given in Table X.

At an extrusion ratio of 5:1, the complete range of speeds from 1 to 80 ipm was investigated and the effect of this range in speed on fluid pressures is seen in Figure 17. It is seen that increases in stem speed up to 20 ipm result in lower pressures but little further lowering of pressures is obtained beyond this speed. At 1 ipm, stick-slip occurred and consequently breakthrough pressures were high but above this stem speed, smooth runout-pressure curves were obtained.

The reductions in pressure requirements down to a constant level, as stem speed increased (shown in Figure 18) were consistent with previous findings with 7075-0 aluminum. Also, the fact that both the stem and fluid pressure readings followed the same pattern indicates that these pressure reductions are real and not due to any temperature change in the fluid due to the adiabatic heat of pressurization. Experiments on the effects of temperature in the accuracy of the manganin pressure-gage confirm these conclusions. They were reported in an earlier section of this report. The exit velocity of the extrusion at 80 ipm stem speed and a ratio of 5:1 was about 62 fpm. This speed is well within the range used in production processes for conventional hot and cold extrusion. It is worthy of note that no problems in sealing were encountered at 80ipm. Probably even higher exit speeds could be used without difficulty.

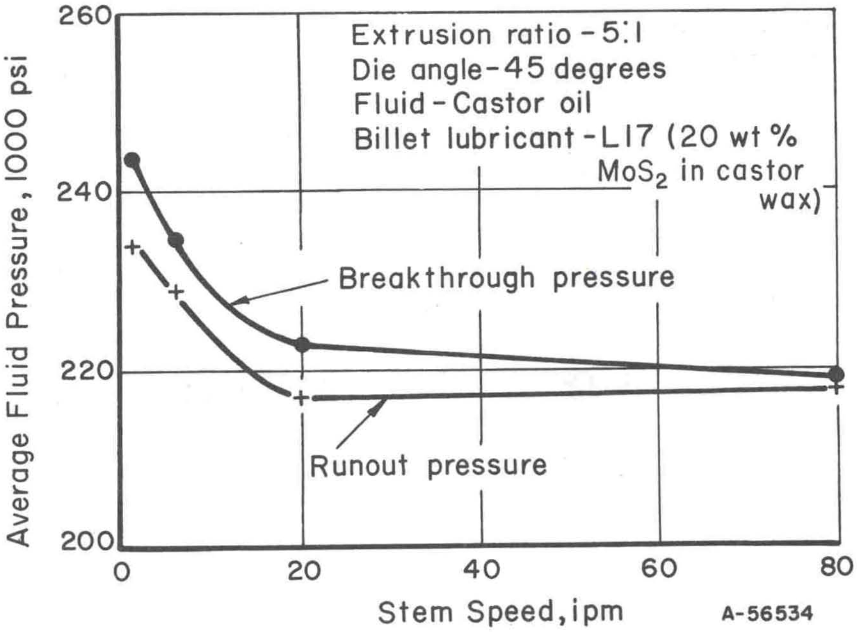


FIGURE 18. EFFECT OF STEM SPEED ON FLUID PRESSURES FOR COLD HYDROSTATIC EXTRUSION OF AISI 4340 AT AN EXTRUSION RATIO OF 5:1

Die Design

The effect of die angles on extrusion pressures was explored at ratios of 5:1 and 6:1. The results taken from Tables IX and X are summarized below in Table XIV.

TABLE XIV. EFFECT OF DIE ANGLE ON EXTRUSION PRESSURES AT TWO RATIOS

Fluid - Castor oil Billet Lubricant - L17 Stem Speed - 20 ipm

Extrusion Ratio	Die Angle (included), degrees	Average Fluid Pressure, 1000 psi Breakthrough	Runout	Number of Trials
5:1	30	--	--	1
	45	220.5	216.3	6
(no billet	60	230.0	227.0	2
coating)	90	--	--	2
6:1	45	231.0	231.0(a)	1
(phosphated billets)	60	236.5	233.5	2

(a) Diminishing stick-slip occurred on runout.

It is seen that at both extrusion ratios minimum extrusion pressures were achieved with the 45-degree die angle, though the requirements for a 60-degree angle were only marginally higher (about 4.5 percent at 5:1 and 1.5 percent at 6:1). In trials at 30- and 90-degree angles at 5:1, breakthrough was not achieved even at pressures greatly in excess of those needed for dies with angles of 45 and 60 degrees. Therefore, it is concluded that a die angle of 45 degrees is close to the optimum for processing AISI 4340 steel at these high ratios. At the low ratio of 2:1 it was shown in previous work(1) that a die angle of 30 degrees gave lower pressures than were required for a 45-degree angle. But the converse was true at 3.3:1 ratio. These general observations are consistent with the findings for several other materials by other research workers that the optimum die angle decreases as extrusion ratio decreases(11, 12).

It is of interest that vestiges of the original machining marks on the billet were noted on the extrusions made with the 45-degree-angle die but not with the 60-degree-angle die. Apparently, sufficient distortion or burnishing of the surface occurs with the 60-degree die to obliterate the machining marks.

Hydrostatic Extrusion of AISI 4340 Steel at Elevated Temperatures

The essential aims of the study at elevated temperatures were:

(1) To determine the effect of elevated temperatures on pressure requirements and product quality

(2) To develop lubrication systems and sealing techniques which would function successfully up to 500 F, the maximum temperature design capability of the hydrostatic extrusion tooling.

Table XI gives data obtained in the evaluation of several lubrication systems (billet lubricants + fluids) at three temperature levels. Most of those systems operated efficiently by giving smooth runout conditions and producing good quality extrusions.

Extrusion at 140 F

Studies at 140 F were an extension of experiments which were initiated in the earlier study(1) with this material. The use of slightly elevated temperatures was explored as a possible means of reducing fluid-pressure requirements. The main purpose here was to prevent an excessive increase in viscosity or solidification of the system resulting from increasing pressure. In these trials, only the fluid was heated to 120 or 140 F. The billet, container, and die were at room temperatures. Data from Tables X and XI and from earlier work(1) are summarized in Table XV.

TABLE XV. COMPARISON OF PRESSURES OBTAINED IN THE HYDROSTATIC EXTRUSION OF AISI 4340 STEEL AT 80, 120, AND 140 F

Fluid – Castor oil   Extrusion Ratio – 5:1

Lubrication	Stem Speed, ipm	Nominal Fluid Temperature, F	Average Fluid Pressure, 1000 psi		Number of Trials
			Breakthrough	Runout	
C1 + L17	20	80	215.5	212.3	4
C1 + L17	20	120	211.0	207.0	1
C1 + L17	20	140	210.5	206.5	2
C1 + L17	6	80	220.0	215.6	3
C1 + L17(a)	6	120	214.6	213.0	3
C1 only(b)	20	80	229.0	215.5	2
C1 only(b)	20	140	222.0	210.0	1

(a) Data from Reference (1).   (b) Stick-Slip before smooth runout.

The pressures for the trials at 120 F and 140 F for a 20-ipm stem speed were little different, yet for each of the three lubrication systems, the higher temperatures consistently gave lower pressures than were obtained at room temperature. The pressure differences in each case were not large, about 2 to 3 percent, but appear to be statistically significant.

It is believed that these small pressure reductions were due entirely to a reduction in viscosity of the castor oil at the higher temperature. However, the marginal benefits of working with castor oil at 120-140 F did not warrant a further study of the technique.

It is well to point out that the practice of preheating castor oil did not change the temperature of the "active" manganin coil sufficiently to have a significant effect on fluid-pressure measurements. This is evident from the essentially identical fluid pressures obtained at both 120 and 140 F given at the top of Table XV. Yet, Figure 18 shows that the reduction in the resistivity of manganin is as great between 80 F and 120 F as it is between 120 and 140 F. These results suggest that the coil itself was not heated much above room temperature, probably because of inadequate time available during the extrusion stroke and the fact that the fluid was cooled to some extent by the cold tooling.

Extrusion at 400 and 500 F

In the hydrostatic extrusion of AISI 4340 steel at 500 F, the variables investigated included fluids, lubricants, and extrusion ratio. In addition, some trials were made at 400 F (shown separately in Table XI) because the fluid used (silicate ester) had a flash point at 470 F. In all of these trials, the fluid, billet, die, and container were at the same temperature. Details of the heating, stem-seal arrangements, and fluid-pressure measurements are given in the section on equipment and procedure.

Effect of Fluid.

The following fluids were evaluated to determine their relative merits from the standpoint of extrusion pressure reduction and operational performance at temperature:

(1) Polyphenyl ether
(2) Tricresyl phosphate
(3) Triaryl phosphate
(4) Chlorinated diphenyl
(5) Silicate ester.

Before these fluids could be evaluated, however, preliminary trials were necessary to select an effective billet lubricant. The polyphenyl ether (PPE) fluid was selected because of its reportedly good high-temperature stability. Based on these trials, a "best" billet lubricant was selected (L31, fluorocarbon telomer), and the other fluids were evaluated. Data listed in Table XVI summarize the results obtained with the various fluids.

At a ratio of 4:1, the data suggest that the silicate ester (SE) fluid requires the least pressure. This is particularly significant, since the extrusion temperature (400 F) in this case was lower than in the other trials. However, at an extrusion ratio of 5:1 there appears to be only a marginal difference between the pressures for SE and PPE. (PPE fluid, at a ratio of 4:1, required the highest pressures.) Such results at higher ratios are not unexpected, however, because of the more severe conditions at the billet-die interface.

TABLE XVI. EFFECT OF FLUID ON PRESSURES FOR WARM HYDROSTATIC EXTRUSION OF AISI 4340 STEEL

Trial	Extrusion Ratio	Extrusion Temperature, F	Fluid(a)	Type of Stem Seal(b)	Extrusion Pressure, 1000 psi: Breakthrough Stem	Breakthrough Fluid	Runout Stem	Runout Fluid	Extruded Surface Finish, microinch, rms: Transverse	Longitudinal
394	4.0	500	PPE	1t	198	196	197	194	26	27
410	4.0	500	TCP	2t	200	187	200	185	28	34
411	4.0	500	TAP	2t	202	192	201	191	50	57
412	4.0	500	CBP	2t	196	186	191	181	31	30
413	4.0	400	SE	2t	189	182	189	180	41	35
418	5.0	500	PPE	2t	243	213	233	206	45	45
420	5.0	500	PPE	2t	230	200	222	197	31	29
422	5.0	400	SE	2t	223	196	214	193	30	25

(a) PPE – Polyphenyl ether
TCP – Tricresyl phosphate
TAP – Triaryl phosphate
CBP – Chlorinated biphenyl
SE – Silicate ester

(b) 1t = one PTFE O-ring used on stem seal; 2t = PTFE O-rings used on stem seal.

Apart from their effects on pressure requirements, it is worthy of note that all of the fluids evaluated performed satisfactorily as pressure media in the 400 to 500 F range. The finishes obtained with each fluid ranged from good to excellent but with triaryl phosphate (Trial 411) some slight scoring was observed.

Effect of Lubricants. A good measure of the effectiveness of the lubricants is given by:

(1) The difference between fluid breakthrough pressure and the corresponding runout pressure for individual trials

(2) The occurrence of stick-slip evident from the pressure curve

(3) Surface finish of the extruded product.

An evaluation of several lubricants on this basis is contained in Table XVII.

With the exception of L33 (55 wt percent MoS_2 and 6 wt percent graphite in sodium silicate), all of the lubricants used at 500 F for AISI 4340 steel and with PPE as the fluid can be rated as good to excellent. Three of the lubricants, L31, L34, and L38, gave outstanding results. For these lubricants, low breakthrough-pressure peaks and uniform or decreasing runout pressures were achieved. In addition, the surface finish of the extrusions was exceptionally good in all three cases. However, the other lubricants are considered satisfactory except where criterion such as surface finish is unusually demanding.

Apparently, good lubrication of AISI 4340 in hydrostatic extrusion at 500 F is readily accomplished. Choice of the lubrication system for a production operation appears to depend on economic factors and availability.

