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# DEVELOPMENT OF THE MANUFACTURING CAPABILITIES OF THE HYDROSTATIC EXTRUSION PROCESS

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BATTELLE MEMORIAL INSTITUTE  
COLUMBUS LABORATORIES

INTERIM ENGINEERING PROGRESS REPORT IR-8-198 (VII)

September 1966

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DEVELOPMENT OF THE MANUFACTURING CAPABILITIES  
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## ABSTRACT

### Development of the Manufacturing Capabilities of the Hydrostatic Extrusion Process

R. J. Fiorentino  
et al.  
Battelle Memorial Institute

The purpose of the present program is to develop the manufacturing capabilities of the hydrostatic extrusion process. Specific applications to be studied are fabrication of wire, tubing, and shapes from relatively difficult-to-work materials such as refractory-metal alloys, high-strength steels and aluminum alloys, titanium alloys, beryllium, and other selected materials.

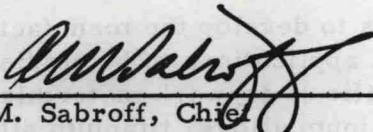
Investigation of critical process variables for cold hydrostatic extrusion of 7075-0 aluminum alloy was continued during this interim report period. Also, work was continued on the cold extrusion of steel and aluminum tubing and T-sections. In addition, warm hydrostatic extrusion of AISI 4340 steel, Ti-6Al-4V titanium alloy and beryllium was conducted. Important results obtained include:

- (1) Production of good-quality thin-walled tubing by re-extruding previously hydrostatically extruded tubing.
  - (a) With AISI 4340 steel, the cumulative reduction was 91 percent
  - (b) With 7075-0 aluminum the cumulative reduction was 98 percent.
- (2) In the extrusion of 7075 aluminum T-sections, a 10 percent reduction in breakthrough pressures has been achieved by using compound-angle dies. At a stem speed of 80 ipm, stick-slip on runout was eliminated.
- (3) A number of fluids and lubricants operated satisfactorily in warm hydrostatic extrusion of AISI 4340. Significant reductions in extrusion pressures were achieved over those required at room temperature.
- (4) Most notable in the warm extrusion of Ti-6Al-4V alloy rounds was the elimination of a billet coating that is required for satisfactory extrusion of this alloy at room temperature. Equally important is the elimination of a breakthrough-pressure peak and stick-slip during runout. Moreover, the extrusion pressures, compared with those at room temperature, were reduced on the order of 15 percent.
- (5) Beryllium extruded at 500 F displayed less cracking and required 50 percent less fluid pressure than that encountered at room temperature.

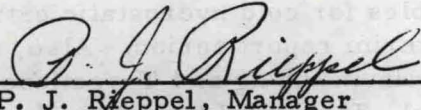
- (6) Preliminary trials in the hydrostatic extrusion and drawing of beryllium wire have provided much information on the experimental procedure, especially in the handling of fine wire. The equipment for applying the drawing stress has been designed and tested.
- (7) Tensile test data obtained on hydrostatic extrusions of AISI 4340 steel and 7075 aluminum rounds indicate significant increases in strength over that in the billet material with good retention of ductility.

#### PUBLICATION REVIEW

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## FOREWORD

This Interim Engineering Progress Report covers the work performed under Contract No. AF 33(615)-1390 from 1 June 1966 through 31 August 1966. It is published for technical information only and does not necessarily represent the recommendations, conclusions, or approval of the Air Force.

This contract with Battelle Memorial Institute of Columbus, Ohio, was initiated under Manufacturing Methods Project No. 8-198, "Development of the Manufacturing Capabilities of the Hydrostatic Extrusion Process". It is being administered under the direction of Mr. Gerald A. Gegel of the Metallurgical Processing Branch (MATB), Manufacturing Technology Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

The program is being conducted at Battelle by the Metalworking Research Division, with Mr. R. J. Fiorentino, Associate Chief, as project engineer. Others who have contributed to the program are the late W. R. Hansen, Research Metallurgist, Mr. B. D. Richardson, Research Metallurgical Engineer, Mr. A. M. Sabroff, Chief, and Mr. F. W. Boulger, Senior Technical Advisor. Mr. R. L. Jentgen, Project Leader in the Experimental Physics Division, is assisting in the fluid and lubrication studies of the program. Dr. J. C. Gerdeen, Research Mechanical Engineer, Mr. E. C. Rodabaugh, Senior Mechanical Engineer, and Mr. T. J. Atterbury, Chief of the Applied Solid Mechanics Division are contributing to the high-pressure-container design study. Mr. R. E. Mesloh, Research Mechanical Engineer of the same division, is assisting in the design of an instrument for measuring fluid pressure at elevated temperatures. Mr. H. L. D. Pugh, Visiting Professor of Metallurgy, Case Institute of Technology, is contributing to the beryllium-wire fabrication study as a consultant. Data from which this report has been prepared are contained in Battelle Laboratory Record Books Nos. 21799, 21990, 23055, 23287, 23585, 23791, and 23836.

\* \* \* \* \*

It is with regret that we announce the loss of Mr. W. R. Hansen, who was killed in an auto accident in mid-August. Mr. Hansen was responsible for conducting most of the experimental work carried out so far in this program and contributed greatly to the developments that have taken place. He was dedicated to the task set in the project and inspired enthusiasm among his colleagues. It is inevitable that a temporary delay in the work has occurred. Mr. Hansen will be missed by all those who had the opportunity to know and work with him.

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# DEVELOPMENT OF THE MANUFACTURING CAPABILITIES OF THE HYDROSTATIC EXTRUSION PROCESS

by

R. J. Fiorentino, W. R. Hansen, B. D. Richardson,  
A. M. Sabroff, and F. W. Boulger

## INTRODUCTION

The purpose of the present research program is to develop the manufacturing capabilities of the hydrostatic extrusion process with the aim of extruding high-quality shapes from materials of interest to the Air Force. It is a continuation of the recently completed program on Contract No. AF 33(600)-43328. The current program is divided into two phases with the following general objectives:

### Phase I. Process-Development Studies

- Part 1. (a) To study the effect of critical process variables on pressure requirements and surface quality in hydrostatic extrusion of AISI 4340 steel, Ti-6Al-4V titanium alloy, and 7075 aluminum alloy.  
  
(b) To correlate all available hydrostatic-extrusion-pressure data with material properties wherever possible in order to assist direction of the experimental effort and maximize the information developed in the present program.
- Part 2. To explore the hydrostatic extrudability of TZM molybdenum alloy (cast and wrought), beryllium, Cb-752 columbium alloy, powder compacts, and other materials to be selected later in the program.
- Part 3. To conduct a design study for high-temperature, high-pressure hydrostatic extrusion tooling based on (1) estimated pressure requirements for high-ratio extrusion of materials of interest to the the Air Force, (2) latest high-pressure-vessel technology, and (3) latest tooling materials available.
- Part 4. To conduct a process economic study on the construction, installation, and operation of equipment with the same operational and size requirements as the tooling developed in the previous program on Contract No. AF 33(600)-43328.

## Phase II. Process-Application Studies

- Part 1. To evaluate the application of the hydrostatic extrusion process for sizing and finishing conventionally hot-extruded (or rolled) structural shapes by various combinations of drawing and extruding. Primary emphasis will be on AISI 4340 steel, although some effort will be devoted to Ti-6Al-4V, 7075-0 aluminum, and selected refractory metals.
- Part 2. To determine the feasibility of producing wire and filaments from beryllium and TZM molybdenum alloy by combinations of hydrostatic extrusion and drawing.
- Part 3. To develop tooling and define process parameters necessary for the reduction of tube blanks to finish tubing from AISI 4340 and a selected columbium alloy.

The study of critical process variables for hydrostatic extrusion (Part 1 of Phase 1) was continued during this report period. Extrusion trials were made both at room temperature and at 500 F as follows:

- (1) Cold hydrostatic extrusion of tubing and T-sections from AISI 4340 and Ti-6Al-4V titanium alloy.
- (2) Warm hydrostatic extrusion of rounds from AISI 4340, Ti-6Al-4V alloy, and beryllium.

Important variables investigated during the interim period included extrusion ratio, billet surface finish, lubricants, and high-temperature fluids.

In addition, initial experiments in the hydrostatic extrusion and drawing of beryllium wire were begun.

The mechanical properties of selected hydrostatic extrusions of 7075-0 aluminum and AISI 4340 steel were determined in this report period.

## **EQUIPMENT AND EXPERIMENTAL PROCEDURE**

### Extrusion Tooling

With one exception, the major components of the hydrostatic extrusion tooling as well as accessory equipment used during this report period were previously described (1,2,3,4,5)\*. A high-temperature, high-pressure gage (HTHP) was designed to measure fluid pressure during trials made at 400 and 500 F. For the most part, the HTHP gage performed within expectations and will be described in the report on the next warm-extrusion trials.

