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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 (IX)

March 1967

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DEVELOPMENT OF THE MANUFACTURING CAPABILITIES
OF THE HYDROSTATIC EXTRUSION PROCESS

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ABSTRACT

Development of the Manufacturing Capabilities of the Hydrostatic Extrusion Process

R. J. Fiorentino
et al.
Battelle Memorial Institute

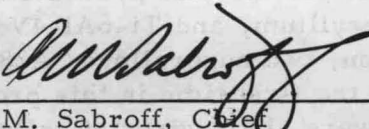
The purpose of the present program is to develop the manufacturing capabilities of the hydrostatic extrusion process. Specific applications being studied are fabrication of wire, tubing, and shapes from relatively difficult-to-work materials such as refractory-metal alloys, high-strength steels and aluminum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

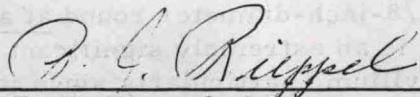
Investigation of critical process variables for the cold hydrostatic extrusion of 7075-0 aluminum, TZM molybdenum alloy, beryllium, and Ti-6Al-4V titanium alloy was continued during this report period. In addition, two superalloys, A286 (iron-base) and Alloy 718 (nickel-base), were investigated for the first time in this program. With the exception of 7075 aluminum, these materials were also hydrostatically extruded at 400 to 500 F. Further work on the extrusion of beryllium wire is also reported. Important developments in the program are given below:

- (1) Beryllium was cold extruded into a 7/8-inch-diameter round at a ratio of 4:1 virtually free of cracks. This is an extremely significant advancement in the cold working of beryllium, particularly since it was achieved using Battelle's double-reduction die concept and without the need of an expensive, fluid counter-pressure system.
- (2) TZM molybdenum alloy was also cold extruded at 4:1 without cracks, using the double-reduction die.
- (3) Two superalloys, A286 and Alloy 718, were cold extruded without cracking through a die of standard design. The maximum extrusion ratios achievable within the 250,000 psi pressure capacity of the tooling were 5:1 and 3.3:1, respectively.
- (4) Fluid pressures to extrude beryllium rounds at 500 F were 1/3 lower than those required at room temperature.
- (5) Two samples of 0.020-inch-diameter beryllium wire of ingot origin were reduced 60 percent at 500 F to 0.0124-inch diameter in a single pass by Battelle's process of hydrostatic extrusion-drawing.
- (6) In further evaluation of the compound-angle billet nose, 7075-0 aluminum extrusions were produced at a ratio of 60:1 without stick-slip.
- (7) 7075-0 aluminum T-sections, 1/4-inch thick, were re-extruded into 1/8-inch and 1/16-inch thick T-sections at ratios of 2:1 and 4:1, respectively.

- (8) A sintered, dispersion-hardened aluminum alloy was readily cold extruded at ratios of 10:1 and 20:1 into substantially sound products.
- (9) Stick-slip was completely eliminated during both cold and 500 F extrusion of Ti-6Al-4V at a ratio of 4:1. The surface finish was excellent.
- (10) Two more specimens of high-quality Ti-6Al-4V tubing, 0.663-inch OD and 0.030-inch wall, were produced in a single-pass reduction of 60 percent at room temperature.
- (11) High-density compacts of Ti-6Al-4V powder were made at hydrostatic pressures of 60,000 psi and 225,000 psi for subsequent hydrostatic extrusion.

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 December 1966 through 28 February 1967. 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 contributing to the program are Mr. B. D. Richardson, Research Metallurgical Engineer, Mr. G. E. Meyer, 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. Data from which this report has been prepared are contained in Battelle Laboratory Record Books Nos. 21799, 21990, 23055, 23287, 23585, 23791, 23836, and 24446.