TABLE XVII. EVALUATION OF LUBRICANTS USED IN EXTRUDING AISI 4340 STEEL AT 500 F

Extrusion ratio – 4:1 Fluid – Polyphenyl ether Stem speed – 20 ipm

Trial	Lubricant	Difference Between Breakthrough and Runout Pressures[a], 1000 psi		Extruded Surface Finish Rating	Type of Extrusion Curve[b]
		Stem	Fluid		
394	L31	1.0	2.5	Excellent	B1
393	L33	--	--	-- --	Breakthrough not reached
397	L34	2.0	1.5	Very good	A2
409	L35	4.0	4.0	Good; some lubrication breakdown	B1
399	L38	2.0	1.5	Excellent	B1
401	L38	3.5	5.0	Excellent	B2
407	L40	9.0	5.5	Good; some lubrication breakdown	B2
406	L43	3.0	4.0	Good; some lubrication breakdown	B2
408	L44	1.0	1.5	Good; small amount of lubrication breakdown	A4

(a) The runout pressure level for the above trials was on the order of 180,000 to 200,000 psi.

(b) See Figure 26.

Effect of Temperature. The effect of temperature (80 F and 400 F) on the fluid runout pressures required to extrude AISI 4340 is shown in Figure 17. Of necessity, the fluids, lubricants, and stem seals used at room temperature are different from those at 400 F. While these differences in conditions may obscure the precise effect of temperature, it is believed that temperature is mainly responsible for the pressure reductions obtained. The fluid runout pressure level obtained at room temperature was lowered by 8 to 10 percent at 400 F.

Tensile Properties of AISI 4340 Steel Hydrostatic Extrusions

The results of tensile tests on AISI 4340 steel extrusions are recorded in Table XVIII. The tensile data obtained on extrusions from Trials 315 and 340 are added to those obtained in the earlier program(1).

As would be expected, increases in extrusion ratio from 3.3 to 6:1 resulted in sizeable increases in both yield and ultimate tensile strength. Yield strength was tripled and the ultimate strength was doubled. However, it is worthy of note that there was no appreciable sacrifice in ductility.

Table XVIII shows that increasing the exit speeds (at a constant extrusion ratio of 5:1) had little effect on tensile or yield strengths, but may actually improve ductility slightly as measured by elongation.

TABLE XVIII. ROOM-TEMPERATURE TENSILE PROPERTIES OF AISI 4340 STEEL ROUNDS PRODUCED BY HYDROSTATIC EXTRUSION

Extrusion Ratio	Reduction in Area of Extrusion, percent	Trial	Speed, ipm		Ultimate Tensile Strength, psi	Yield Strength (0.2 Percent Offset), psi	Reduction in Area in Tension, percent	Elongation in 1 Inch, percent
			Stem	Exit[a]				
1	0	As-received bar stock			94.6	55.4	49.0	33.0
3.3	70	176	6	60	160.9	136.5	32.6	11
4	75	183	6	60	170.4	142.9	29.4	10
5	80	189	6	60	180.4	151.9	27.8	9.5
5	80	167	1	10	188.6	163.4	27.9	8.0
5	80	315	20	185	179.0	161.7	28.8	13.0[b]
5	80	340	80	740	178.8	160.9	29.8	11.5[b]
6	83	190	6	60	196.6	170.4	26.1	8.5

(a) Die orifice diameter constant (0.75 inch), billet diameter varied to achieve ratio except in Trials 315 and 340 where billet diameter was 1.75 inch.

(b) Percent elongation in 2 inches.

IX

HYDROSTATIC EXTRUSION OF Ti-6Al-4V TITANIUM ALLOY ROUNDS

Experiments in the hydrostatic extrusion of Ti-6Al-4V alloy were directed mainly towards developing an efficient lubrication system for extrusion ratios of 3.3 and 4:1. Work on this alloy in the previous program(1) had indicated that lubrication was the major problem because, even though extrusions were obtained at ratios up to 4:1, stick-slip and very poor finishes were obtained using the lubricants then developed. Moreover, when the lubricant broke down to an extent where metal-to-metal contact took place the alloy tended to gall or adhere severely to the die.

In this program, extrusions of excellent quality were achieved following the development of a few lubrication systems. At room temperature, it was found necessary to apply an anodized coating prior to billet lubrication. At elevated temperatures, however, billet lubricants alone were satisfactory and no benefit was gained by the use of the coating. Table XIX gives data obtained in the evaluation of lubrication systems at room temperature and Table XX gives the data obtained at 400 and 500 F.

Extrusion Ratio

Ti-6Al-4V alloy has a high yield strength and consequently the maximum practical extrusion ratio attainable within the 250,000 psi pressure capacity of the current tooling was 4:1. The pressure data obtained are plotted in Figure 19 for three temperatures (80, 120, and 400 F) to indicate the developments made during the program and the possibilities in the future. The curve designated 120 F originated from data obtained in the previous program at ratios ranging from 1.6 to 4:1(1). It is seen by extrapolation that extrusion ratios of greater than 10:1 may be possible at pressures of about 400,000 psi, providing efficient lubrication can be achieved at those pressures.

Lubrication at 80 F

Evaluation of Billet Lubricants Without Billet Coatings

While the application of a fluoride-phosphate coating, C2, gave the best results in the previous program(1), ten billet lubricants were evaluated in this program without billet coatings with the aim of developing an efficient and low-cost lubrication system. Section I of Table XIX gives the data obtained under constant extrusion conditions with these lubricants. Without exception, each trial resulted in either stick-slip or seizure at the billet-die interface.

Lubricants L24 through L27 and L39 contained substantial quantities of iodine. The purpose of the iodine was to react chemically with the billet surface to form a product that would offer less frictional resistance than the titanium alloy itself. Except for L27 and L39, these iodine-containing lubricants appeared to reduce the tendency towards stick-slip but the improvements were not significant. With lubricant L39, which contained

TABLE XIX. EXPERIMENTAL DATA FOR 80 F HYDROSTATIC EXTRUSION OF Ti-6Al-4V ROUNDS

Die angle - 45 degrees (included)
Fluid - Castor oil

Billet diameter - 1-3/4 inches
Billet surface finish - 60 to 120 microinches

				Extrusion Pressure, 1000 psi						
		Billet Lubrication(b)		Breakthrough		Runout		Type of Curve	Length of Extrusion,	
xtrusion Ratio(a)	Stem Speed	Coating	Lubricant	Stem	Fluid	Stem	Fluid	(Fig. 26)	inches	Comments
				Section I						
3.2	6	--	L17	240	222	240	117	C4	1	
3.3	6	--	L17	244	225	242	222	C4	1	
3.3	6	--	L17	202	188	--	--	C4	6	
3.3	6	--	L17	239	219	232	213	D3	7	
3.2	6	--	L24	202	190	216	198	C1	11	
3.3	6	--	L25	224	210	212	196	C1	11	
3.2	6	--	L26	223	203	210	186	D1	12	
3.3	6	--	L26	152	114	--	--	--	--	P_b not reached; fluid apparently s
3.2	6	--	L27	217	196	219	188	C4	5	
3.3	6	--	L28	245	226	--	--	--	1	P_b not reached
3.2	6	--	L28	249	225	--	--	--	--	Billet cocked; die broke
3.3	6	--	L28	240	224	--	--	--	--	P_b not reached
3.3	6	--	L29	240	225	237	222	C4	1	
3.2	6	--	L29	262	235	--	--	--	--	P_b not reached
3.3	6	--	L29	246	222	--	--	--	--	Billet cocked; die broke
3.3	6	--	L30	214	202	212	200	C4	1	
3.2	6	--	L30	250	223	--	--	D1	--	Billet cocked; die broke
3.2	6	--	L30	228	207	240	208	D3	9	
3.3	6	--	L31	240	223	235	220	C4	1	
3.2	6	--	L31	264	237	--	--	--	--	P_b not reached
3.3	6	--	L32	226	210	221	208	C4	1	
3.3	6	--	L39	268	240	--	--	--	--	P_b not reached
3.3	6	--	L39	276	242	--	--	--	--	P_b not reached

							Section II				
264	3.3	6	C2	L17	214	200	206	197	C4	2	
286	3.2	6	C2	L17	202	186	200	180	C4	6	
362	3.3	6	C2	L31	248	226	226	206	B1	9	
363	3.3	20	C2	L31	250	226	224	203	B1	11	
373	3.3	20	C2	L49	226	213	208	203	B1	11	
358	3.3	6	C2	L34	242	221	232	211	D3	5	
359	3.3	6	C2	L35	238	216	230	209	D3	5	
360	3.3	6	C2	L45	242	222	221	197	B1	10	
361	3.3	20	C2	L45	241	222	219	201	B1	11	
370	3.3	20	C2	L50	249	228	223	211	B3	10	
							Section III				
379[e]	3.3	6	C5	L8	272	245	--	--	--	--	P_b not reached
378	4	6	C5	L8	275	247	--	--	--	--	P_b not reached
368	3.3	6	C5	L17	230	211	219	201	C1	8	
374	3.3	6	C5	L17	223	207	207	196	B1	10	
369	3.3	20	C5	L17	228	213	218	202	B3	11	
376	4.0	6	C5	L17	271	244	242	224	D3	9	Small transverse cracks on extrusion
487	4.0	20	C5	L17	266	228	240	207	B1	13	Compound angle nose, A = 1.2[f]
450	3.3	6	C5	L31	250	222	220	196	C4	6	
466	4.0	6	C5	L31	285	250	--	--	--	--	P_b not reached
427	4.0	6	C5	L33	291	245	--	--	--	--	P_b not reached
426	3.3	6	C5	L38	232	204	216	192	B4	6	
372	3.3	6	C5	L45	243	218	216	203	B1	9	
							Section IV				
367	3.3	6	C6	L17	257	230	230	207	D3	5	Extrusion and die broke during runout

---

(a) Ratios of 3.2:1 were attempted using dies whose orifices were remachined to remove score marks obtained when extruding at 3.33:1.

(b) Billet coatings listed in Table IV; billet lubricants listed in Table III.

(c) Billet surface was roughened by grit blasting followed by vapor blasting.

(d) Fluid used was polyphenyl ether.

(e) Billet used in Trial 378 was used in Trial 379.

(f) See page 41 for details of compound-angle nose.

TABLE XX. EXPERIMENTAL DATA FOR HYDROSTATIC EXTRUSION OF Ti-6A1-4V ROUNDS AT 400 AND 500 F

Die angle - 45 degrees (included)   Billet surface finish - 60 to 120 microinches   Billet diameter - 1-3/4 inch

Trial	Extrusion Ratio	Stem Speed, ipm	Type of(a) Stem Seal	Billet Lubricant (Details in Table III)	Extrusion Pressure, 1000 psi				Type of Curve (Fig. 26)	Length of Extrusion, inches	Comments
					Breakthrough		Runout				
					Stem	Fluid	Stem	Fluid			
					Extrusion Temperature 400 F		Fluid - Silicate Ester				
415	3.3	6	2t	L33	178	170	177	168	A2	8	
416	4.0	6	2t	L33	212	198	206	194	B2	8	
496	4.0	20	1t + 1r	L33(b)	210	195	198	187	B2	12	Compound-angle nose, A = 1.2 inches(c)
					Extrusion Temperature 500 F		Fluid - Polyphenyl Ether				
400	3.3	6	1t	L30	205	210	--	--	--	2	P_b not reached
402	3.3	6	1t	L30	201	199	189	184	C4	3	
395	3.3	6	1t	L33	190	196	185	184	B2	10	
396	3.3	20	1t	L33	181	192	177	182	B2	11	
419	4.0	6	2t	L33	225	195	206	185	C1	10	
421	4.0	6	2t	L33	210	184	201	181	C1	12	
398	3.3	6	1t	L38	175	185	170	182	B3	8	
403	3.3	6	1t	L40	211	213	--	--	--	2	P_b not reached
404	3.3	6	1t + 1r	L43	191	182	188	181	C4	2	
405	3.3	6	1t + 1r	L44	226	216	--	--	--	2	P_b not reached

(a) 1t = 1 PTFE O-ring; 2t = 2 PTFE O-rings; 1t + 1r = 1 PTFE plus 1 rubber O-ring.

(b) Anodized coating applied.

(c) Details of compound-angle nose given on p 41.