\*References are listed at the end of the text.

## Materials

Specific details on the billet materials used during this interim period are described in Interim Report Numbers I and VI. (1,5)

### Lubricants and Fluids

Table 1 lists billet lubricants used during the last quarterly period. Several of the lubricants listed were previously used for room-temperature hydrostatic extrusion. Several new lubricants-L38, L40, L43, and L44- were selected particularly for extrusion at 500 F. Lubricant L52 was tried in an attempt to eliminate stick-slip during the extrusion of 7075-0 aluminum at slow stem speeds.

Room-temperature trials were made with castor oil as the fluid medium. Table 2 lists fluids used for warm extrusion. All five of the fluids listed in Table 2 were selected because of their good stability and potentially good lubricity at elevated temperatures. At 100 F, the fluids gave a kinematic viscosity range of about 40 to 400 centistokes.

## COLD HYDROSTATIC EXTRUSION OF 7075-0 ALUMINUM ROUNDS

As mentioned in previous reports<sup>(4,5)</sup>, 7075-0 aluminum shows a tendency to crack during conventional hot extrusion; to prevent cracking, exit extrusion speeds are usually limited to one or several feet per minute. With hydrostatic extrusion, sound products of 7075-0 aluminum have been produced at extrusion ratios of 20:1, 40:1, and 60:1, with exit speeds up to about 3000 ipm. However, at relatively low stem speeds, stick-slip occurs during extrusion as a result of momentary lubrication breakdown. This causes high breakthrough-pressure peaks and, sometimes, surface cracking.

A new lubricant, stearyl stearate (L52), was investigated for extrusion of 7075-0 aluminum rounds under conditions known to result in stick-slip. The extrusion conditions used and the results obtained are listed below:

Billet diameter	1-3/4 inches
Extrusion ratio	40:1
Area reduction	97.5 percent
Die angle	45 degrees (included)
Stem speed	20 ipm
Exit speed	1480 ipm
Fluid	Castor oil

TABLE 1. BILLET LUBRICANTS USED FOR HYDROSTATIC EXTRUSION DURING THIS INTERIM REPORT PERIOD

Lubricant	Source	Description	Billet Material Treated
L17	Battelle	20 w/o MoS <sub>2</sub> in castor wax	7075A1
L30	Commercial and Battelle	50 w/o Cindol 4616 in castor wax	Ti-6Al-4V
L31	Commercial	Fluorocarbon telomer	4340 and Be
L33	Battelle	55 w/o MoS <sub>2</sub> and 6 w/o graphite in sodium silicate	4340 and Ti-6Al-4V
L34	Battelle	50 w/o MoS <sub>2</sub> in castor wax	4340
L35	Battelle	20 w/o graphite in castor wax	4340
L38	Commercial	P. T. F. E. lacquer	4340 and Ti-6Al-4V
L40	Commercial	Fluorocarbon-thickened fluorosilicone grease	4340 and Ti-6Al-4V
L43	Battelle	20 w/o MoS <sub>2</sub> in extreme-temperature-range grease	4340 and Ti-6Al-4V
L44	Battelle	20 w/o I <sub>2</sub> in extreme-temperature-range grease	4340 and Ti-6Al-4V
L48	Battelle	20 w/o MoS <sub>2</sub> in castor wax, plus metallic lead, copper flake, and graphite	4340 and 7075A1
L52	Commercial	Stearyl stearate	7075A1

TABLE 2. FLUIDS USED IN WARM HYDROSTATIC EXTRUSION

Fluid Identification	Description	Kinematic Viscosity, centistokes	
		At 100 F	At 500 F
PPE	Mixed isomeric five-ring polyphenyl ether	363	1.2
CBP	Chlorinated biphenyl	44	--
TCP	Tricresyl phosphate	35	--
TAP	Triaryl phosphate	46	--
SE	Silicate ester	6.8	0.88 <sup>(a)</sup>

(a) Viscosity at 400 F.

<u>Trial</u>	<u>Extrusion Pressure, 1000 psi</u>			
	<u>Breakthrough</u>		<u>Runout</u>	
	<u>Stem</u>	<u>Fluid</u>	<u>Stem</u>	<u>Fluid</u>
424	220	191	162	137

The fluid-breakthrough-pressure peak with L52 lubricant was about 5.8 percent higher than that obtained under similar extrusion conditions with L17 lubricant. Also, the extrusion runout was accompanied by severe stick-slip. However, portions of the 75-inch length of extruded rod indicate that sound material with excellent surface finish was obtained. Attempts will be made to modify stearyl stearate lubricant with additions of MoS<sub>2</sub> and/or graphite in order to reduce the breakthrough-pressure peak and to minimize or eliminate stick-slip. As discussed later, extrusion of 7075-0 aluminum tubing with L52 lubricant indicates that further investigation is warranted.

### COLD HYDROSTATIC EXTRUSION OF 7075-0 ALUMINUM AND AISI 4340 TUBING

Extrusion data for trials made to produce tubing from 7075-0 aluminum and AISI 4340 steel are given in Table 3.

#### 7075-0 ALUMINUM

The extrusion trials with this high-strength aluminum alloy were directed toward:

- (1) Further evaluation of lubricants
- (2) Re-extrusion of tubing that had been previously hydrostatically extruded with the aim of obtaining thin-wall tubing.

In the lubrication studies, two new lubricants were evaluated: 20 w/o MoS<sub>2</sub> in castor wax, plus metallic lead, copper flake, and graphite (L48); and stearyl stearate (L52).

Data obtained with those lubricants, given in Table 3, indicate that they compare favorably in performance with L17 (References 4 and 5) in that they produce an excellent surface finish and that their pressures are of the same order.

In preparation for the re-extrusion trials, 7075-0 aluminum was annealed to about 65 Bhn. The billets were prepared by taking a cleaning cut on the billet surface to remove oxide formed during annealing.

TABLE 3. EXPERIMENTAL DATA FOR COLD HYDROSTATIC

Die Angle: 45 Deg  
 Fluid: Castor Oil

Item	Trial	Die Orifice, in.	Billet Diameter, in.		Extrusion Ratio	Wall Thickness, in.	Stem Speed, ipm	Billet Lubricant
			OD	ID				
<u>7075-O</u>								
1	388	1.107	1.750	0.748	3.8	0.180	20	L48
2	425	1.107	1.750	0.748	3.8	0.180	20	L52
3	385(a)	0.875	1.104	0.748	3.2	0.063	20	L17
4	384(a)	0.782	1.104	0.748	12.9	0.017	20	L17
<u>AISI</u>								
5	389	1.107	1.750	0.748	3.8	0.180	20	L48
6	391	1.001	1.750	0.748	5.7	0.125	6	L48
7	386(b)	0.875	1.106	0.748	3.2	0.063	6	L48
8	390(c)	0.875	1.106	0.748	3.2	0.063	20	L48

- (a) Re-extrusion of tubing previously extruded in Trial 351 at a ratio of 3.77:1 and then annealed to 65 Bhn.
- (b) Re-extrusion of tubing previously extruded in Trial 355 at a ratio of 3.77:1 and not annealed.
- (c) Same as (b), except annealed after extrusion.

EXTRUSION OF 7075-O ALUMINUM AND AISI 4340 STEEL TUBING

Billet Surface Finish: 25-100  $\mu$ in.  
 Diameter of Mandrel: 0.7485 in. at top  
 0.7395 in. at bottom

Extrusion Pressure, 1000 psi				Length of Extrusion, in.	Comments
Breakthrough		Runout			
Stem	Fluid	Stem	Fluid		
<u>Aluminum</u>					
49	48	50	48	12-7/8	No $P_b$ peak; uniform $P_r$
51	49	48	47	11	Very slight $P_b$ peak followed by uniform $P_r$
33	30	32	29	6-7/8	Slight $P_b$ peak followed by slight stick-slip
178	161	--	--	4	$P_b$ not reached; stopped at indicated pressure
<u>4340</u>					
162	151	160	149	12-3/4	Slight $P_b$ peak followed by uniform runout
240	209	232	202	5-1/2	Slight $P_b$ peak followed by severe stick-slip
163	149	--	--	1-1/2	$P_b$ not reached; stopped at pressure indicated
164	149	146	129	4-3/4	Moderate $P_b$ peak followed by severe stick-slip

Two extrusion ratios were attempted in re-extruding tubing--3.2:1 and 12.9:1. At the lower ratio (Trial 385), approximately 7 inches of 0.056-inch-thick-wall tubing with excellent surface finish was produced. The cumulative reduction (Trials 351 and 385) in producing the tubing is 91 percent.