TABLE OF CONTENTS

	<u>Page</u>
INTRODUCTION	1
EQUIPMENT AND EXPERIMENTAL PROCEDURE	3
Extrusion Tooling	3
Materials	3
Lubricants, Coatings, and Fluids	4
COLD HYDROSTATIC EXTRUSION OF 7075-0 AND DISPERSION-HARDENED SINTERED ALUMINUM	5
7075-0 Aluminum Rounds	5
Extrusion and Re-extrusion of 7075-0 T-Sections	7
Dispersion-Hardened Sintered Aluminum.	7
HYDROSTATIC EXTRUSION AND COMPACTION OF Ti-6Al-4V TITANIUM ALLOYS	10
Rounds	10
Tubing	12
Compaction	12
HYDROSTATIC EXTRUSION OF WROUGHT TZM MOLYBDENUM ALLOY AND BERYLLIUM ROUNDS	13
HYDROSTATIC EXTRUSION OF SUPERALLOYS ALLOY 718 AND A286	21
HYDROSTATIC EXTRUSION AND DRAWING OF BERYLLIUM WIRE	21
REFERENCES	23

LIST OF TABLES

	<u>Page</u>
Table 1. Billet Lubricants Used for Hydrostatic Extrusion During This Report Period	5
Table 2. Experimental Data for Cold Hydrostatic Extrusion of 7075-0 Aluminum and Dispersion-Hardened Aluminum	9
Table 3. Experimental Data for Hydrostatic Extrusion of Ti-6Al-4V Titanium Alloy.	11
Table 4. Experimental Data for Hydrostatic Extrusion of TZM Molybdenum Alloy and Beryllium	16
Table 5. Experimental Data for Hydrostatic Extrusion of Superalloys	22

LIST OF FIGURES

Figure 1. Classification of Pressure - Displacement Curves Obtained in Hydrostatic Extrusion	26
Figure 2. Multipiece T-Section Dies Evaluated in Program	8
Figure 3. Standard Die Profile and Two Dies Designed to Eliminate Cracking in Brittle Materials.	15
Figure 4. Influence of Double Reduction Die on Cracking of Hydrostatic Extrusions of Wrought TZM Molybdenum Alloy	17
Figure 5. Influence of Die Design on Cracking in Hydrostatic Extrusions of Beryllium	19
Figure 6. Photomicrographs of Beryllium Cold Extruded at a Ratio of 4:1 by Hydrostatic Extrusion Without Fluid Counterpressure	20

DEVELOPMENT OF THE MANUFACTURING CAPABILITIES OF THE HYDROSTATIC EXTRUSION PROCESS

by

R. J. Fiorentino, B. D. Richardson, G. E. Meyer,
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 previous 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, beryllium, A286 iron-base superalloy, Alloy 718 nickel-base superalloy, powder compacts, and other selected materials.
- 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 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, TZM molybdenum alloy, and Ti-6Al-4V titanium 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 steel, 7075-0 aluminum, and Ti-6Al-4V titanium.

In addition to the continuing study of critical process variables, experimental efforts during the last quarter were directed toward extrusion of advanced materials, extrusion of tubing, sizing of shapes, fabrication of wire, and compaction of powders. The specific areas covered with each material are given below:

<u>Material</u>	<u>Parameters Evaluated</u>
7075-0 Aluminum	Lubricants Billet nose design Extrusion ratio T-sections Re-extrusion of T-sections
Dispersion-Hardened Aluminum	Extrusion ratio
Ti-6Al-4V Titanium Alloy	Stem speed Lubricant coating at 400 F T-section Powder compaction
TZM Molybdenum Alloy and Beryllium	Die design 500 F extrusion
Beryllium Wire	Die design 500 F extrusion
Superalloys Alloy 718 and A286	Extrusion ratio 500 F extrusion

EQUIPMENT AND EXPERIMENTAL PROCEDURE

Extrusion Tooling

The major components of the hydrostatic-extrusion tooling used during this report period were previously described. (1, 2, 3, 4, 5, 6)* Modifications in die-orifice design are discussed in the appropriate section of this report. The stem-seal arrangement used in the 400 and 500 F trials was described in Interim Report VII. (5)

The die-seal arrangement, described in Interim Report VIII(6) which consisted of a single O-ring located in the base of the die, was evaluated with several dies. At room temperature and at pressures of 240,000 psi, no leaks occurred with this seal arrangement. Sealing was not achieved at 500 F with a PTFE O-ring. Other O-ring materials will be evaluated for use at this temperature.

All billets are 1-3/4 inch in diameter x 6 inches long plus a 45-degree conical nose, unless otherwise noted.