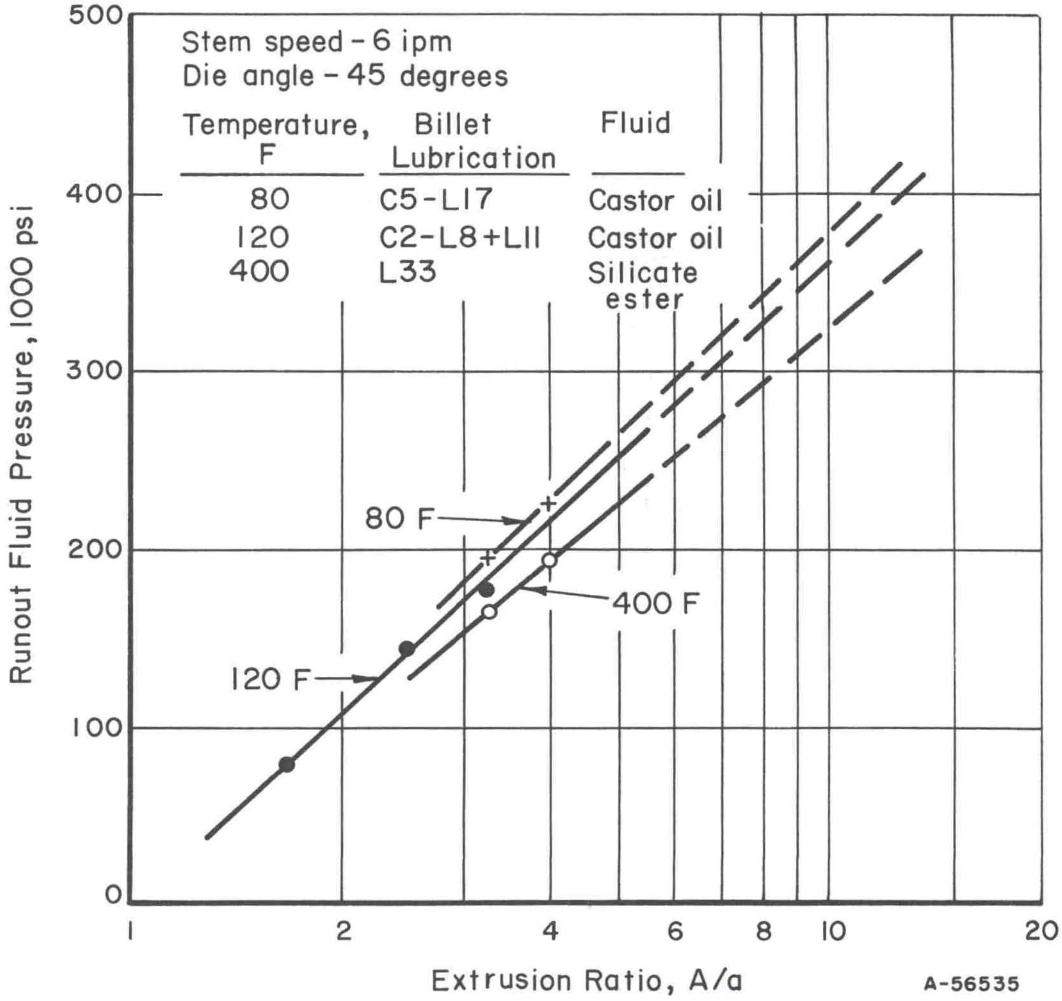


FIGURE 19. EFFECT OF EXTRUSION RATIO ON RUNOUT-FLUID PRESSURES FOR Ti-6Al-4V ROUNDS

20 wt percent MoS_2 in addition to the iodine, breakthrough did not occur despite the high pressure reached.

A single trial (Trial 375) was conducted to evaluate a grit-blast billet finish. Although this technique did not assist in eliminating stick-slip, breakthrough pressures were marginally reduced as compared with those of Trials 278 and 279 which were conducted under otherwise similar conditions.

During these trials without billet coatings, die wear was considerable. In removing the wear scars from the die-entry angle and die bearing by grinding, the opening of some of the dies was altered. This resulted in some slight reductions in extrusion ratio below the nominal ratio of 3.33:1. These reductions, however, were not sufficiently large to affect the lubrication-evaluation program.

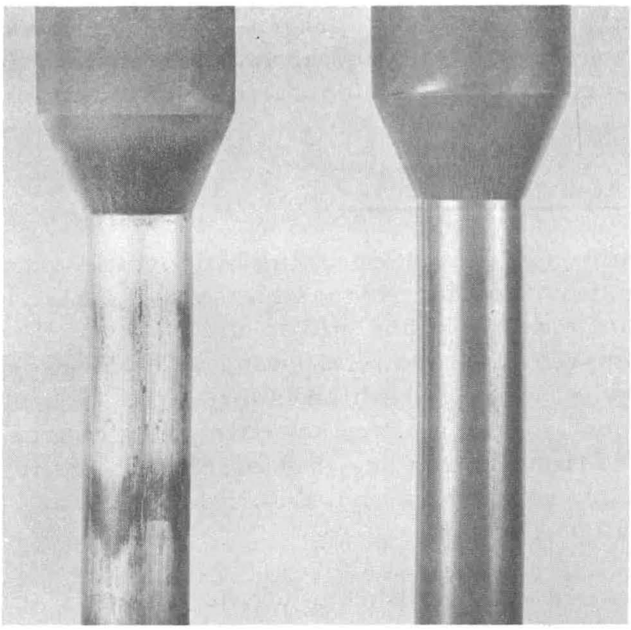
Evaluation of Billet Lubricants with Billet Coatings

Sections II, III, and IV of Table XIX give the data obtained with several billet lubricants applied with billet coatings C2, C5 and C6, respectively. In these trials, extrusion ratios of 3.3 and 4:1 and stem speeds of 6 and 20 ipm were evaluated also.

Several lubrication systems based on coatings C2 and C5 produced extrusions without stick-slip occurring. However, the system that produced extrusion with the most satisfactory surface finish was Coating C5 in conjunction with Lubricant 17 and castor oil as the fluid medium. Coating C5 was an anodized coating developed by Watervliet Arsenal primarily to improve wear resistance of titanium and has been designated as "titanium hardcoat" by the developers(13).

In two trials with this system at a ratio of 3.3:1 and 6-ipm stem speed (Trials 368 and 374), little or no stick-slip occurred during runout and excellent extruded surfaces were obtained. One of these is shown in Figure 20 in comparison with an extrusion obtained without the C5 coating. The surface finish obtained with C5 was in the order of 20 to 40 microinches in the transverse direction. Without the C5 coating, the surface was in the range of 120-150 microinches. At a ratio of 4:1 and stem speed of 6 ipm (Trial 376), stick-slip occurred on exit. While the product surface finish was excellent, small transverse cracks were observed at periodic points along the extruded surface. It is believed that the cracks were associated with the stick-slip cycles. In Trial 487, also conducted at a ratio of 4:1 but at a faster stem speed of 20 ipm and with a compound-angle billet nose, stick-slip was completely eliminated. An excellent surface finish without cracks was obtained. This improvement is attributed partly to increasing the stem speed and partly to using a compound-angle nose on the billet, but the extent of contribution of each variable is not known. One or both of these factors apparently also contributed to a 6-7 percent lowering of breakthrough and runout pressures.

In Trial 372, the C5 coating was used in conjunction with Lubricant L45. The extrusion pressure and the shape of the extrusion curve were similar to that obtained with C2 coating and L45 (Trial 360). However, the extruded surface quality was considerably better with C5 than it was with the C2 coating. Appreciable die wear occurred with C2, whereas no wear was noted with C5.



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	Trial 375	Trial 368
Lubrication System	(L17 lubricant alone)	(L17 lubricant + C5 anodized coating)
Surface Finish, microinches, rms (transverse)	120-150	20-40

FIGURE 20. COMPARISON OF SURFACE FINISHES ON COLD HYDROSTATIC EXTRUSIONS OF Ti-6Al-4V MADE AT A RATIO OF 3.33:1

Products having good finishes were obtained with both L31 and L38 in conjunction with C5. However, severe lubricant breakdown occurred in both cases during runout resulting in a rapid pressure rise. Lubricants L31 and L33 were evaluated at an extrusion ratio of 4:1 but breakthrough was not achieved in either case. It is worthwhile to point out, however, that L33 was a very effective lubricant in the hydrostatic extrusion of Ti-6Al-4V at 400-500 F even without the C5 coating. This was also true for L38 which, as mentioned above, was not effective at 3.3:1 and at room temperature. Two trials with L31 and C2 coating resulted in smooth runouts after some small initial stick-slip. The surface finish here was badly scored.

In view of the low pressure levels achieved with L31 and L45 on Coating C2, modifications were made to these lubricants in an attempt to improve the surface quality of the product. An addition of 20 wt percent graphite was made to both L31 and L45 which resulted in Lubricants L49 and L50, respectively. With C2 + L49, pressure levels were lowered further by about 6 percent but the surface finish was still poor. With C2 + L50, there was no improvement on the basis of pressure or surface finish, which indicated that the graphite addition to L45 had no apparent effect.

By comparison with coatings C2 and C5, the diffused-nickel-plate coating, C6, which was evaluated with L17 (Trial 367), did not perform satisfactorily. Standard bench tests of the sliding-friction/stick-slip type, however, showed that for commercial-purity titanium, the C6 coating reduced the friction coefficient and minimized stick-slip(14).

Fluids at Room and Elevated Temperatures

The selection of fluids for evaluation in the hydrostatic extrusion of Ti-6Al-4V alloy was guided somewhat by the practices established for AISI 4340. Consequently at 400 and 500 F only silicate ester (SE) and polyphenyl ether (PPE) were evaluated. The fluid used for all the room-temperature trials was, with one exception, castor oil. The exception was a polyphenyl ether (PPE) which in Trial 364 was intended to assist the lubrication of an iodine-containing lubricant by acting as a charge transfer medium to facilitate the formation of titanium iodide, the desired lubricating compound. However, the true effectiveness of this system was not determined because the fluid apparently solidified at about 114, 000 psi.

PPE and SE fluids were evaluated in the warm extrusion of Ti-6Al-4V titanium rounds at ratios of 3.3:1 and 4:1 with Lubricant L33. Comparison of the pressure data in Table XX indicates that at 3.3:1 the SE fluid reduces fluid-runout pressures on the order of 7 percent. However, at 4:1 there was no appreciable difference in pressure requirements between the fluids. These are similar to the results obtained with AISI 4340 at ratios of 4:1 and 5:1, respectively. It appears that the SE fluid is more effective than PPE in reducing pressure at the lower pressure levels (about 170, 000 psi for the lower ratios) than at the higher levels (about 195, 000 psi for the higher ratios). This may be due to some appreciable loss in lubricity resulting from the higher pressures and temperature developed at the billet-die interface during extrusion at the higher ratios.

Billet Lubricants at 400 and 500 F

The results obtained in studies with several billet lubricants at elevated temperatures are given in Table XX. No special billet coatings were applied before lubrication except in Trial 496 where coating C5 was evaluated.

One of the most significant findings was that Lubricant L33 alone (55 wt percent MoS_2 and 6 wt percent graphite in sodium silicate) was effective in completely eliminating stick-slip during both breakthrough and runout at extrusion ratios of 3.3:1 and 4:1 with the SE fluid (Trials 415 and 416) and 3.3:1 with the PPE fluid (Trial 395). Of particular importance is the fact that this was possible without any of the special coatings found essential for hydrostatic extrusion of this alloy at room temperature. The very low breakthrough pressure peaks and excellent surface finishes obtained indicated the complete effectiveness of Lubricant L33. Machining marks carried through from billet to extrusion gave additional evidence of its effectiveness. In Trial 496, the C5 coating was evaluated, together with increasing stem speed and using a compound-nose billet, with the aim of obtaining further improvements. The surface finish obtained was equally as excellent as that in Trial 416. However, the combination of the coating, higher stem speed, and compound-angle nose appeared to have had only a marginal effect on pressure levels.

PTFE lacquer (L38, Trial 398) also yielded a good extruded surface finish and a low breakthrough-pressure peak. However, continuous increase in the runout pressure after breakthrough indicated some lubrication breakdown.

Other lubricants investigated (L30, L40, L43, and L44) either did not permit breakthrough at relatively high pressures or, if breakthrough was achieved, broke down to the extent that severe stick-slip occurred during runout.

Effect of Temperature

Pressures for hydrostatic extrusion of Ti-6Al-4V at 400 F are plotted against extrusion ratio in Figure 19 with the pressures required at 80 F and 120 F. It is seen that in comparison with the pressures required at 80 F the pressures were reduced by 12 to 15 percent when extruding at 400 F. It is recognized, however, that a portion of this reduction may be attributable to other process conditions (including lubricants and fluids) which, of necessity, were changed for extrusion at 400 F.