In Trial 384, re-extrusion at a ratio of 12.9:1 was attempted. A short length of high-quality 0.22-inch-wall tubing was produced. Here, the cumulative area reduction (Trials 351 and 384) was 97.6 percent. While the fluid-pressure versus ram-travel curve for this trial did not indicate a sharply defined extrusion breakthrough, it did show that a significant displacement of the billet had occurred. Upon removal of the extrusion from the container, it was found that the billet had not only extruded, but had also uniformly upset from about 1.1 to 1.5 inches in diameter. Upsetting apparently occurred because of an additional end load on the billet resulting from the unsupported area of the floating mandrel (see Figure 1). This caused an end pressure on the billet that exceeded the fluid pressure by roughly the compressive flow strength of the billet material. Other factors such as mandrel friction would probably influence the end pressure at which billet upsetting would occur. It can be seen from Figure 1 that the billet-end pressure exceeds the fluid pressure,  $p$ , by

$$\frac{p A_m}{A} ,$$

where

$A_m$  = the mandrel cross-sectional area

$A$  = the billet cross-sectional area.

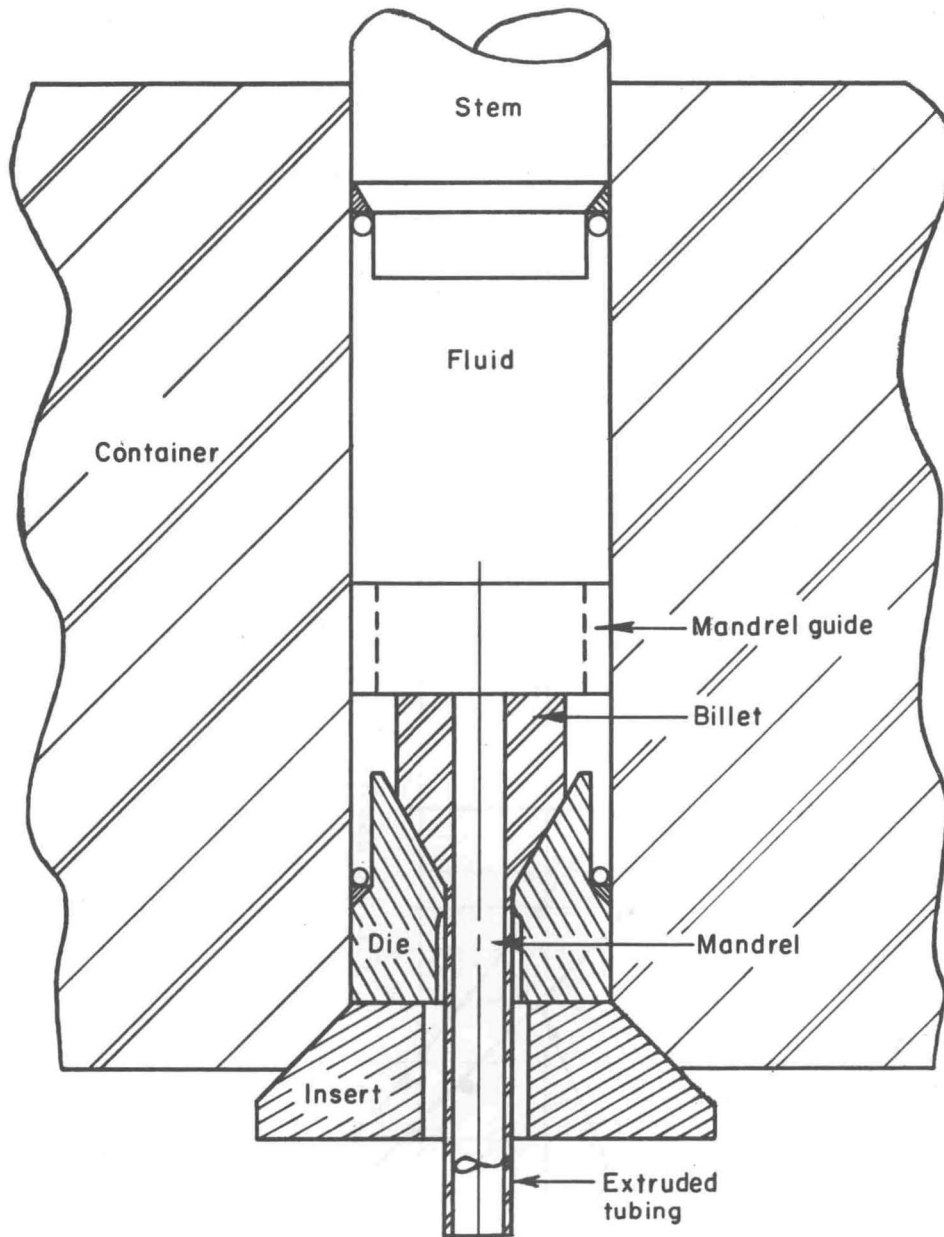
The influence of extrusion ratio on the extrusion runout pressure for 7075-0 aluminum tubing is shown in Figure 2. Two curves are shown. One gives the measured fluid pressure, and the other shows the billet-end pressure, which, in fact, is the "effective" extrusion pressure. Extrapolation of the fluid-pressure curve shown indicates that 7075-0 aluminum tubing can be produced with the present tooling at extrusion ratios up to about 100:1, provided (1) lubrication is adequate at the higher ratios or (2) the unbalanced pressure,  $\frac{p A_m}{A}$ , is not greater than about the compressive yield strength of the material.

#### AISI 4340 STEEL

Table 3 lists the extrusion data obtained in the hydrostatic extrusion of tubing from AISI 4340 steel. In two trials, extrusion ratio and stem speed were investigated.

At an extrusion ratio of 3.8:1, 0.18-inch-thick wall tubing was produced at an exit speed of 140 ipm (Trial 389). At a higher extrusion ratio of 5.7:1 (Trial 391), 0.125-inch-wall tubing was produced at an exit speed of 63 ipm. Further trials will be made in this case, however, in an attempt to eliminate stick-slip during runout.





$$\text{Total pressure on end of billet} = \frac{p(A + A_m)}{A} = p + \frac{p A_m}{A}$$

where

$p$  = fluid pressure  
 $A_m$  = area of mandrel  
 $A$  = area of billet

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FIGURE 1. FLOATING MANDREL ARRANGEMENT FOR HYDROSTATIC EXTRUSION OF TUBING

Analysis Is Given Showing Difference Between Fluid Pressure and Billet End Pressure