Materials

A number of new materials were extruded during this report period. Description of these materials is as follows:

(1) Dispersion-hardened sintered-aluminum-product (SAP)

- (a) Composition: 99.999% pure aluminum plus a 6 wt % dispersion of Al_2O_3 .
- (b) Condition: 80 to 85 percent of theoretical density and yield strength of 35,000 psi.
(Supplied by Oak Ridge National Laboratory, Oak Ridge, Tennessee, and produced on AEC activity No. 0440-02041.)

(2) Ti-6Al-4V prealloyed powder

- (a) Composition as supplied by the vendor:

Ti	Bal	Fe	900 ppm	Mn	11 ppm
Al	6%	C	200 ppm	W	10 ppm
V	4%	B	<0.1 ppm	Cu	10 ppm
H	50 ppm	Co	<0.2 ppm	Si	68 ppm
N	60 ppm	Cd	<0.2 ppm	Hf	60 ppm
O	1800 ppm				

*References are listed at the end of the text.

(b) Condition: The powder was characterized as having 90 percent of the particles between -100 and +325 mesh and the balance of the material at -325 mesh.

(Supplied by Penn Nuclear Company, Penn, Pennsylvania)

(3) Alloy 718

(a) Composition as provided by the manufacturer:

<u>Max %/Min %</u>		<u>Max %/Min %</u>		<u>Max %/Min %</u>	
C	0.10/0.03	Cr	21.0/17.0	Ti	1.15/0.65
Mn	0.35/ --	Ni	55.0/50.0	Al	0.80/ --
Si	0.35/ --	Co	1.0/ --	B	0.006/ --
P	0.015/ --	Cb+Ta	5.50/5.00	Cu	0.10/ --
S	0.015/ --	Mo	3.30/2.80	Fe	Bal

(b) Condition: Material was received in the form of hot-worked bar stock in the solution-treated condition; hardness was 16 R_C.

(Supplied by Latrobe Steel Company, Latrobe, Pennsylvania).

(4) A-286

(a) Composition as provided by the manufacturer:

<u>C</u>	<u>Mn</u>	<u>Si</u>	<u>Cr</u>	<u>Ni</u>	<u>Mo</u>	<u>Ti</u>	<u>Al</u>	<u>B</u>	<u>Fe</u>	<u>V</u>
0.05	1.40	0.40	15.00	26.00	1.25	2.15	0.20	0.003	54.00	0.30

(All values are in percentages.)

(b) Condition: The material was received in the form of hot-worked bar stock in the solution-treated condition; hardness was <10 R_C.

(Supplied by Allegheny Ludlum Steel Corporation, Pittsburgh, Pennsylvania).

The remaining materials evaluated during this report period were detailed in Interim Reports Nos. I, VI, and VIII. (1, 5, 6)

Lubricants, Coatings, and Fluids

Table 1 lists the billet lubricants used in this report period. No new lubricants were evaluated but investigation of stearyl stearate (L52) with 7075 aluminum was continued at higher ratios. Lubricant L38 (PTFE) was the billet lubricant used with all of the TZM, beryllium, and superalloy billet materials.

Coating C3 was applied to all the Ti-6Al-4V titanium billets before lubrication. It was evaluated for the first time at 400 F.

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

Lubricant	Source	Description	Billet Material Traced
L17	Battelle	20 wt% MoS ₂ in castor wax	Ti-6Al-4V
L33	Battelle	55 wt% MoS ₂ and 6 wt% graphite in sodium silicate	Ti-6Al-4V (400 F trial)
L38	Commercial	PTFE	Be, TZM, Alloy 718, A286
L52	Commercial	Stearyl stearate	7075-0 Al
L53	Commercial and Battelle	20 wt% MoS ₂ in stearyl stearate	7075-Al, dispersion-hardened Al

Castor oil was the fluid medium for all the room-temperature trials. In the elevated-temperature trials, silicate ester (SE) was used at 400 F and polyphenyl ether (PPE) at 500 F.

The effectiveness of lubrication systems is judged by their effect on the fluid pressure vs. displacement curve and the extruded-product finish. Figure 1 shows the classifications of the types of curves generally obtained and which were described in detail in Interim Report VIII⁽⁶⁾. For easy reference Figure 1 is placed on a fold-out page at the end of the text.