Mechanical Properties of Ti-6Al-4V Titanium Alloy Rounds Produced by Cold Hydrostatic Extrusion

Tensile evaluations on Ti-6Al-4V alloy extrusions were not conducted in this research program because a study of their mechanical properties was made earlier(1). A summary of the data for extrusion ratios of 3.3:1 and 4:1 is given in Table XXI.

TABLE XXI. TENSILE PROPERTIES OF Ti-6Al-4V ALLOY ROUNDS PRODUCED BY HYDROSTATIC EXTRUSION

Stem speed - 6 ipm Exit speed - 60 ipm Die orifice - 3/4-inch diameter

Extrusion Ratio	Reduction in Area of Extrusion, percent	Trial	Ultimate Tensile Strength, 1000 psi	Yield Strength (0.2 Percent Offset), 1000 psi	Reduction in Area in Tension, percent	Elongation in 1 Inch, percent
1	0	As-received billet stock	143	135	39	21
1	0	Typical heat-treated properties	165	150	50	15
3.3	70	191	181	162	31	11
4	75	193	184	165	31	11

The strength levels obtained were significantly high for this alloy and were combined with reasonably good ductility. In fact, by heat treatment, a tensile strength of 165,000 psi and a yield strength of 150,000 are near the limits obtainable. Heat treatment, however, clearly provides for greater ductility than obtained by extrusion at this lower strength level.

Judging by the marginal increase in strength obtained in raising the extrusion ratio from 3.3 to 4:1, further increases in ratio probably may not cause any marked changes in the levels obtained.

X

HYDROSTATIC COMPACTION AND HYDROSTATIC EXTRUSION OF POWDER COMPACTS OF Ti-6Al-4V ALLOY POWDER

Hydrostatic Compaction of Ti-6Al-4V Titanium Alloy Powder

The hydrostatic extrusion process might be incorporated in several possible approaches in the area of compaction and extrusion of metal powders:

(1) Simultaneous hydrostatic compaction and extrusion of powder billets with or without subsequent sintering.

(2) (a) Hydrostatic compaction of powder billet
    (b) Sintering of billet
    (c) Hydrostatic extrusion of sintered billet.

The second approach was selected for investigation in this program, although it would be worthwhile to explore the first method as well sometime in the future.

Ti-6Al-4V prealloyed powder was selected for evaluation because of the strong current interest in it for aerospace applications, and also because of the opportunity to compare its mechanical properties with those obtained from the wrought alloy previously hydrostatically extruded in the program. The as-received Ti-6Al-4V powder was made by mechanical attrition and was shipped to Battelle under a helium atmosphere to minimize oxygen contamination.

In preparation for compaction, five rubber bags with nominal internal dimensions of 1-7/8-inch-diameter by 10 inches long were filled with powder. The compacts were vibrated during loading and the maximum fill density achieved was 2.04 g/cc or 46 percent of theoretical density based on the theoretical density of 4.43 g/cc for this titanium alloy.

Two billets were produced by compacting at a fluid pressure of 60,000 psi and three billets were compacted at fluid pressure of 225,000 psi. Each compact was held at pressure for between 10 and 15 seconds before the maximum pressure was slowly released. To compensate for shrinkage during compaction and the consequent lowering of fluid level in the container, the three billets pressed at 225,000 psi were compacted at two intermediate pressures, 15,000 and 65,000 psi. On attaining these pressures, the pressure was removed and fluid added, but the compacts were not disturbed.

The compacted billets were sintered at 2200 F for 1 hour in an argon atmosphere and water quenched as is customary with this alloy. One of the billets pressed at 60,000 psi broke up upon quenching. The theoretical densities of the billets before and after sintering are given below:

Compacting Pressure, psi	Percent of Theoretical Density	
	Before Sintering	After Sintering
60,000	93.4	93.2
225,000	97.5	97.5

A vacuum-fusion chemical analysis was made on the starting Ti-6Al-4V alloy powder and on samples of a billet after pressing to determine the oxygen and hydrogen levels. The results follow:

	O_2, ppm	H_2, ppm
Powder, as-received	700±20	140±3
Billet, pressed-and-sintered	1900±20	175±5

Although the oxygen level increased during processing it was only 100 ppm greater than industry specification for titanium and this level should not have caused the quench cracking obtained. The oxygen and hydrogen pick-up could have originated from either loading prior to compaction or in sintering. Metallographic examination showed the voids in the microstructure to be expected for material 97 percent dense. No directional effects were noted in the microstructure.

Two tensile tests were conducted on the specimens hydrostatically compacted at 225,000 psi. The ultimate tensile strength values were 120,800 and 107,500 psi. Both specimens exhibited brittle fractures and elongation values of zero. The properties of sintered compacts made by conventional compacting techniques (cold pressing) were reported by the powder supplier to be approximately 150,000 psi ultimate tensile strength and 4 percent elongation. The sintering conditions detailed above were generally as recommended by the powder supplier but they apparently were inadequate and perhaps resulted from incomplete bonding between the particles during sintering. Additional precautions to prevent oxygen pick-up might also assist in improving mechanical properties.

Hydrostatic Extrusion of Powder Compacts of Ti-6Al-4V Alloy Powder

Two billets obtained from hydrostatic compaction of Ti-6Al-4V alloy powder at 225,000 psi were prepared for hydrostatic extrusion. A nose conforming to the die-entry angle of 45 degrees was machined on the billets. They were anodized with the C5 coating, lubricated with L17 and attempts were made to extrude them at a ratio of 3.3:1 at a stem speed of 6 ipm. These were the extrusion parameters that produced good extrusions from wrought Ti-6Al-4V alloy billets. Each powder-compacted billet, however, fractured in the die entrance at about 225,000 psi and no material could be salvaged for testing (Trials 527 and 532). The breakthrough pressures required for wrought material under the same conditions are about 210,000 psi. Severe galling that occurred between the powder-compact billet and die during extrusion caused considerable die wear. This galling probably accounted for the higher pressure requirements. The poor extrusion behavior exhibited here was not surprising in view of the low ductility detected in tensile tests on the materials.

XI

HYDROSTATIC EXTRUSION OF SUPERALLOYS ALLOY 718 AND A286

The objective of this series of trials was to determine the extrudability of the A286 (iron-base) and Alloy 718 (nickel-base) superalloys. The results obtained are shown on Table XXII. With both alloys, good lubrication and excellent extrusion surface finishes were obtained.

TABLE XXII. EXPERIMENTAL DATA FOR HYDROSTATIC EXTRUSION OF SUPERALLOYS

Die angle – 45 degrees (included
Fluid – Castor oil at 80 F
Polyphenyl ether (PPE) at 500 F

Billet surface finish – 60-100 microinches (RMS)
Billet lubricant – L38
Billet diameter – 1-3/4 inches
Stem speed – 20 ipm

Trial	Extrusion Ratio	Extrusion Temperature, F	Extrusion Pressure, Breakthrough, Stem	Extrusion Pressure, Breakthrough, Fluid	Extrusion Pressure, Runout, Stem	Extrusion Pressure, Runout, Fluid	Type of Curve(b)	Length of Extrusion, inch
Superalloy – A286 (iron-based)								
479	3.3:1	80	198	173	190	165	B1	13
480	5:1	80	280	234	258	217	B1	19
500	5:1	500	235	(a)	217	--	B3	5
Superalloy – Alloy 718 (nickel-based)								
481	3.3:1	80	273	225	258	217	B1	15
484	3.3:1	80	285	238.5	270	226.5	B1	11
499	3.3:1	500	245	(a)	228	--	B3	3

(a) Fluid pressure gage out of order.
(b) See Figure 26.

A286 and Alloy 718 billets were received in the solution-treated condition. The initial hardnesses were 12 R_C and 16 R_C, respectively. All billets were lubricated with L38 (PTFE) and extruded at a stem speed of 20 ipm through standard-profile dies of 45-degree included angle.

It is particularly noteworthy that all extrusions were free of cracks. Extrusion at 500 F at the same ratios reduced the pressure requirements by about 15 percent. The lubrication here was not quite so effective as at room temperature because on runout lubricant breakdown resulted in increasing pressures.

Tensile Properties of Hydrostatic Extrusions of Alloy 718 and A-286 Superalloys

The results of tensile tests and hardness measurements made on hydrostatic extrusions of these superalloys are shown in Table XXIII. Prior to testing, each alloy was given a recommended aging heat treatment. Alloy 718 was heated at 1325 F for 8 hours, cooled 100 F/hr to 1150 F, held at 1150 F for 8 hours and air cooled. The A286 samples were heated at 1325 F for 12 hours and air-cooled.

TABLE XXIII. TENSILE PROPERTIES AND HARDNESS OF HYDROSTATIC EXTRUSIONS MADE FROM SUPERALLOYS A-286 AND ALLOY 718

Alloy	Extrusion Ratio	UTS, ksi	Yield Strength, ksi	Elongation, percent	Hardness, R_C		
					As Received	As Extruded	As Aged
A286	3.3:1	197.5	179.8	15	12	27-30	33
A286	5:1	200.0	180.9	11	12	27-30	34
A718	3.3:1	298.1	270.1	(a)	16	42-43	49

(a) Specimen broke outside the gage marks and no elongation value was obtained.

The results shown in Table XXIII exceed the tensile values commonly reported for the alloys. Typical tensile properties for A286 conventionally cold worked at 75 percent reduction and aged, are 150 ksi ultimate tensile strength, 140 ksi yield strength, and 2 percent elongation. Tensile properties reported for Alloy 718 similarly cold worked and aged are 250 ksi ultimate tensile strength, 230 ksi yield strength, and 6 percent elongation. Hydrostatic extrusions of Alloy 718 have been independently produced and evaluated by Watervliet Arsenal(15). The tensile results are in substantial agreement, but the extrusion ratio for the Watervliet experiments was not published and a direct comparison of the data cannot be made.

XII

COLD HYDROSTATIC EXTRUSION OF DISPERSION-HARDENED SINTERED ALUMINUM

The hydrostatic extrudability of an experimental dispersion-hardened sintered-aluminum product was evaluated at extrusion ratios of 10, 20, and 40:1. The billets were supplied by Oak Ridge National Laboratory and were 2-inch diameter x 2 inches long. In the as-received condition, their density was approximately 80 percent theoretical density. The billets were machined to 1-3/4-inch diameter and each was sandwiched between standard 7075-0 aluminum billets using a 1/8-inch-deep cylindrical counterbore joint. This joint is described later in Section II in connection with tandem-extrusion investigations. The sandwich-billet construction was used because the sintered-aluminum billets were too small to permit machining a 45-degree nose on one end. The construction also permitted complete extrusion of the billets. The billet lubricant was applied to the sandwich billet and to the joint interfaces. The extrusion data are contained in Table XXIV.

TABLE XXIV. EXPERIMENTAL DATA FOR COLD HYDROSTATIC EXTRUSION OF DISPERSION-HARDENED SINTERED ALUMINUM

Die Angle - 45 degrees (included)
Fluid - Castor oil
Billet Diameter - 1-3/4 inch
Billet Surface Finish - 60 to 120 microinches, rms
Stem Speed - 20 ipm

			Extrusion Pressure, 1000 psi					
			Breakthrough		Runout			
Trial	Extrusion Ratio	Billet Lubricant	Stem	Fluid	Stem	Fluid	Type of Curve(a)	Length of Extrusion, inch
475	10	L53	104	99	99	92	B2	20
476	20	L53	156	139	135	117	B2	40
490(b)	40	L53	221	202	--	--	C4	1/2
519(b)	40	L38	240	203	--	--	C4	0

(a) See Figure 26.

(b) Compound angle billet nose; 45 degrees at apex, 30 degrees beyond 0.75 inch diameter.

At ratios of 10 and 20:1, the whole billet was extruded in one piece. While the extruded products were craze cracked at the leading end, the remainder appeared to be sound. The extruded products were returned to ORNL for further evaluation. At 40:1, the lubricant film apparently broke down resulting in seizure at the die-billet interface after extruding about 1/2 inch of product. The 7075-0 aluminum nose had extruded satisfactorily. The discontinuity at the joint may have contributed to the failure of the lubricant film.

The runout pressure levels obtained at 10 and 20:1 ratios were marginally lower than those reported earlier for 7075-0 aluminum. The breakthrough pressures were probably influenced by the joint with the leading 7075 aluminum billet.