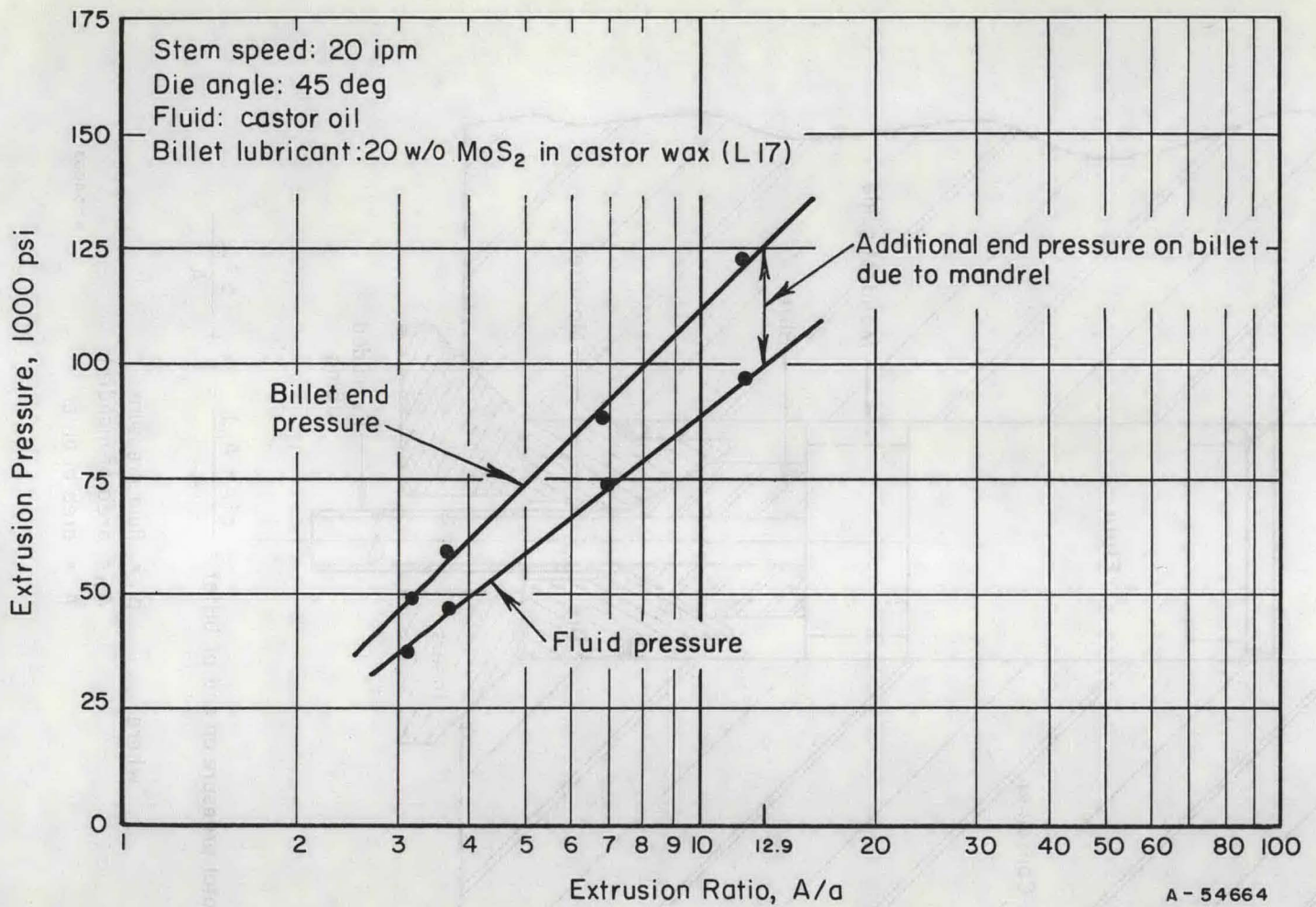


FIGURE 2. EFFECT OF EXTRUSION RATIO ON PRESSURE FOR COLD HYDROSTATIC EXTRUSION OF 7075-0 ALUMINUM TUBING

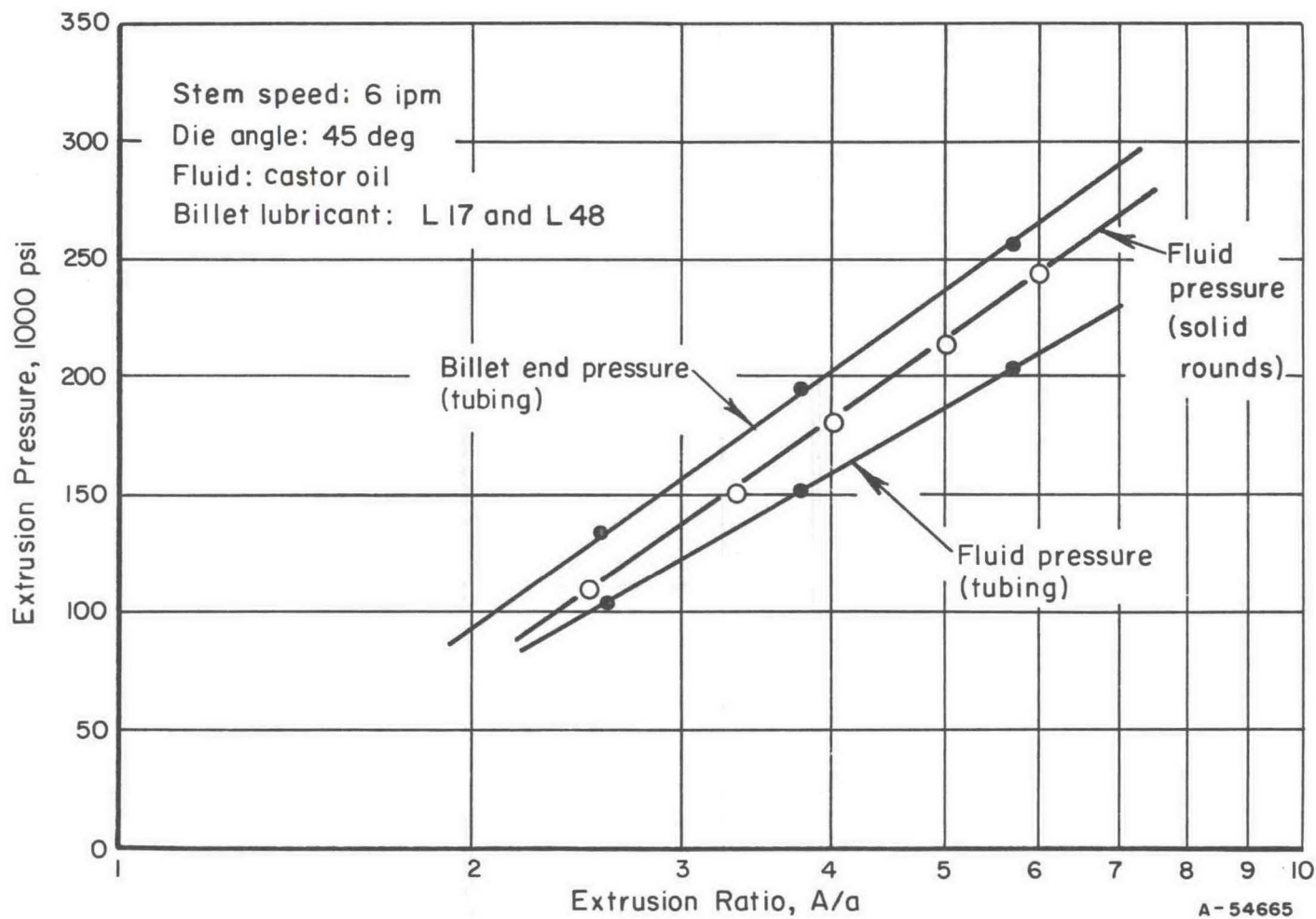


FIGURE 3. EFFECT OF EXTRUSION RATIO ON PRESSURE FOR COLD HYDROSTATIC EXTRUSION OF AISI 4340 STEEL TUBING AND ROUNDS

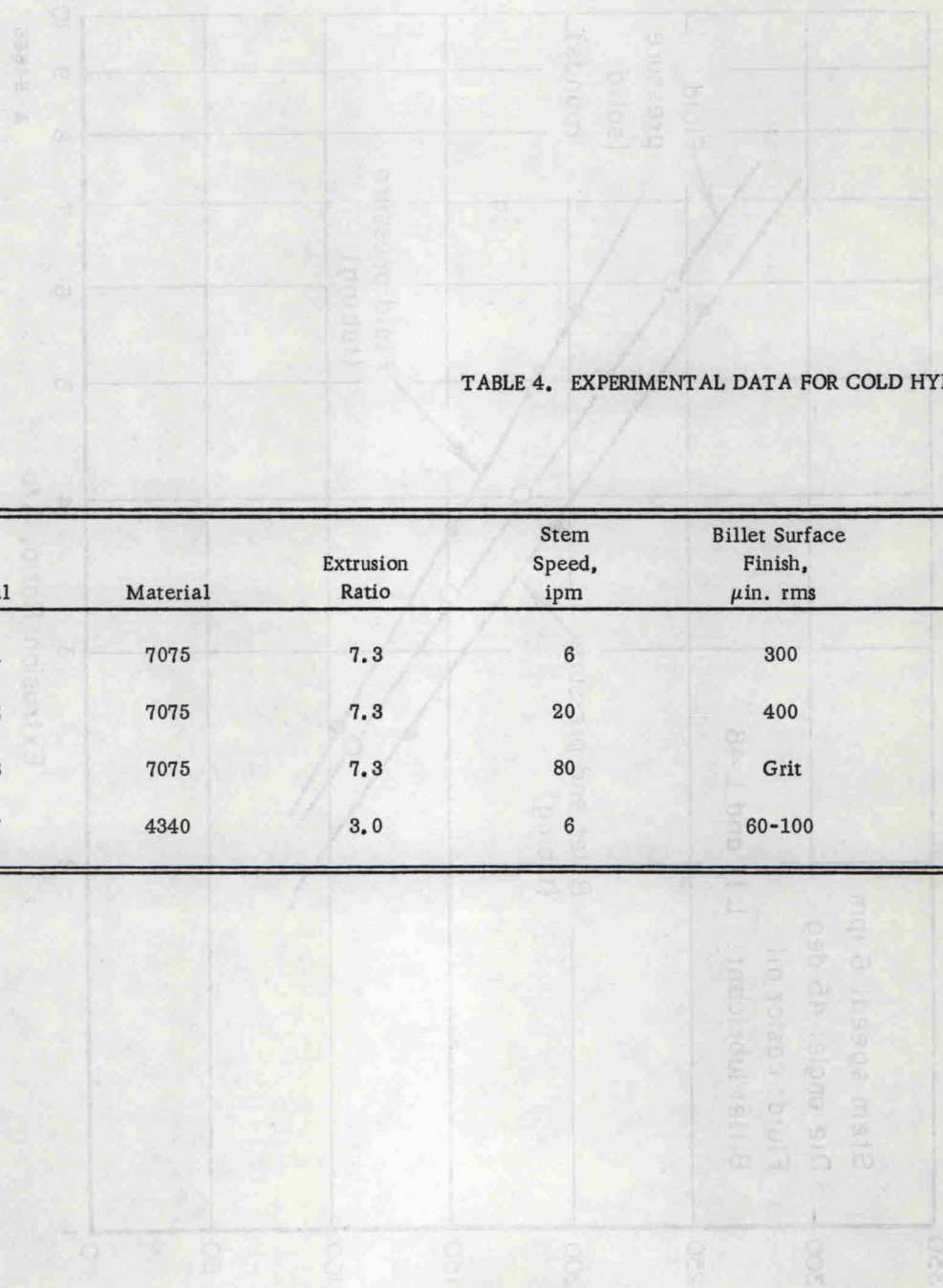


TABLE 4. EXPERIMENTAL DATA FOR COLD HYDROSTATIC

Fluid:  
Die Design:

Item	Trial	Material	Extrusion Ratio	Stem Speed, ipm	Billet Surface Finish, $\mu$ in. rms	Billet Lubricant
1	381	7075	7.3	6	300	L17
2	382	7075	7.3	20	400	L17
3	383	7075	7.3	80	Grit	L17
4	387	4340	3.0	6	60-100	L48

EXTRUSION OF 7075-O ALUMINUM AND AISI 4340 STEEL T-SECTIONS

Castor Oil  
Single-Angle, 45-Deg

Extrusion Pressure, 1000 psi				Length of Extrusion, in.	Comments
Breakthrough		Runout			
Stem	Fluid	Stem	Fluid		
151	135	114	101	20	High $P_b$ peak followed by severe stick-slip
154	142	116	103	14	High $P_b$ peak followed by severe stick-slip
135	126	118	104	11	Moderate $P_b$ peak followed by uniform runout; die cracked
284	244	--	--	--	$P_b$ not reached; stopped at pressure indicated; die cracked

Data from Trial 391 and from previous trials were used to establish the curves in Figure 3. Although the data are concerned with two different lubricants, L17 and L48, past results indicate that pressures required were similar. Again, two curves are shown for tubing, one indicating fluid pressure and the other billet-end pressure. The difference between the billet-end pressure for tubing and the fluid pressure for solid rounds largely represents the friction between the tube and mandrel.

In re-extrusion of AISI 4340 tubing, a 4-3/4-inch length of 0.063-inch-wall tubing (Trial 390) was produced at a ratio of 3.2:1, which represents a cumulative reduction of 92 percent. These results indicate that very-thin-walled steel tubing can be produced successfully by hydrostatic extrusion.

### COLD HYDROSTATIC EXTRUSION OF 7075-0 ALUMINUM AND AISI 4340 STEEL T-SECTIONS

The study of the effect of critical variables in hydrostatic extrusion of 7075-0 aluminum T-sections was continued. Stem speed, billet surface finish, and die design were investigated. The data for these trials are given in Table 4. Sufficient information is now available from these and previous trials to evaluate the above variables. In particular, a comparison may be made of the effect of the single-angle 45-degree die and the compound-angle die (45-degree angle leading into a 160-degree angle) described in Interim Report V. The comparison of data is given in Table 5.

TABLE 5. EFFECT OF T-DIE PROFILES ON FLUID PRESSURES  
FOR 7075-0 ALUMINUM

Fluid: castor oil  
Billet lubricant: L17  
Extrusion ratio = 7.3:1

Trial	Stem Speed, ipm	Billet Surface Finish (Axial), $\mu$ in, rms	Fluid Pressure, 1000 psi	
			Breakthrough	Runout
<u>Single-Angle Die</u>				
381	6	300	135	101
382	20	400	141	103
383	80	Grit	125	104
<u>Compound-Angle Die</u>				
320	6	200	118	106
321	6	Grit	122	110
325	20	Grit	115	109
326	80	130	119	104

It is considered noteworthy that the breakthrough pressures for the compound-angle die are about 10 percent lower than those obtained with the single-angle design. This is particularly significant in view of fact that the machining of the compound-angle die is less expensive and presents fewer technical problems than the single-angle design. Moreover, the compound-angle-die concept is more amenable to segmented construction.

In the study of stem speed, it was found that, with both die designs, a stem speed of 80 ipm eliminated the stick-slip during runout experienced at the lower speeds. Further, the results in Table 5, coupled with previous observations made with solid round extrusions, suggest that the range of billet-surface finishes evaluated had no appreciable effect on pressure requirements.

In a single attempt to extrude an AISI 4340 T-section at a ratio of 3:1, breakthrough was not achieved at 244,000 psi, at which point the trial was stopped. On disassembly, the die was found to be cracked. In view of the pressure reductions obtained with the compound-angle die with 7075-0 aluminum, future trials will be made with this die design. Furthermore, dies of segmented construction will be used with the aim of alleviating the die-cracking problem.

## EVALUATION OF SYSTEM VARIABLES FOR HYDROSTATIC EXTRUSION AT 500 F

Two modifications of the hydrostatic system were made for extrusion at 500 F. These were: (1) replacement of the manganin gage used for cold hydrostatic fluid-pressure measurements, and (2) redesigning of the stem seal. The reason for the modifications and their influence on the results obtained in hot hydrostatic extrusion are discussed below.

### FLUID PRESSURE MEASUREMENT

The resistance of manganin wire is sensitive to small changes in pressure and large changes in temperature, and thus is unsuitable for high-pressure measurement when temperature variations are large. In order to measure the hydrostatic fluid pressure at elevated temperatures, a gage was designed and constructed on this program. Details of the gage will be included in a subsequent report.

For trials carried out during this interim report period, the new gage was calibrated against a manganin gage with both gages at room temperature. The calibration curve obtained with the new gage compared very well with that of the manganin gage from the standpoint of sensitivity and reproducibility. Initial experiments with fluid under pressure at 500 F indicated some hysteresis effects, but these appeared to diminish in later trials, indicating stabilization of the gage at temperature and pressure.

### STEM-SEAL DETAILS

In designing a new stem to incorporate the high-temperature, high-pressure gage, the stem-seal angle (Figure 4) was increased to 65 degrees from 45 degrees with the existing design, with the aim of reducing the stem pressure by decreasing the friction between seal and container. The modification was made as a result of the stem-seal experience of Fuchs<sup>(6)</sup> with this design. In room-temperature calibration trials, it was found that the stem pressure/fluid pressure difference was reduced by approximately 25 percent when using the 65 degree stem-seal angle.

During the warm trials, three O-ring arrangements were used in the investigation as a result of leakage problems. Three combinations were used:

- (1) A single PTFE O-ring
- (2) A PTFE O-ring plus a Buna-N rubber O-ring
- (3) Two PTFE O-rings.



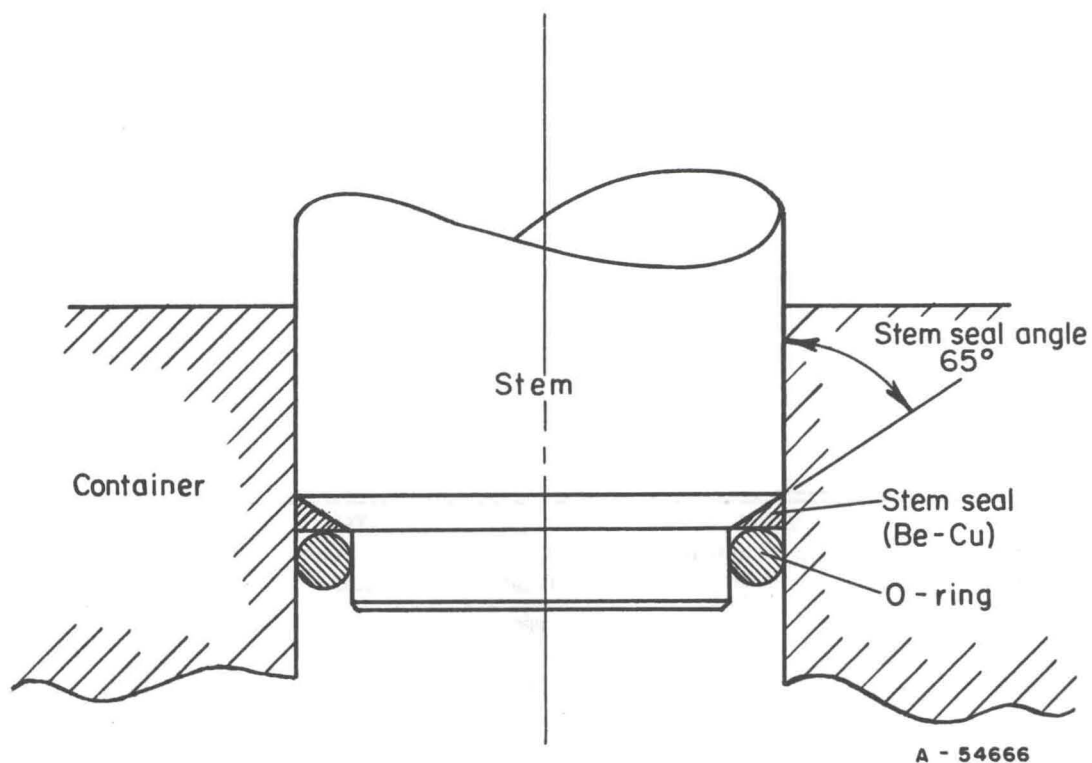


FIGURE 4. STEM-SEAL ARRANGEMENT USED FOR WARM HYDROSTATIC EXTRUSION

With the single PTFE O-ring, fluid leaks were frequent. The other two O-ring arrangements successfully contained fluids at pressures up to about 220,000 psi. In none of the arrangements, however, were the rings reusable, because of distortion or breakage.