COLD HYDROSTATIC EXTRUSION OF 7075-0 AND DISPERSION-HARDENED SINTERED ALUMINUM

The experimental data obtained in the hydrostatic extrusion of 7075-0 Al and a dispersion-hardened sintered-aluminum alloy are contained in Table 2.

7075-0 Aluminum Rounds

The compound billet nose, which was described in Interim Report VIII⁽⁶⁾, was used at extrusion ratios of 40, 60, and 200:1. This nose design consists of a dual-angle cone having an apex angle of 45 degrees up to a predetermined diameter, A, followed by a reduced angle of 30 degrees. (The standard billet nose is a single-angle cone of 45 degrees.) Above ratios of 20:1 with 7075-0 aluminum the compound angle nose was found to be necessary to aid in achieving thick-film lubrication on breakthrough and thereby prevent stick-slip⁽⁶⁾. Stick-slip always occurred at ratios of 40:1 and above when the standard nose was used.

Lubricant 52 (stearyl stearate) was evaluated at a ratio of 40:1 using the compound billet nose. The aim of this trial (473) was to determine how effective the 20 wt % MoS₂ additive to stearyl stearate (L53) had been in a previously reported trial (Trial 470 in Table 2). A comparison of the results indicates that while the overall pressure levels were a little higher for the plain stearyl stearate, the smooth runout pressures indicated good lubrication and the surface finish of the product was excellent.

The compound billet nose and L52 also provided smooth runout conditions at a ratio of 60:1 (Trial 474), and stem speed of 20 ipm, conditions which have always resulted in stick-slip when the standard nose was used. Here again the product surface finish was excellent and was crack free.

An attempt was made to extrude 7075-0 Al at a ratio of 200:1 (Trial 504). The stem speed was 6 ipm and the billet lubricant was L53. In addition, a compound nose was used on the billet. Seizing between billet and die took place at a fluid pressure of 184,000 psi, when the extruded material leaving the die represented an extrusion ratio of 22:1. At this point, however, the extrusion ratio being attempted at the die entry cone was 150:1 (as measured on the unextruded part of the compound angle nose). The breakdown of lubrication at the billet-die interface which apparently occurred will be investigated further in future trials at these high ratios.

A single trial (Trial 472) was conducted to evaluate the effect of stopping and restarting on pressure requirements during extrusion of a single billet. This was done because, in previous tandem extrusion trials⁽⁶⁾, restarting extrusion after stopping to load a tandem billet required unduly high breakthrough pressures and resulted in severe stick-slip, although no P_b peak or stick-slip was encountered initially. It was thought that this might have been partly due to disturbing the first billet in the die while loading the tandem billet. However, Trial 472 shows that, on restarting 10 seconds after stopping, similar high breakthrough pressures and stick-slip were obtained.

This behavior is probably mainly due to lubrication breakdown although the precise mechanism is not clear. One contributing factor may be the temperature increase of the lubrication system (fluid + billet lubricant) developed at the billet-die interface during initial extrusion. Simultaneously, the lubrication system is subjected to the fluid pressure required for extrusion and the viscosity of the system is apparently still sufficient to prevent a high P_b and stick-slip. On depressurization of the fluid, however, the residual temperature at the billet-die interface may still be high enough to effect a sharp viscosity drop and/or an unfavorable chemical change in the lubrication system. Thus, mere repressurization of the fluid may not necessarily renew the same state of lubrication existing during initial extrusion. Other contributing factors may be that:

- (1) The change in the surface characteristics of the billet nose may have reduced the contribution of "squeeze" lubrication during re-extrusion.
- (2) Sufficient work hardening of the billet nose occurred, in spite of adiabatic heating, that higher pressures were required for re-extrusion.

Extrusion and Re-extrusion of 7075-0 Aluminum T-Sections

Extrusion of a T-section from a round billet at a ratio of 7.3:1 was made (Trial 488) to evaluate the effectiveness of a three-piece T-die design consisting of a die insert, a conical shell insert, and a die case. Details of the design are shown in Figure 2a. The entry-angle configuration was similar to the compound-angle T-die design reported previously⁽⁴⁾. The die insert is sized for a hand press-fit into the die case. The die operated satisfactorily and this design will be used for future extrusion of shapes. Its main advantage is that a worn die or shell insert would be cheaper to replace than the whole die itself. It is also believed to be possible for a single-piece die insert to be used in the cracked condition.