ORNL reported the mechanical properties of the hydrostatically extruded products(16). Table XXV gives the data obtained by tensile tests at 840 F, the reference-test temperature for this alloy. Also given are data for conventional hot (750 F) extruded rod.

TABLE XXV. MECHANICAL PROPERTIES OF SINTERED-ALUMINUM PRODUCT AS WORKED BY COLD HYDROSTATIC EXTRUSION AND HOT CONVENTIONAL EXTRUSION

Test Temperature - 840 F

Extrusion Process	Extrusion Ratio	Strength, psi: Yield, 0.2 Percent Offset	Strength, psi: Ultimate Tensile	Elongation, percent
Hydrostatic (cold)	10.0	6,800	7,400	0.45
Hydrostatic (cold)	20.0	7,050	7,550	0.39
Conventional (hot)	28.4	11,225	12,335	1.1

It is apparent that cold working of the dispersion-hardened SAP by hydrostatic extrusion did not develop as high a strength level as did conventional hot extrusion. The lower strength and ductility obtained possibly may have been associated with some microcracking or fluid penetration of the billet surface during hydrostatic extrusion of this material. Alternatively, the higher temperatures and ratios used in the conventional extrusion process may well have further consolidated the powder compact in addition to possibly improving intermolecular bonding.

XIII

HYDROSTATIC EXTRUSION OF BRITTLE MATERIALS

The aim of this series of trials was to establish the production capabilities of the hydrostatic extrusion of brittle materials. Two materials were selected which were known to behave in a brittle manner when subjected to cold work. These were:

(1) Wrought TZM molybdenum alloy (both the stress-relieved and recrystallized conditions)

(2) Beryllium (powder-metallurgy origin).

Tables XXVI and XXVII give the experimental data obtained in the developments leading up to and including the cold hydrostatic extrusion of crack-free products of both materials. This achievement was accomplished by use of a novel die design which eliminated the need for fluid back pressure. These developments, especially in the case of beryllium, are truly significant and represent a major breakthrough in the deformation of brittle materials. Furthermore, in the hydrostatic extrusion of beryllium at 500 F, the data in Table XXVI indicate that sizeable extrusion ratios, up to 8:1, are possible within the present capacity of 225, 000 psi at that temperature level.

Both TZM and beryllium displayed similar tendencies towards cracking. The cracks typically exhibited by these materials are circumferential ("rattle-snake" or "fir-tree" type) and longitudinal cracks. Historically, crack-free extrusions of both these materials were generally obtainable only when the product was hydrostatically extruded into a chamber producing a fluid back-pressure(17, 18): this technique is sometimes referred to as differential-pressure hydrostatic extrusion or fluid-to-fluid extrusion. An alternative method involving only die design was investigated in this program with the aim of eliminating the complexity and limitations of a second high-pressure fluid container.

Extrusion Ratio

The data in Tables XXVI and XXVII are plotted in Figure 21 to show the relationship between extrusion ratio and extrusion pressure at 80 and 500 F. The figure gives fluid pressures for the results obtained at 80 F but at 500 F, stem pressures are plotted. (The fluid pressure gage was out of action at this time. Fluid pressures at 500 F would be at least a few percent lower than the stem pressures plotted.) Beryllium and stress-relieved TZM apparently require similar pressures at 80 F. At 500 F, beryllium requires about 20 percent less and TZM (SR) about 7 percent less pressure than that at 80 F.

Extrapolation of the curve at 80 F indicates that both beryllium and TZM can be extruded at ratios of about 30:1 by a fluid pressure of 450, 000 psi. The estimated ratio achievable at 500 F for beryllium within that pressure capacity is approximately 50:1.

It is of interest to compare the data reported by Pugh in his review papers on hydrostatic extrusion(2). All the results he quoted indicated that beryllium required much higher pressures than those shown in Figure 21. The lowest of these pressures

TABLE XXVI. EXPERIMENTAL DATA FOR HYDROSTATIC EXTRUSION OF TZM ROUNDS AT 80 AND 500 F

Die angle - 45 degrees (included)   Billet diameter - 1-3/4 inches   Billet surface finish - 60 to 120 microinches

Trial	Extrusion Ratio	Die Design[a]	Stem Speed, ipm	Billet Lubricant (Details in Table 3)	Extrusion Pressure, 1000 psi				Type of Curve (Fig. 26)	Length of Extrusion, inches	Cracks[b]	
					Breakthrough		Runout					
					Stem	Fluid	Stem	Fluid			Circumferential	Longitudinal
					Wrought TZM, Stress Relieved							
Temperature - 80 F, Fluid - Castor Oil												
441	2.5	S	6	L17	156	140	136	122	B4	5.0	Nose only	3
442	2.5	C1	6	L17	156	140	140	127	B4	4.0	Ditto	4 split at nose
469	2.5	D1	6	L38	157	141	142	129	B1	4.0	None	3
452	3.3	C2	6	L17	240	210	184	165	C1	10	Nose only	3
455	3.3	C2	6	L38	224	198	184	165	C2	10	Ditto	3
478	4.0	D2	6	L38	280	242	--	--	B4	1.0	None	None
505	4.0	D4	6	L38	252	218	205	183	B1	5.0	Nose only	4
514	4.0	D5	20	L38	245	215	--	--	--	3.5	None	None[c]
443	5.0	S	6	L17	280	237	240	207	C3	7.5	Nose only	2 split at nose
Temperature - 500 F, Fluid - Polyphenyl Ether												
501	4.0	D3	6	L38	--	--	--	--	--	--	Die seal leak	
502	4.0	D4	6	L38	178	(d)	171	(d)	B2	7.0	None	None
					Wrought TZM, Recrystallized							
Temperature - 80 F, Fluid - Castor Oil												
460	3.3	C2	6	L38	172	155	137	125	C2	10.0	Nose only	3
483	4.0	D3	20	L38	198	176	194	168	B1	12.0	None	None

(a) S = standard die; C = controlled-relief die; D = double-reduction die (further details are given in Figure 22)

(b) Cracks occurred on the nose only when extruding through double reduction die with space between bearings.

(c) Lubricant breakdown due to previous pressurizing up to 216,000 psi when automatic cut-out on press functioned prematurely.

(d) Fluid pressure gage out of order.

TABLE XXVII. EXPERIMENTAL DATA FOR HYDROSTATIC EXTRUSION OF BERYLLIUM ROUNDS AT 80 AND 500 F

Die angle – 45 degrees (included) Billet diameter – 1-3/4 inches Billet surface finish – 60 to 120 microinches

Trial	Extrusion Ratio	Die[(a)] Design	Stem Speed, ipm	Billet Lubricant (Details in Table 3)	Extrusion Pressure, 1000 psi Breakthrough Stem	Breakthrough Fluid	Runout Stem	Runout Fluid	Type of Curve (Fig. 26)	Length of Extrusion, inches	Cracks[(b)] Circumferential	Longitudinal
					Temperature – 80 F, Fluid-Castor Oil							
377	2.5	C1	6	L17	142	139	134	130	D1	8	Many	Many
461	3.3	C2	6	L38	213	189	168	149	B2	11.5	Mostly at nose; few during runout	Five
495	4.0	D3	20	L38	234	205	228	200	B1	10	None	None
519	4.0	D5	20	L38	264	228[(c)]	--	--	--	2	None	None
520	4.0	D5	20	L38	234	203	216	193	B3	15	"	None[(d)]
528	4.0	D5	20	L38	228	202	--	--	--	3	"	None[(e)]
529	4.0	D5	20	L38	246	212	234	203	B3	18	Many	Many[(f)]
					Temperature – 500 F, Fluid – Polyphenyl Ether							
417	2.5	C1	6	L31	82	81	91	85	C4	5	Few	Few
503	4.0	D4	20	L38	150	(g)	143	(g)	B1	14	Numerous	Numerous

(a) S – Standard die; C = controlled-relief die; D = double-reduction die (further details are given in Figure 22.)

(b) Cracks occurred in the nose only when extruding through double-reduction die with space between bearings.

(c) Excessive billet-end pressure, due to billet-guide design, caused lubricant breakdown; maximum pressure indicated.

(d) Extrusion bent on exit and broke up on hitting a projection beyond the die.

(e) Press stopped prematurely after breakthrough.

(f) Heavy seizure of Be on the entry and surface of the second bearing, indicating severe lubrication breakdown.

(g) Fluid pressure gage out of order.

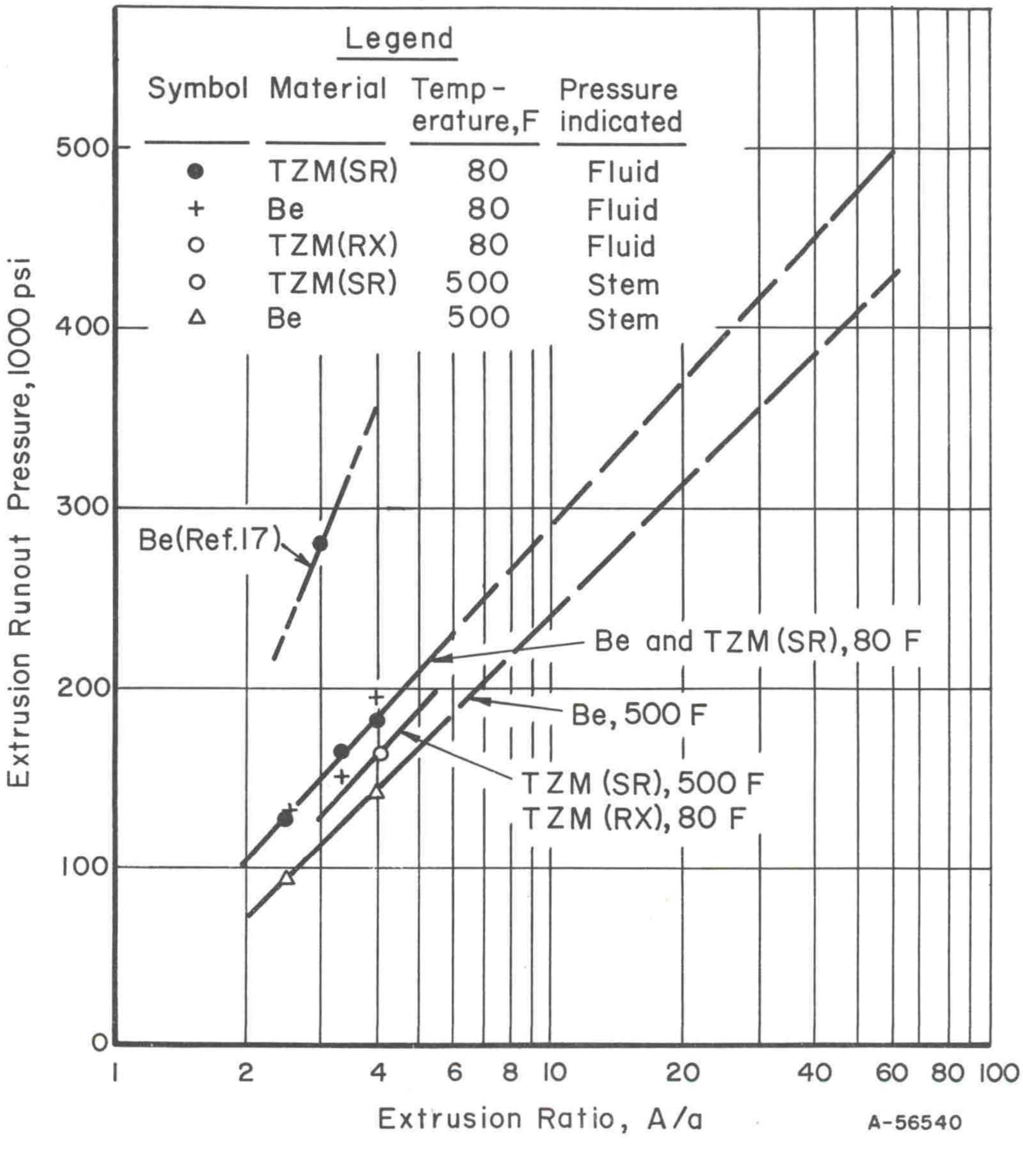


FIGURE 21. INFLUENCE OF EXTRUSION RATIO ON PRESSURES FOR BERYLLIUM AND WROUGHT TZM

TZM (SR) - stress-relieved TZM
TZM (RX) - recrystallized TZM

for an extrusion ratio of 3:1 is shown plotted in the upper curve in the figure. Even this result is significantly higher than the remainder in Figure 21. Due to the lack of more details of the billet material and extrusion conditions used by other workers however, it is not possible to account for these discrepancies.