In the several trials conducted successfully with a single PTFE O-ring, it is noted that the stem pressure/fluid pressure differences were least of the three combinations. Thus, an increase in stem-seal/container friction is obtained with a dual O-ring system. Because of this, future trials will be made with the aim of eliminating the need for two O-rings.

#### WARM HYDROSTATIC EXTRUSION OF AISI 4340 STEEL ROUNDS

In hydrostatic extrusion of AISI 4340 rounds at 500 F, the variables investigated included fluids, lubricants, and extrusion ratio. In addition, some trials were made at 400 F because one of the fluids had a relatively low flash point (470 F). In the trials, the fluid, billet, and tooling were at the same temperature. Tables 1 and 2 contain information pertaining to lubricants and fluids used. The experimental extrusion data for these trials made with 4340 steel are given in Table 6.

TABLE 6. EXPERIMENTAL DATA FOR 500 F

Die Angle: 45 deg  
Stem Speed: 20 ipm

Item	Trial	Extrusion Ratio	Hydrostatic Fluid	Type of Stem Seal <sup>(a)</sup>	Billet Lubricant	Extrusion Pressure, Breakthrough	
						Stem	Fluid
1	394	4	PPE	1t	L31	198	196
	418	5	PPE	2t	L31	243	213
	420	5	PPE	2t	L31	230	200
2	393	4	PPE	1t	L33	195	195
3	397	4	PPE	1t	L34	187	199
4	409	4	PPE	2t	L35	194	190
5	399	4	PPE	1t	L38	199	204
	401	4	PPE	1t	L38	193	200
6	407	4	PPE	1t 1r	L40	195	189
7	406	4	PPE	1t 1r	L43	202	198
8	408	4	PPE	1t 1r	L44	199	192
9	410	4	TCP	2t	L31	200	187
10	411	4	TAP	2t	L31	202	192
11	412	4	CBP	2t	L31	196	186
12	413	4	SE <sup>(b)</sup>	2t	L31	189	182
	423	4	SE	2t	L33	198	173
	414	5	SE	2t	L31	--	--
	422	5	SE	2t	L31	223	196

(a) 1t = one Teflon O ring on stem; 2t = two Teflon O rings on stem; 1t 1r = one Teflon and one Buna N rubber O-ring on stem.

(b) Because SE fluid flashed at about 470 F, trials with this fluid were made with fluid, tooling, and billet at 400 F.

HYDROSTATIC EXTRUSION OF AISI 4340 STEEL ROUNDS

Billet Surface Finish: 60-120  $\mu\text{in.}$ , rms

1000, psi		Length of Extrusion, in.	Comments
Runout	Fluid		
Stem	Fluid		
197	194	13-1/4	Slight $P_b$ peak; uniform $P_r$
233	206	8	Moderate $P_b$ peak; apparent stick-slip followed by die-seal failure
222	197	14-3/4	Slight $P_b$ peak; uniform $P_r$ followed by stem-seal failure
--	--	13/16	$P_b$ not reached; stopped at indicated pressure
185	197	14-1/4	Very slight $P_b$ peak; uniform $P_r$
190	186	11-3/4	Slight $P_b$ peak; uniform $P_r$
197	203	5	Slight $P_b$ peak; uniform $P_r$ followed by die seal failure
189	197	12-3/4	Slight $P_b$ peak; uniform $P_r$
186	183	9-3/4	Slight $P_b$ peak; slight stick-slip followed by uniform $P_r$
199	194	10	Slight $P_b$ peak; mostly uniform $P_r$
198	191	9-3/8	Very slight $P_b$ peak; very slight stick-slip
200	185	11-1/2	Very slight $P_b$ peak; uniform $P_r$
201	191	11-7/8	Very slight $P_b$ peak; uniform $P_r$
191	181	10-1/8	Moderate $P_b$ peak; uniform $P_r$
189	180	10-1/4	Slight $P_b$ peak; uniform $P_r$
192	170	12-1/2	Slight $P_b$ peak; uniform $P_r$
--	--	2-3/4	Stopped because of stem-seal failure at 216,000 psi fluid pressure
214	193	13-1/8	Slight $P_b$ peak; uniform $P_r$

## Effect of Fluid

Before fluids could be evaluated, preliminary trials were necessary to select an effective billet lubricant. A polyphenyl ether (PPE) fluid was selected because of its reported good high-temperature stability. Based on these trials a "best" billet lubricant was selected (L31), and the other fluids were evaluated. Data listed in Table 7 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 7. EFFECT OF FLUID ON PRESSURES FOR WARM HYDROSTATIC EXTRUSION OF AISI 4340 STEEL

Die angle: 45 deg      Lubricant: L31

Trial	Extrusion Ratio	Extrusion Temperature, F	Fluid(a)	Type of Stem Seal <sup>(b)</sup>	Extrusion Pressure, 1000 psi			
					Breakthrough		Runout	
					Stem	Fluid	Stem	Fluid
394	4.0	500	PPE	1t	198	196	197	194
410	4.0	500	TCP	2t	200	187	200	185
411	4.0	500	TAP	2t	202	192	201	191
412	4.0	500	CBP	2t	196	186	191	181
413	4.0	400	SE	2t	189	182	189	180
418	5.0	500	PPE	2t	243	213	233	206
420	5.0	500	PPE	2t	230	200	222	197
422	5.0	400	SE	2t	223	196	214	193

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

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

### 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 8.

With the exception of L33, all of the lubricants used at 500 F with AISI 4340 and 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 extruded surface finish was exceptionally good in all three cases. However, the other lubricants are considered satisfactory except where criteria such as surface finish are unusually demanding.

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

### Effect of Temperature

The effect of temperature on the stem breakthrough pressures required to extrude AISI 4340 is shown in Figure 5. 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.

Figure 5 shows that the stem breakthrough pressure at room temperature is reduced by 8 to 10 percent at 400 F. The same reduction is achieved in fluid pressure at a ratio of 5:1, but at 4:1 the reduction was only marginal. This is inexplicable, and further trials will be necessary to clarify this finding.

## WARM HYDROSTATIC EXTRUSION OF Ti-6Al-4V TITANITUM-ALLOY ROUNDS

Experimental data for warm hydrostatic extrusion of Ti-6Al-4V alloy rounds are given in Table 9. Variables investigated included fluids, lubricants, stem speed, and extrusion ratio.