Efforts were also directed toward re-extrusion of T-sections previously hydrostatically extruded on the program. The potential advantage of this operation would be to avoid the costly operation of conventional drawing of previously extruded or rolled-shapes to finish dimensions.

During the past quarter, 1/4-inch-thick T-sections (produced earlier in the program) were extruded into 1/8-inch and 1/16-inch thick T-sections at ratios of 2:1 and 4:1, respectively. The results are listed in Table 2.

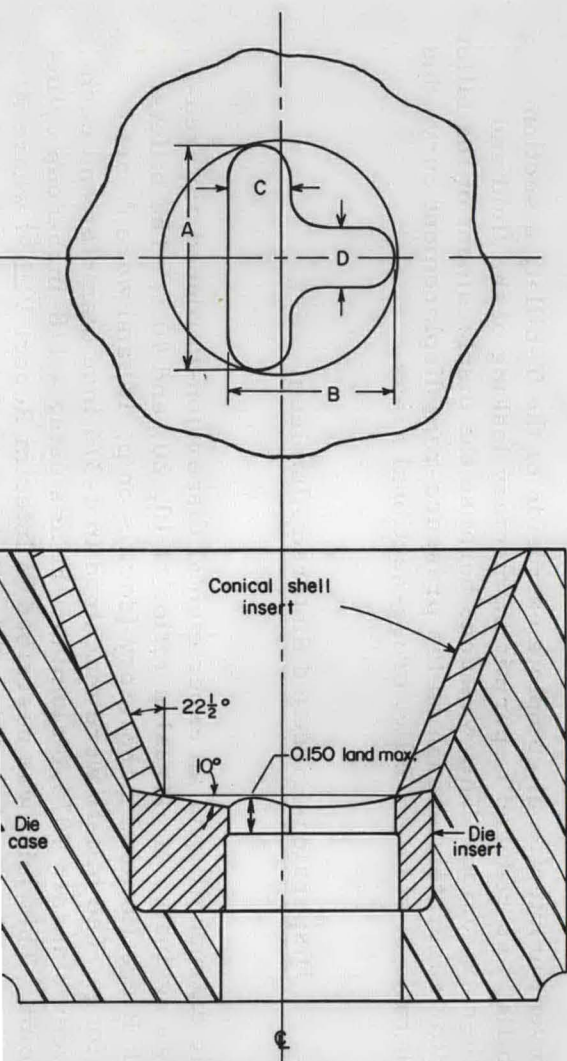
A shaped nose was machined on each 1/4-inch T-billet to provide a fluid seal with the die. It was intended to shape the nose to conform with the die-entry angle, but this proved to be difficult. Consequently the billet nose-die seals failed at low fluid pressures. In one case, Trial 482, the nose-die seal was good up to 34,500 psi and some extrusion began before any fluid leaked.

The sealing problem was solved by casting a Wood's alloy plug around the billet nose while partially inserted in the die orifice. In Trials 507 and 489, this seal was used successfully to extrude a 1/4-inch-thick T-billet to 1/8-inch and 1/16-inch-thick T-sections, respectively.

In order to prevent unintentional complete extrusion of the T-billet, a section near the tail of the billet was reduced to permit momentary leaking of the fluid and thereby prevent further extrusion. This was done because the displacement of the billet was difficult to monitor accurately enough on the pressure-ram displacement curve due to the relatively high ratio of stem-to-billet cross-sectional areas.