Bobrowsky and Stack(18) obtained an extrusion from recrystallized TZM at a ratio of 4:1 without back pressure. The extrusion pressure of 175,000 psi lies close to the 169,000 psi level obtained in this program. Information on the surface condition of the extrusion, however, was not reported.

Die Designs

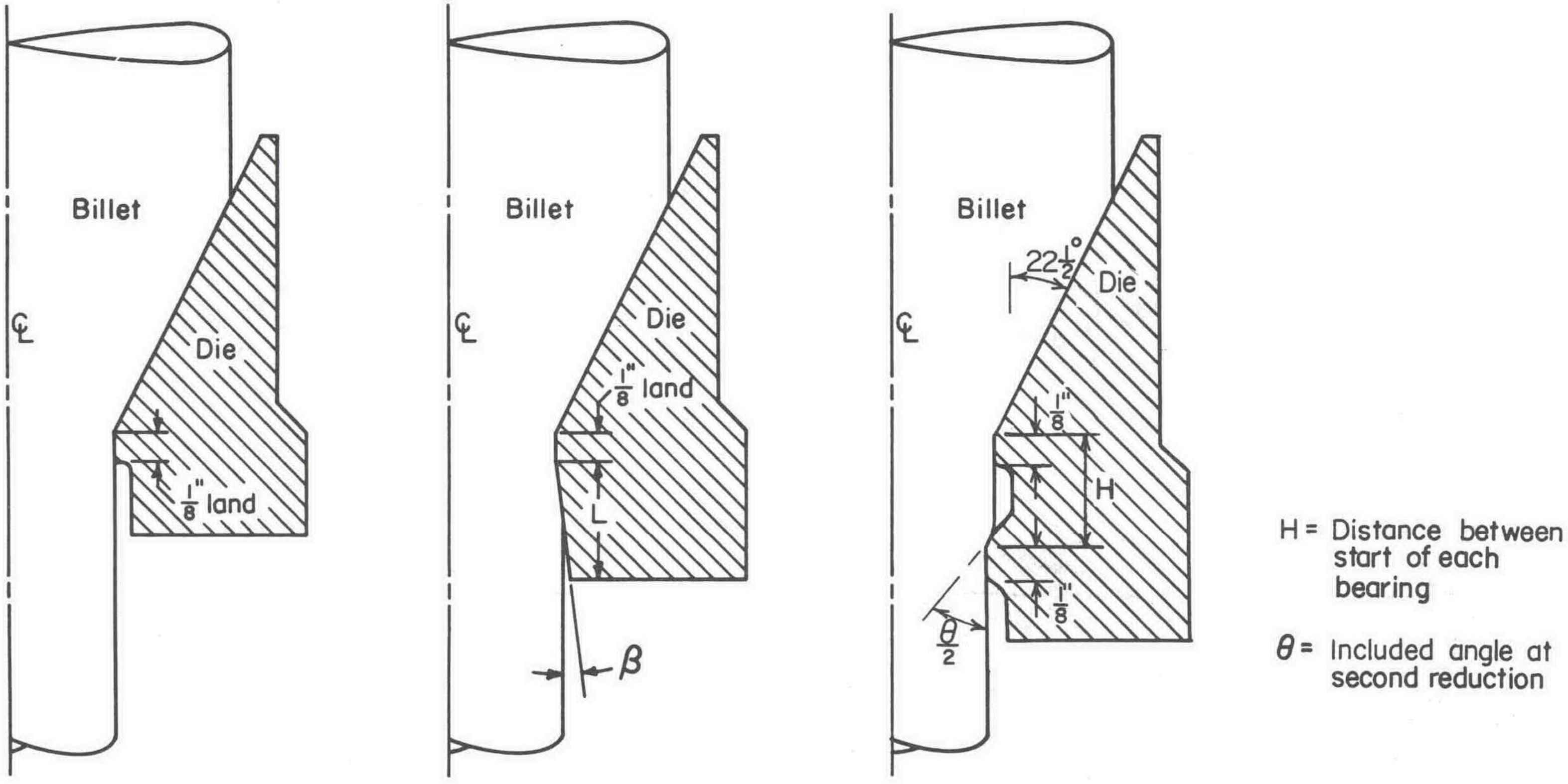
Figure 22 shows two die designs intended for use with materials which exhibit circumferential cracking or longitudinal cracking. The standard die design is also included. The controlled-relief die was designed to effect a gradual release of the elastic stresses present in the extrusion on exit from the die land. These elastic stresses are believed to be one of the major factors causing circumferential cracks on exit from the die bearing. To determine the amount of taper relief required, the elastic strain on exit was calculated based on an estimated flow strength of the extruded product. Two dies of this type were made: one for use at a ratio of 2.5:1 where the controlled relief was 10' (minutes of a degree) x 1/4-inch long (β x L in Figure 22) and the other for use at a ratio of 3.3:1 where the controlled relief was 1' 35" x 2 inches long. These were designated Dies C1 and C2, respectively.

The double-reduction die in Figure 22 was designed to take a very small reduction of the product at a second land shortly beyond the first. It is believed that the second reduction, in addition to preventing transverse cracks by imposing a longitudinal compressive stress, could prevent longitudinal cracking by effecting a favorable change in the residual stress pattern. Specifically, a favorable change would be in the direction of reducing the level of residual hoop tensile stresses in the product which give rise to longitudinal cracking. The magnitude of change in the pattern would appear to depend on extrusion conditions including the size of the second reduction, the distance between lands, the relief configuration after each land, billet material, die angle, extrusion speed, and extrusion temperature. It was only possible to investigate the effect of some of these variables in this program.

Several double-reduction die designs were evaluated:

Double-Reduction Die Designation	Second Reduction, percent	Distance Between Lands, H, inch[(a)]	Included Angle of Second Reduction, θ degrees[(a)]	Total Reduction, percent
D1	1.5	5/8	45	60
D2	3.3	5/8	45	75
D3	2.0	5/8	45	74.6
D4	2.0	1/8	45	74.6
D5	2.0	5/8	22	74.6

(a) See Figure 22 for details.



Standard Die

Controlled-Relief Die

Double-Reduction Die

A-54904

FIGURE 22. STANDARD DIE PROFILE AND TWO DIES DESIGNED TO ELIMINATE CRACKING IN BRITTLE MATERIALS

Effect of Die Design, Extrusion Ratio, and Temperature on TZM

The data given in Table XXVI are for wrought TZM in both the stress relieved and recrystallized conditions. While lower pressures were required for extrusion of the recrystallized TZM, both materials displayed a similar cracking behavior. Thus, both materials are treated together in the following discussion.

Evaluation of Die Designs at an Extrusion Ratio of 2. 5:1

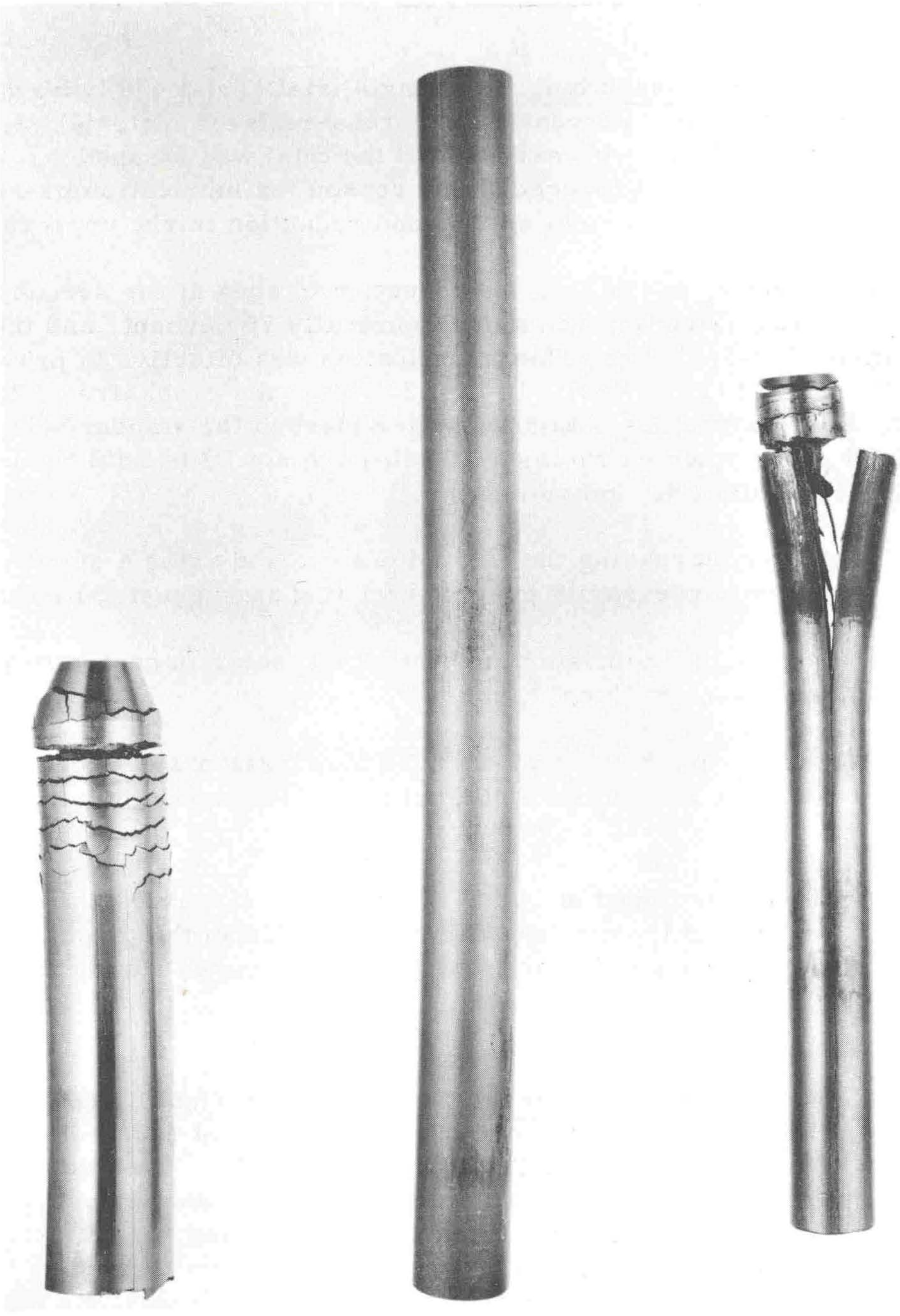
All three die designs (standard, controlled-relief, and double reduction) were evaluated with stress relieved TZM at an extrusion ratio of 2. 5:1. With the standard-profile die, the extrusion exhibited two types of cracking as shown in Figure 23. For about the first 2 inches of extrusion, the circumferential type occurred, and on the remainder a few longitudinal or axial cracks appeared on the product which otherwise had an excellent surface finish. The longitudinal cracks were generally of the fine, hairline type. The billet lubricant was L17 (20 wt percent MoS_2 in castor wax). Under the same conditions, the short controlled-relief die, C1, reduced the severity and extent of the circumferential and axial cracks. At the same ratio, the double-reduction die, D1, completely eliminated the circumferential cracks although three fine longitudinal cracks persisted. This die was designed with radial ports which could be open or closed to the fluid pressure in the pressure chamber. The purpose of using the die with the ports open was to lubricate at the second reduction. However, the fluid leaked at the second die bearing before runout was completed and, for later trials, the dies used were without ports as shown in Figure 23. With all three die designs, breakthrough pressures were the same but runout pressures for the controlled-relief and double-reduction dies were about 4 percent higher than with the standard die.

Controlled-Relief Die – Extrusion Ratio 3. 3:1

At a higher extrusion ratio of 3. 3:1, the long, controlled-relief Die C2 was used with Lubricants L17 and L38 on the stress-relieved material and L38 on the recrystallized material. The three extrusions obtained with these lubricants (Trials 452, 455, and 460) exhibited a few circumferential cracks on the nose only and three hairline longitudinal cracks on each. Lubricant L38 gave the better surface finish. A comparison of Trials 455 and 460 shows that the recrystallized material required about 22 percent lower pressures for both breakthrough and runout. Otherwise the performance of the two materials was identical. (The hardnesses of the as-received material were 196 and 276 DPH for the recrystallized and stress-relieved stock, respectively, which accounts for the appreciable difference in pressure requirements.)