TABLE 8. EVALUATION OF LUBRICANTS USED IN EXTRUDING AISI 4340 STEEL AT 500 F

Extrusion ratio = 4:1 Fluid: Polyphenyl ether

Trial	Lubricant	Difference Between Breakthrough and Runout Pressures <sup>(a)</sup> , 1000 psi		Extruded Surface Finish Rating	Type of Extrusion Curve
		Stem	Fluid		
394	L31	1.0	2.5	Excellent	$P_f$ decreasing and then becoming uniform
393	L33	--	--	--	Breakthrough not reached
397	L34	2.0	1.5	Very good	$P_f$ decreasing slightly
409	L35	4.0	4.0	Good; some lubrication breakdown	Uniform $P_f$
399	L38	2.0	1.5	Excellent	Uniform $P_f$
401	L38	3.5	3.0	Excellent	Slightly decreasing $P_f$
407	L40	9.0	5.5	Good; some lubrication breakdown	Slight stick-slip followed by uniform $P_f$
406	L43	3.0	4.0	Good; some lubrication breakdown	Mostly uniform $P_f$
408	L44	1.0	1.5	Good; small amount of lubrication breakdown	Very slight stick-slip

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

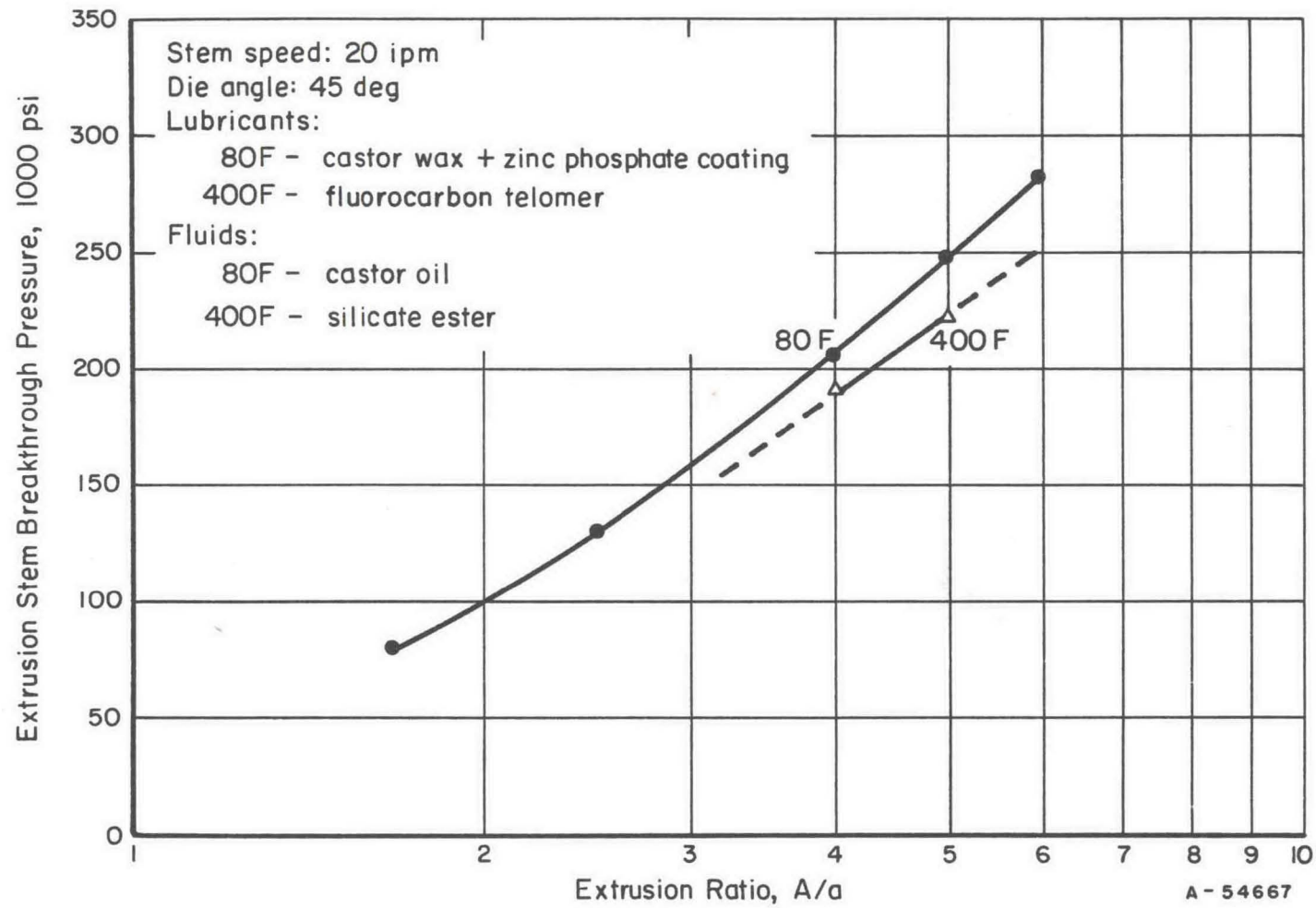


FIGURE 5. COMPARISON OF PRESSURES FOR HYDROSTATIC EXTRUSION OF AISI 4340 STEEL ROUNDS AT TWO TEMPERATURES

TABLE 9. EXPERIMENTAL DATA FOR 500 F

Die Angle: 45 deg

Item	Trial	Extrusion Ratio	Stem Speed, ipm	Hydrostatic Fluid	Type of Stem Seal	Billet Lubricant
1	400	3.3	6	PPE	1t	L30
	402	3.3	6	PPE	1t	L30
2	395	3.3	6	PPE	1t	L33
	396	3.3	20	PPE	1t	L33
	419	4	6	PPE	2t	L33
	421	4	6	PPE	2t	L33
3	398	3.3	6	PPE	1t	L38
4	403	3.3	6	PPE	1t	L40
5	404	3.3	6	PPE	1t1r	L43
6	405	3.3	6	PPE	1t1r	L44
7	415	3.3	6	SE <sup>(b)</sup>	2t	L33
	416	4	6	SE	2t	L33

(a) 1t = one Teflon O ring; 2t = two Teflon O rings; 1t1r = one Teflon and one Buna N rubber O ring.

(b) Because SE fluid flashed at about 470 F, trials with this fluid were made at 400 F.



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

Billet Surface Finish: 60-100  $\mu$ in. rms

Extrusion Pressure C, 1000 psi				Length of Extrusion, in.	Comments
Breakthrough		Runout			
Stem	Fluid	Stem	Fluid		
205	210	--	--	2-1/8	$P_b$ not reached; stopped at indicated pressure
201	199	189	184	2-3/4	High $P_b$ peak followed by severe stick-slip
190	196	185	188	10-1/2	Slight $P_b$ peak; uniform $P_f$ with decreasing pressure toward end
181	192	177	187	11-1/8	Slight $P_b$ peak; uniform $P_f$ with decreasing pressure toward end
225	195	206	180		Moderate $P_b$ peak followed by moderate stick-slip
210	184	201	181		Slight $P_b$ followed by slight stick-slip
175	185	170	182	8-1/2	Slight $P_b$ peak; uniform $P_f$ increasing toward end
211	213	--	--	2-7/16	$P_b$ not reached; stopped at indicated pressure
191	182	188	181	2	Slight $P_b$ peak followed by severe stick-slip
226	216	--	--	2-1/4	$P_b$ not reached; stopped at indicated pressure
178	170	177	168	8	$P_b$ peak; uniform $P_f$ decreasing toward end
212	198	206	194	8-1/4	Slight $P_b$ peak; uniform $P_f$ decreasing toward end

### Effect of Fluid

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 9 indicates that at 3.3:1 the SE fluid reduces stem pressures on the order of 5 percent. However, at 4:1 there is 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.

### Effect of Lubricant

The results obtained in studies with several lubricants, which are described in detail in Table 1, are given in Table 9. No special billet coatings were applied before lubrication.

One of the most significant findings is that Lubricant L33 (55 w/o MoS<sub>2</sub> and 6 w/o 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. 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. In fact, even the most satisfactory coating (anodized coating C3), gave rise to high breakthrough peaks, although it did eliminate stick-slip during runout. The elimination of such billet coatings simplifies the lubrication process, and thus would effect appreciable cost savings in a production operation. Furthermore, the surface finish of all extrusions made with L33 could be rated as "very good" to "excellent". Machining marks carried through from billet to extrusion give additional evidence of the effectiveness of L33 lubricant.

PTFE lacquer (L38) 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 two temperatures are plotted against extrusion ratio in Figure 6. Curves A and B represent the pressure-extrusion ratio relationships at room temperature for two lubrication systems. (The lower pressures shown in Curve B are a result of improved lubrication developed recently.) From Curve C, it is seen that 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.