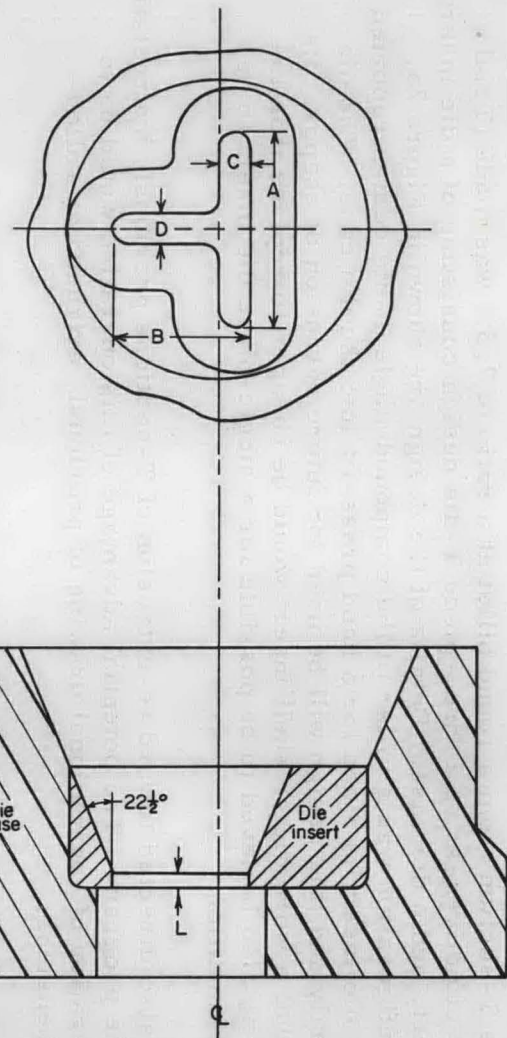
Dispersion-Hardened Sintered Aluminum

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 (details on p. 12) and were 2 inch diameter x 2 inches long. 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 was described in Interim Report VIII⁽⁶⁾ where it was used in tandem-extrusion investigations. The sandwich-billet construction was used because sintered-aluminum billets were too small to machine a 45-degree nose on one end, and it also permitted complete extrusion of the billets. Lubricant 53 was applied



Round-to-T-Section Extrusion Die

A 0.38 in. B 0.688 in. C 0.250 in. D 0.250 in.



b. T-Section Re-extrusion Die

Nominal Web Dimension, in.	A	B	C	D	L
$\frac{1}{8}$	0.875 in.	0.625 in.	0.125 in.	0.125 in.	0.025 in.
$\frac{1}{16}$	0.842 in.	0.593 in.	0.062 in.	0.062 in.	0.020 in.

FIGURE 2. MULTIPIECE T-SECTION DIES EVALUATED IN PROGRAM

TABLE 2. EXPERIMENTAL DATA FOR COLD HYDROSTATIC EXTRUSION OF 7075-0 ALUMINUM AND DISPERSION-HARDENED ALUMINUM

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

Billet surface finish - 60-100 microinches, rms

Trial	Objective or Variable	Extrusion Ratio	Stem Speed, ipm	Billet Nose Design ^(a)	Billet Lubricant	Extrusion Pressure, 1000 psi				Type of Curve, p 25	Length of Extrusion, inch	Comments	
						Breakthrough		Runout					
						Stem	Fluid	Stem	Fluid				
<u>7075-0 Aluminum Rounds</u>													
472	Stop-start investigation	20	20	S	L53	156	135	138	121	B2	65	Stopped after partial extrusion, Restarted extrusion after 10 sec Interim Report VIII	
						211	172	--	--	D2			
470	Reference trial	40	20	C, A=0.75 in.	L53	168	150	154	137	C2			140
473	Lubricant	40	20	C, A=0.75 in.	L52	177	157	149	143	B2			120
474	Ratio	60	20	C, A=0.70 in.	L52	192	178	175	153	B2			200
504 ^(b)	Ratio	200	6	C, A=0.3 in.	L53	210	189	--	--	C4	49		
<u>7075-0 Aluminum T-Sections</u>													
488	T-Die design	7.3	20	S	L53	120	105	108	99	D1	25		
<u>Re-extrusion of 7075-0 T-Sections</u>													
482	Re-extrusion	2	20	S	L17	--	34.5	--	--	--	1-1/2	Leaked	
507	Re-extrusion	2	6	S	L17	42	40.5	--	--	--	14	Woods metal aided sealing	
489	Re-extrusion	4	20	S	L53	75	73.5	55	52.5	D2	24	Woods metal aided sealing	
<u>Dispersion-Hardened Aluminum</u>													
475	Ratio	10	20	S	L53	104	99	99	92	B2	20		
476	Ratio	20	20	S	L53	156	139	135	117	B2	20		
490	Ratio	40	20	C, A=0.75 in.	L53	221	202	--	--	C4	1/2		

(a) S - Standard nose. C - compound-angle nose, 45 degrees at apex, 30 degrees beyond diameter A in.