Standard Die – Extrusion Ratio 5:1

In previous work at National Engineering Laboratory in Scotland(17), it was reported that cracks were eliminated by extruding above a critical extrusion ratio through standard dies. With molybdenum, the base metal of TZM, this was found to be 3:1. However, in the current program with TZM, cracks persisted even up to a ratio of 5:1, the maximum reduction attempted. This specimen from Trial 443 is included among those shown in Figure 23. The die used in this case had the standard relief profile.



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Trial	441	483	443
Extrusion Ratio	2.5:1	4:1	5:1
Billet Lubricant	L17	L38	L17
Die	Standard	Double Reduction(D3)	Standard

FIGURE 23. INFLUENCE OF DOUBLE-REDUCTION DIE ON CRACKING OF HYDROSTATIC EXTRUSIONS OF WROUGHT TZM MOLYBDENUM ALLOY

Double-Reduction Die – Extrusion Ratio 4:1

Size of Second Reduction. In a single trial (Trial 478) with double-reduction Die D2 (second reduction – 3.3 percent), and stress-relieved material, runout pressures rose rapidly due to lubrication breakdown and the trial was stopped. A crack-free, 1-inch length of extrusion was produced. The reason for lubrication breakdown was not clear, but it was believed that a smaller, second reduction might improve runout conditions.

Consequently, in Die D3, the reduction in area at the second bearing was 2.0 percent, the overall reduction remaining nominally 75 percent, and the space between bearings remained at 5/8 inch. This modification was effective in preventing cracks in recrystallized TZM (Trial 483). Figure 23 shows the crack-free TZM extrusion along with two other extrusions obtained earlier through the standard-die. The fact that cracking did not occur when extruding at 4:1 through die D3 but did occur at 5:1 (Trial 443) through the standard die indicates that:

(1) Merely increasing the extrusion ratio and using a standard-die profile may not necessarily prevent cracking as suggested by Pugh(17).

(2) Die design itself is a very important factor in controlling the conditions that cause cracking.

The surface finish of crack-free TZM extrusion was excellent (30 to 45 microinches, rms), even though the PTFE lubricant was apparently scraped off at the second bearing.

Die D3 was also used at 500 F (Trial 501). However, the die seal which was located in the base of the die (see Figure 9b) failed in this experiment. The O-ring material apparently expanded and was probably pinched during the lowering of the container.

The Space Between Bearings. A double-reduction die with no space (H = 1/8) between the bearing of the first reduction and the second reduction, designated Die D4, was evaluated with stress-relieved TZM at 80 and 500 F. The size of the second reduction, 2. 2.0 percent, was the same as for Die D3. At 80 F, the extrusion was cracked both circumferentially and longitudinally, but at 500 F a sound, crack-free extrusion was obtained. It appears that the crack-free product obtained here may have been due more to temperature than die design, particularly since TZM exhibits a marked increase in ductility at 500 F. The reduction in area in a room-temperature tensile test on stress-relieved TZM is about 55 percent whereas, at 500 F, the figure is 90 percent(19). A trial with the standard die at 500 F would determine whether elevated temperature was the sole factor here in controlling cracking.

The Angle at the Second Reduction. Although a crack-free extrusion of TZM was produced with an excellent surface finish through Die D3 (Trial 483), the lubricant (L38) had scraped off at the second bearing. In an attempt to prevent the lubricant from being scraped off and thus possibly reduce the extrusion pressures, Die D3 was modified to Die D5 in which the included entry angle to the second bearing was reduced from 45 to 22 degrees.

Die D5 was evaluated (Trial 514) with stress-relieved TZM and at a stem speed of 20 ipm. During breakthrough the automatic cutout on the press functioned prematurely. The fluid pressure at the point of cut-out was 215,000 psi and a 3 1/2-inch length of crack-free extrusion was produced. The die was also used later and successfully with beryllium.

Effect of Die Design, Extrusion Ratio, and Temperature on Beryllium

Most of the trials with beryllium were conducted at a ratio of 4:1 using the double-reduction dies, but useful information was also gained with controlled-relief dies. All of the data are recorded in Table XXVII.

Controlled-Relief Die

The short controlled-relief die (C1) was evaluated at both room temperature and 500 F (Trials 377 and 417) at a ratio of 2.5:1. In both cases the extrusions cracked badly although they remained in one piece. Fewer cracks occurred on the extrusion made at 500 F as shown in Figure 24. Lubricant L17 appeared to have performed adequately at 80 F. However, Lubricant L31 used at 500 F broke down immediately after the start of runout and severe galling occurred. In spite of galling, the pressure levels at 500 F were 40 percentlower than those obtained at 80 F where the lubrication was good.

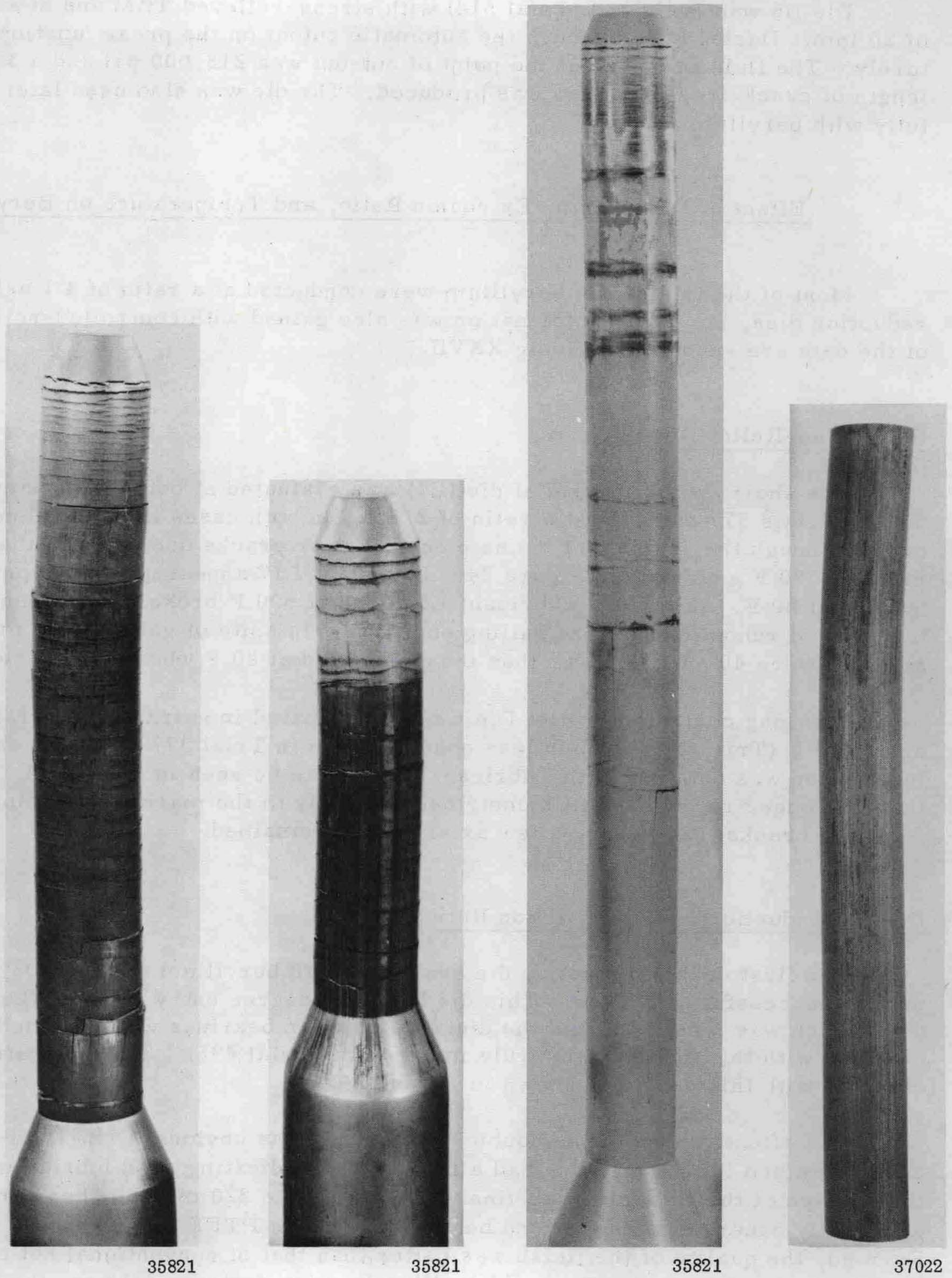
The long controlled-relief Die C2 was evaluated in extrusion at a ratio of 3.3:1 and at 80 F (Trial 461). Much less cracking than in Trial 377 occurred and excellent lubrication was obtained with Lubricant L38 as can be seen in Figure 24. It is believed that the longer relief in Die C2 contributed greatly to the marked reduction in circumferential cracks. However, a few axial cracks remained.

Double-Reduction Die – Extrusion Ratio 4:1

The first double-reduction die evaluated with beryllium was Die D3, which had proved successful with TZM. This die had a 45-degree entry angle to the second reduction (which was 2 percent) and the distance between bearings was 5/8-inch. The results obtained with this design were truly impressive (Trial 495). The crack-free extrusion obtained with this design is shown in Figure 24.

The effectiveness of the double-reduction die is obvious. The fluid-pressure curve obtained in this instance had a flat runout, indicating good lubrication. However, the surface of the extrusion was finely scored (130 to 220 microinches, rms), and this apparently occurred at the second bearing where the PTFE lubricant was scraped off. Even so, the quality of the finish was better than that of conventional hot extrusions. In the conventional hot extrusions of beryllium rod for commercial use, the billet is clad in a steel jacket and requires a billet temperature of 1850-1950 F.

Nondestructive inspection of the beryllium extrusion did not reveal any evidence of cracking on the surface in the extruded section beyond the nose. Severe transverse and longitudinal cracking occurred at the nose because the first 5/8 inch was extruded without the benefit of counterpressure from the second reduction (since the distance between bearings was 5/8 inch). Photomicrographs of the transverse and longitudinal sections



Trial	377	417	461	495
Extrusion Ratio	2.5:1	2.5:1	3.3:1	4:1
Temperature	80 F	500 F	80 F	80 F
Billet Lubricant	L17	L31	L38	L38
Die	Controlled Relief (C1)	Controlled Relief (C 1)	Controlled Relief (C2)	Double Reduction (D3)

FIGURE 24. INFLUENCE OF DIE DESIGN ON CRACKING IN HYDROSTATIC EXTRUSIONS OF BERYLLIUM

of the beryllium extrusion (Trial 495) are shown in Figure 25. The severely elongated grains in the longitudinal section are typical of a heavily cold-worked microstructure. In the preparation of the specimens shown in Figure 25, a single hairline crack about 0.15 inch long was seen in the longitudinal section. No corresponding crack was seen in the surface of the mating specimen. It is believed that this crack may have been a direct result of sectioning and not of extrusion.

The second double-reduction die evaluated with beryllium was Die D5. As discussed in the previous section on TZM, Die D5 was modified from D3 by reducing the entry angle to the second reduction from 45 to 22 degrees. This was done with aim of retaining the lubricant at the second bearing. Trials with this die were also conducted to obtain further lengths of crack free-extrusion for tensile evaluation.