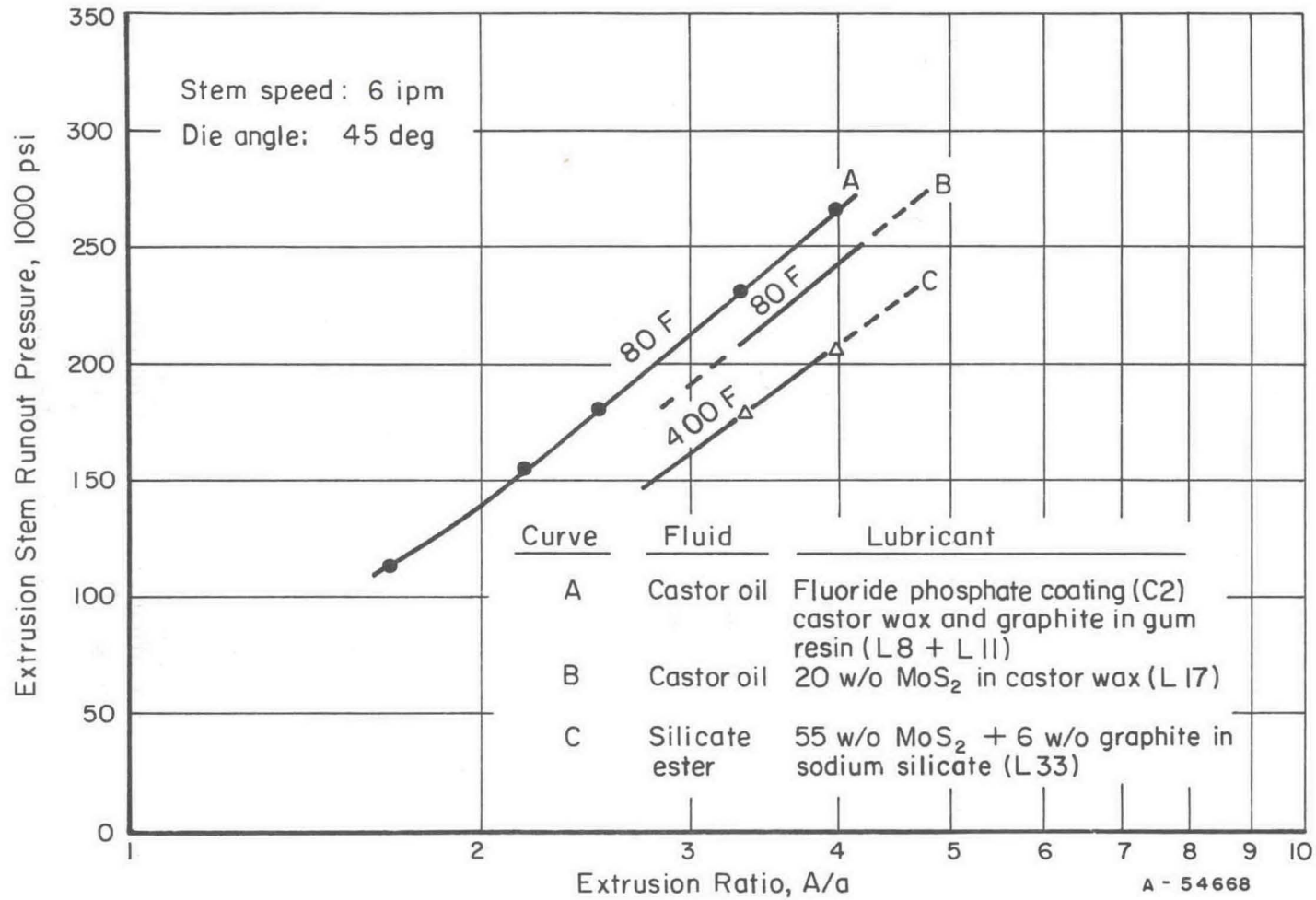


FIGURE 6. COMPARISON OF PRESSURES FOR THE HYDROSTATIC EXTRUSION OF Ti-6Al-4V TITANIUM-ALLOY ROUNDS AT TWO TEMPERATURES

## WARM EXTRUSION OF BERYLLIUM ROUNDS

A single trial was made to extrude a beryllium round at 500 F. The extrusion conditions and experimental data obtained are listed below.

Trial	417
Billet diameter	1-3/4 inches
Extrusion ratio	2.5:1
Area reduction	60 percent
Stem speed	6 ipm
Die angle	45 degrees
Lubricant	L31
Fluid	Polyphenyl ether
Stem seal	2 PTFE O-rings

Although an extruded product 4-1/2 inches long was produced, neither a well-defined breakthrough nor runout pressure was achieved. Comparing the fluid-compressibility curves with other trials using the same fluid, it appears that extrusion started at approximately 65,000 psi (less than half the pressures required to extrude at room temperature) and continued to extrude up to a pressure of 124,000 psi, when the trial was stopped. On examination of the extrusion and die, it was found that lubrication breakdown had occurred, resulting in severe galling on one side of the die land. The die used here was designed to effect a gradual release of the elastic stresses present in the extrusion on exiting from the die land.

The extruded product exhibited less cracking than that when beryllium was extruded at room temperature. These results show considerable promise for the warm extrusion of beryllium, and further warm trials will be conducted with new die designs and improved lubricants, and at higher ratios.

### Hydrostatic Extrusion and Drawing of Beryllium Wire

The aim of this portion of the program is to determine the technical feasibility of producing beryllium wire down to a target diameter of 0.001 inch by hydrostatic extrusion and drawing. In this Battelle-developed process, the wire is subjected to hydrostatic fluid pressure on the entry side of the die and draw stress on the exit side.

Equipment has been designed and assembled to provide control over the draw stress and exit velocity of the extruded wire. This is accomplished with a variable-speed drive motor through an electromagnetic torque limiter. The output shaft, which carries a 2.8-inch-diameter coiling reel has a speed range of 0 to 50 rpm and a stepless control of torque in the range of 2 to 12 in.-lb. For a starting wire diameter of 0.020 inch, the expected draw load at the first reduction would be about 5 pounds. A load-measuring device has been designed and calibrated to monitor the draw load. It is able to measure draw loads as low as 0.1 pound.

Consideration has been given to the method by which the wire within the container is uncoiled. Two methods are being evaluated:

- (1) A horizontal-axis spool mounted in a ball race
- (2) A freely suspended coil on a vertical axis.

The wire to be used in the experiments has been purchased from the following two sources:

- (1) Brush Beryllium Company annealed wire originating from powder-metallurgy material
- (2) The Beryllium Corporation cast-ingot wire as drawn with a nickel sheath (as used in production wire drawing) left intact
- (3) Same as (2), but with the nickel sheath removed, followed by annealing.

Chemical pointing of the wire, nominally 0.020 inch in diameter, was readily achieved in a bath of 50 percent ammonium bifluoride ( $\text{NH}_4\text{HF}_2$ ) solution under a ventilated hood.

The Brush Beryllium Company has been subcontracted in this program to provide evaluation services on the beryllium wire in all stages of the work. The characterization of the wire to be used in the experiments by room-temperature tensile testing and metallographic examination has been completed and will be included in a subsequent report.

Preliminary trials were made with soft copper wire and Nichrome A to evaluate the load-measuring device, the coiling device, and the uncoiling method. These trials showed the most promising procedure of uncoiling to be the vertical-free-coil method. The trials also showed that the load-measuring and coiling devices performed satisfactorily.

In the initial trials with beryllium wire, the pressure requirements were found to be excessive, and the experiments were stopped before any wire was drawn. Subsequent examination of the die-angle profiles indicated that the entry angle was much smaller than specified, which undoubtedly contributed to the high pressure requirements. The die profiles are to be modified by the vendor.

#### TENSILE PROPERTIES OF AISI 4340 STEEL AND 7075-0 ALUMINUM HYDROSTATIC EXTRUSIONS

The results of tensile tests on AISI 4340 steel from extrusions made at 20 and 80 ipm are recorded in Table 10. Extruding at a 5:1 ratio tripled the yield strength and doubled the ultimate strength compared with the as-received billet.

The table shows that increasing exit speeds have little effect on tensile or yield strengths, but may actually improve ductility as measured by elongation.

Tensile data for 7075-0 aluminum extrusions produced at extrusion ratios of 20:1, 40:1, and 60:1 are also listed in Table 10. The yield and tensile strengths of the

material in the annealed condition were almost tripled and doubled, respectively, by extrusion at ratios up to 60:1 without any appreciable sacrifice in ductility. Exit speed does not appear to influence mechanical properties at an extrusion ratio of 20:1.

TABLE 10. ROOM-TEMPERATURE TENSILE PROPERTIES OF AISI 4340 STEEL AND 7075 ALUMINUM ROUNDS PRODUCED BY HYDROSTATIC EXTRUSION

Extrusion Ratio	Reduction in Area of Extrusion, percent	Trial	Speed, ipm		Ultimate Tensile Strength, psi	Yield Strength (0.2% Offset), psi	Reduction in Area in Tension, percent	Elongation in 2 Inches, percent
			Stem	Exit				
<u>AISI 4340 Steel</u>								
5.0	80	167	1	10.0	188.6	163.4	27.9	8.0(a)
5.0	80	189	6	60.0	180.4	151.9	27.8	9.5(a)
5.0	80	315	20	185	179.0	161.7	28.8	13.0
5.0	80	340	80	740	178.8	160.9	29.8	11.5
1	0	As-received bar stock			94.6	55.4	49.0	33.0(a)
<u>7075 Aluminum</u>								
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

(a) Percent elongation in 1 inch.

#### FUTURE WORK

During the next interim-report period it is expected that work will be continued on the extrusion of shapes, and also, of beryllium rounds and wire. Also, work will commence on the extrusion of TZM molybdenum alloy. In addition, efforts will be made to obtain and prepare refractory alloys and powder products included in the program for extrusion.

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