(b) Billet diameter 1.414 in., die orifice 0.10 in diameter.

to the sandwich-billet and to the joint interfaces. The extrusion data are contained in Table 2.

At ratios of 10 and 20:1, the whole billet was extruded in one piece. While the extruded products were craze cracked at the beginning, 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 obtained with 7075-0 aluminum. The breakthrough pressures were probably influenced by the joint with the leading 7075 aluminum billet. It is noteworthy that at both these ratios, the rear part of the sandwich billet failed to extrude and seized at the die interface. This is believed to be due to compaction of the sintered aluminum which was initially 85 percent dense. The compaction probably left a protruding edge of 7075 aluminum on the rear billet which caused metal-metal contact and hence seizure.

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

The experimental data for hydrostatic extrusion of Ti-6Al-4V rounds and tubes are given in Table 3. Data previously reported are also presented for comparison purposes. In addition to the hydrostatic extrusion trials, several hydrostatic compacts of Ti-6Al-4V alloy powder were produced.

Rounds

In previous hydrostatic extrusions of Ti-6Al-4V at a ratio of 4:1 (Trial 193⁽¹⁾ and Trial 376⁽⁷⁾), severe stick-slip occurred and, in one instance, transverse cracking occurred in the areas of sticking during stick-slip. In Trial 487, stick-slip was completely eliminated and an excellent surface finish without surface cracks was obtained. This improvement is attributed partly to increasing the stem speed from 6 to 20 ipm and partly to using a compound-angle nose on the billet. Both of these factors apparently also contributed to a 6-7 percent lowering of breakthrough and runout pressures.

The pressures obtained at room temperature were further reduced by about 10 percent by extruding at 400 F, where the only difference in conditions was, of necessity, the billet lubricant and fluid. At this temperature, the anodized coating (C3) was used for the first time (Trial 496). The data for Trial 416 conducted previously at 400 F is given for comparison. The pressures for Trial 496 were only marginally lower than for Trial 416 (by about 3 percent), and there was no difference in the surface finish of the products, both being excellent. The coating, higher stem speed, and compound nose in Trial 496 appeared to have had only marginal effect on pressure levels. It is believed that Lubricant L33 (55 wt% MoS₂ and 6 wt% graphite in sodium silicate) was largely responsible for the good surface finish and smooth runout conditions obtained at 400 F.

TABLE 3. EXPERIMENTAL DATA FOR HYDROSTATIC EXTRUSION OF Ti-6Al-4V TITANIUM ALLOY

Die angle - 45 degrees included
Fluid - Castor oil

Nominal tube dimensions -
Billet - 0.750 OD x 0.069 wall
Extrusion - 0.663 OD x 0.030 wall

Trial	Objective or Variable	Temperature, F	Extrusion Ratio	Stem Speed, ipm	Billet Nose Design ^(a)	Billet Lubrication		Extrusion Pressure, 1000 psi				Type of Curve, p 25	Length of Extrusion, inch	Comments	
						Coating	Lubricant	Breakthrough		Runout					
						Rounds									
						Stem	Fluid	Stem	Fluid						
376	Reference	80	4	6	S	C3	L17	271	242	242	222	D3	9	Interim Report VI	
487	Elimination of stick-slip	80	4	20	C, A=1.2	C3	L17	266	228	240	207	B1	13		
416 ^(b)	Reference	400	4	6	S	None	L33	212	198	206	194	B2	8-1/4	Interim Report VII	
496 ^(b)	Lubricant	400	4	20	C, A=1.2	C3	L33	210	195	198	187	B2	12		
						Tubing (80 F)									
						Mandrel Dimensions									
						Max Diameter, in.	Taper, in./in. on Diameter	Stem	Fluid	Stem	Fluid				
437	Reference	0.613	0.0012	2.5 ^(c)	6	S	C3	L17	85	79.5	87	77	B3	4-1/2	Interim Report VIII
485	Mandrel	0.613	0.002	2.5 ^(c)	20	S	C3	L17	84	79	81	78	B3	10	
506	Mandrel	0.606	0.0008	2.5 ^(c)	20	S	C3	L17	85	75	84	74	B3	6-1/2	

(a) S - Standard nose; C - compound nose, 45 degrees at apex, 30 degrees beyond diameter A in.