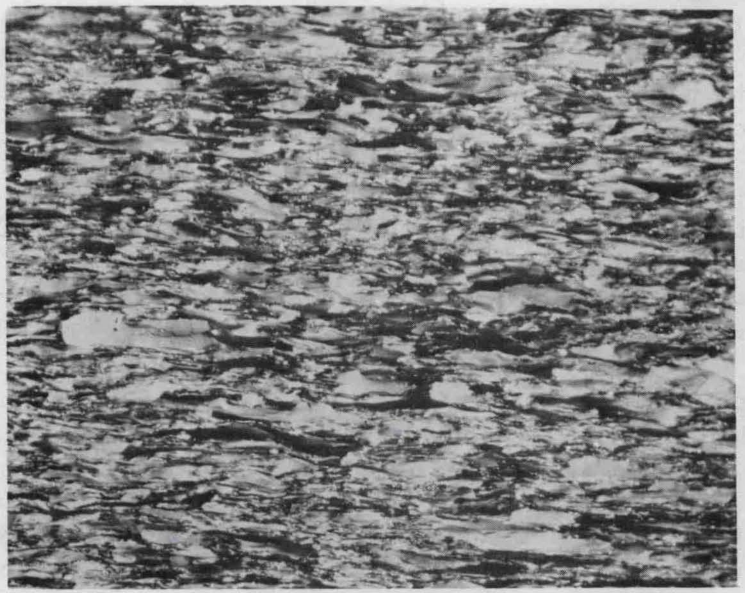
A die-entry plug was designed to stop the billet at a point where the unextruded billet length was about 1 inch. This would leave a long length of extrusion for evaluation. The purpose of the plug was to seal the die entry from fluid at pressure thus preventing further extrusion. Manual press control techniques were normally used to stop the extrusions, but the stem travel plotter could not indicate the billet position accurately enough during extrusion. The plug was mounted on the rear of the billet and was designed to move with it during extrusion.

However, the plug outside diameter of 2.313 inches apparently did not allow sufficient clearance between it and the container wall. During pressuring action by the ram, the plug apparently acted as a throttle to fluid flow which tended to cause unequal pressures above and below it. Thus at the point of breakthrough in Trial 519, irregularities in the pressure-ram travel curve were observed and the trial was stopped. A 2-inch length of crack-free extrusion was obtained.

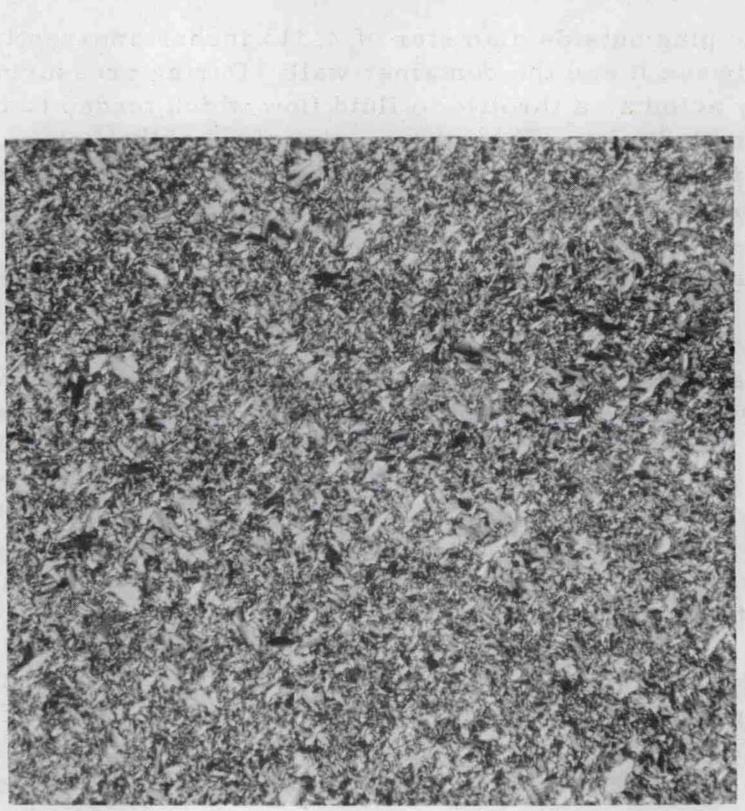
In three further trials with beryllium and double reduction Die D5, the die entry plug was not used. In Trial 520 a crack-free product having a similar finish to that in Trial 495 was obtained. Unfortunately, the extrusion bent slightly on exit and broke up on hitting a protrusion below the die. This caused fluid pressures to rise towards the end of runout. However, several continuous lengths of sound extrusion were obtained for tensile evaluation. A comparison of the pressure data obtained in these trials with Dies D5 and D3 is given in Table XXVII. A 3-1/2-percent reduction in runout pressure was achieved by reducing the angle at the second reduction but breakthrough pressures were equal because the size of the first reduction was unaltered. The small reduction in runout pressures was probably due to improved lubrication at the second reduction or due directly to the change in die angle.

The protrusion below the die was removed for the remaining two trials (528 and 529) with Die D5 in case these extrusions also bent on exit. In the first, the press was stopped unintentionally after a low breakthrough pressure had been achieved and only 1 inch of crack-free product was obtained. In the following trial, however, high breakthrough pressures occurred, the exiting extrusion was badly cracked and an uneven increasing runout pressure was obtained. An examination of the die after removal of the extrusion revealed that heavy seizure or galling had occurred at the entry surface of the second bearing.

The reason for seizure occuring at the second bearing in this particular trial was unaccountable since the conditions here were unchanged from those in which sound extrusions were produced. The greater friction caused by galling at the second bearing



100X a. Longitudinal 6B551



100X b. Transverse 6B007

FIGURE 25. PHOTOMICROGRAPHS OF BERYLLIUM COLD HYDROSTATICALY EXTRUDED AT A RATIO OF 4:1 THROUGH BATTELLE'S DOUBLE-REDUCTION DIE

and the attendant heavy surface scraping apparently counteracted any beneficial effects of the second reduction. Clearly, more work is necessary in this area of die design with the aim of preventing galling or failure of the lubricant film. Such a design might be a double-reduction die similar to D5 but without relief after the first reduction, or perhaps even a long straight or tapered bearing single-reduction die.

A single trial (No. 503) with the double-reduction die, Die D4, was conducted at 500 F. In this die, the second reduction immediately follows the 1/8-in.-long bearing at the first reduction. TZM was extruded crack free with this die under the same conditions. However, the resulting beryllium extrusion was badly cracked indicating that elevated temperature alone may be sufficient to prevent cracking of TZM but not of beryllium and that the die design with adjacent bearings apparently was not effective under the extrusion conditions used. More trials would be necessary to substantiate these findings.

The Potential of Die Design

To date, sound hydrostatic extrusions of beryllium and TZM have been obtained by other workers(17, 18) when the product was extruded into a high fluid pressure environment (fluid-to-fluid extrusion). The extrusion ratios were in the order of 2:1. The provision of a fluid back pressure requires expensive tooling on the exit side of the die sufficient in length to accommodate the long extrusion. Also, the main pressure chamber must contain pressures in excess of those required in fluid-to-air extrusion by the amount of back pressure. This severely limits the pressure level available for extrusion.

In the double-reduction die, a compressive stress is applied to the exiting extrusion. The magnitude of this stress is small because the results obtained so far indicate that the second reduction of 2.0 percent does not require any appreciable extra fluid pressure over that required for the first reduction. For this reason, it is believed that the function of the die is different from that obtained in fluid-to-fluid extrusion, where counter pressures up to 200,000 psi are required to obtain sound extrusions from brittle materials. The double-reduction die probably prevents cracking by setting up a different pattern of residual stresses in the material leaving the die. The critical effect results from the small deformations obtained at the second reduction.

Clearly, the results obtained so far are very encouraging and open up new potential applications of the hydrostatic extrusion process. For example, it appears possible that brittle materials may now be extruded into long lengths economically at temperatures previously considered impossible. Unique mechanical properties may well be obtained with these materials. Improvements in lubrication, dimensional tolerances, and contamination control can be expected at low working temperatures. In the case of beryllium, the problem of toxicity can be avoided without difficulty.

Mechanical Properties of Hydrostatic Extrusions of Beryllium

Four tensile specimens were prepared from the extrusion obtained in Trial 520. Special preparation techniques for this material were used, i. e., machining followed by chemical etching to remove damaged surface layers. The tensile properties of the specimens are given in Table XXVIII below along with those for the as-received material.

TABLE XXVIII. TENSILE PROPERTIES OF BERYLLIUM HYDROSTATICALLY EXTRUDED COLD AT AN EXTRUSION RATIO OF 4:1

Specimen	UTS, 1000 psi	0. 2 Percent Yield Strength, 1000 psi	Elongation, percent in 2 inches
As Received	51. 2	36. 9	2. 5
1	124. 5	98. 4	<1
2	120. 0	105. 7	<1
3	112. 1	104. 1	<1
4	117. 8	100. 0	<1

The material has been markedly strengthened by cold working. In fact, the ultimate and yield strengths obtained are about 50 and 100 percent higher, respectively, than typical values for commercial hot-extruded bar having the same oxide content. It is believed that hydrostatic extrusion of this material at higher ratios and at temperatures up to about 500 F could increase strength even further. Working at 500 F would permit an increase in extrusion ratio while still being within the cold working range.

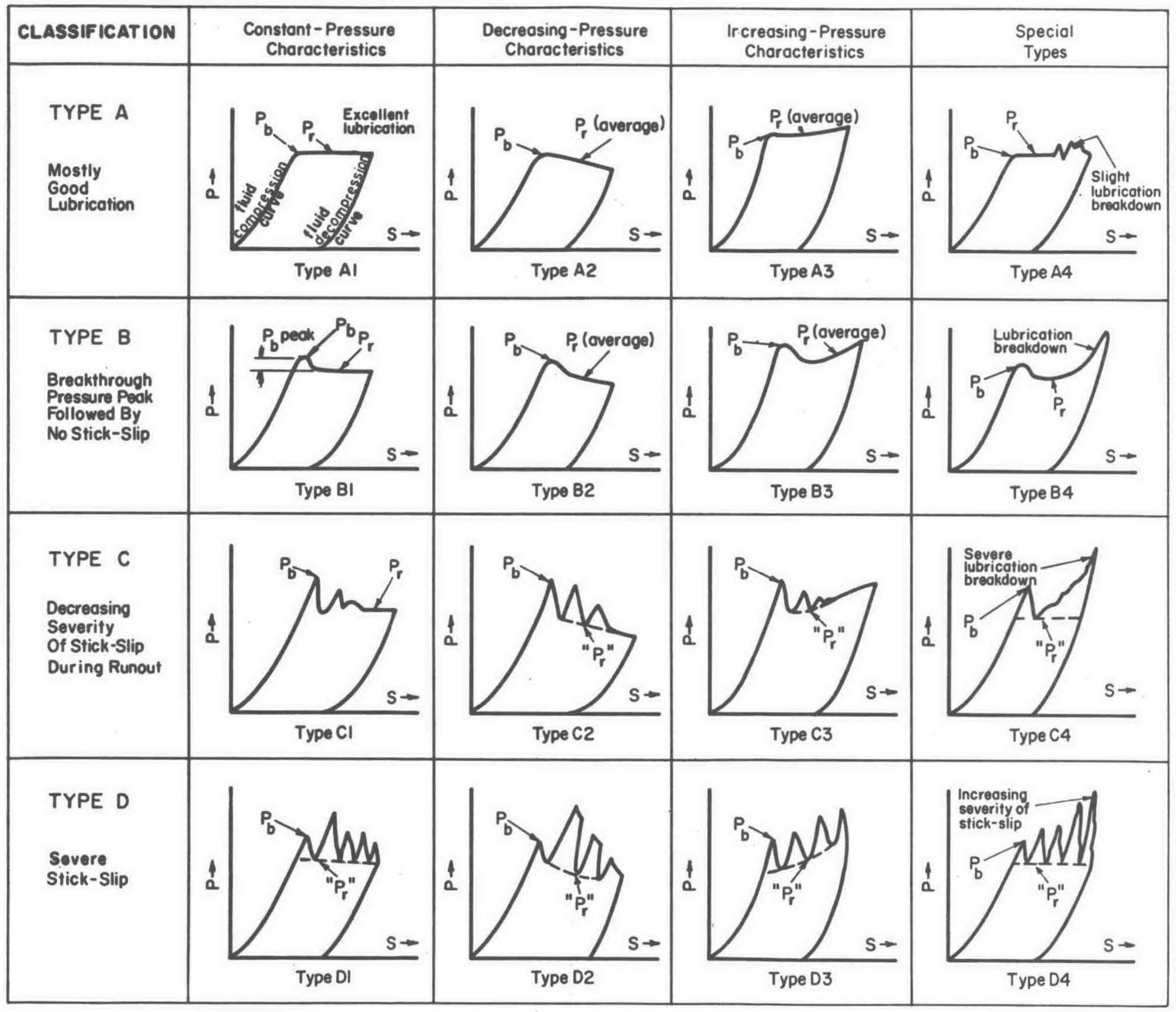


FIGURE 26. CLASSIFICATION OF PRESSURE-DISPLACEMENT CURVES OBTAINED IN HYDROSTATIC EXTRUSION