(b) Fluid - silicate ester.

(c) Nominal ratio; ratio varied slightly over tube length because mandrel was tapered.

Tubing

Hydrostatic extrusion of Ti-6Al-4V tubing was continued with the aim of investigating the mandrel-design requirements for continuous extrusion of tubing from thin-wall tube blanks without billet upsetting. Data are given in Table 3 for the two trials conducted in the current series and for a single trial (Trial 437) previously reported in which billet upsetting had occurred. Billet upsetting occurs when the billet end pressure developed from the mandrel guide exceeds the fluid pressure by roughly the billet's compressive yield strength. (The billet end pressure can be greater than the fluid pressure because of the unsupported cross-sectional area of the mandrel).

In Trial 485, the mandrel taper was increased over that used in Trial 437 and this allowed a longer length of extrusion to be obtained. However, the diameter on the mandrel at the point where billet upsetting commenced for both these trials was approximately 0.608 inch. This diameter represented an extrusion ratio of about 2.7:1 in both cases. The billet end pressure was apparently sufficiently high at this ratio to preferentially cause upsetting rather than to effect further extrusion.

Thus in Trial 506, the maximum mandrel diameter was reduced in order to lower the maximum extrusion ratio to below 2.7:1 in an attempt to prevent or minimize billet upsetting. However, the effectiveness of this procedure was not determined because some lubricant breakdown occurred at the commencement of extrusion and progressively became more severe, possibly resulting in premature billet upsetting. The reduced extrusion ratio caused the extrusion pressures to be lower by about 5 percent compared with Trials 437 and 485.

Compaction

The hydrostatic extrusion process offers 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 has been selected for investigation at this point, 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 Ti-6Al-4V powder was made by mechanical attrition and was shipped under a helium atmosphere to minimize oxygen contamination. Five rubber bags with nominal internal dimensions of 1-7/8-inch-diameter by 10 inches long were filled with powder.

Despite the fact that the compacts were vibrated during loading the maximum apparent densities achieved were 2.04 g/cc or 46 percent of theoretical density based on 4.43 g/cc.

Two billets were compacted at fluid pressures of 60,000 psi and the remaining three billets were compacted at fluid pressures 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. One of the billets pressed at 60,000 psi broke up upon quenching. The water densities of the billets before and after sintering are given below:

Compacting Pressure, psi	Water Density, percent of theoretical density	
	Before Sintering	After Sintering
60,000	93.4	93.2
225,000	97.5	97.5

The sintered compacts are to be machined into billets and extruded into rods under conditions that have been successful for extruding wrought Ti-6Al-4V. The resulting extrusions will then be evaluated and compared to the wrought material extrusions from the standpoint of surface finish and mechanical properties.

HYDROSTATIC EXTRUSION OF WROUGHT TZM MOLYBDENUM ALLOY AND BERYLLIUM ROUNDS

Efforts were continued in the study of die design with the aim of preventing cracking of relatively brittle materials during hydrostatic extrusion without the necessity of a fluid counter-pressure system. In the previous Interim Report VIII⁽⁶⁾, two basic die concepts were explored: the controlled taper-relief and double-reduction die designs. These are illustrated in Figure 3 along with the standard die profile. 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. This was found to be effective in reducing the number of circumferential or transverse cracks that occurred in hydrostatic extrusion of TZM and beryllium, although longitudinal hairline cracking still persisted.

The double-reduction die was designed to take a very small reduction of the product at a second land shortly beyond the first. It was 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 degree of change in the pattern would appear to depend on extrusion conditions including the size of the first and second reductions, the distance between lands, the relief

configuration after each land, billet material, die angle, extrusion speed, and extrusion temperature.

Several double-reduction die designs have been investigated thus far:

<u>Double-Reduction Die Designation</u>	<u>Second Reduction, percent</u>	<u>Distance Between Lands, H, inch^(a)</u>	<u>Included Angle of Second Reduction, θ degrees^(a)</u>	<u>Total Reduction, percent</u>
A	1.5	5/8	45	60
B	3.3	5/8	45	75
C	2.0	5/8	45	74.6
D	2.0	0	45	74.6

(a) See Figure 3 for details

The experimental data obtained with these dies are contained in Table 4.

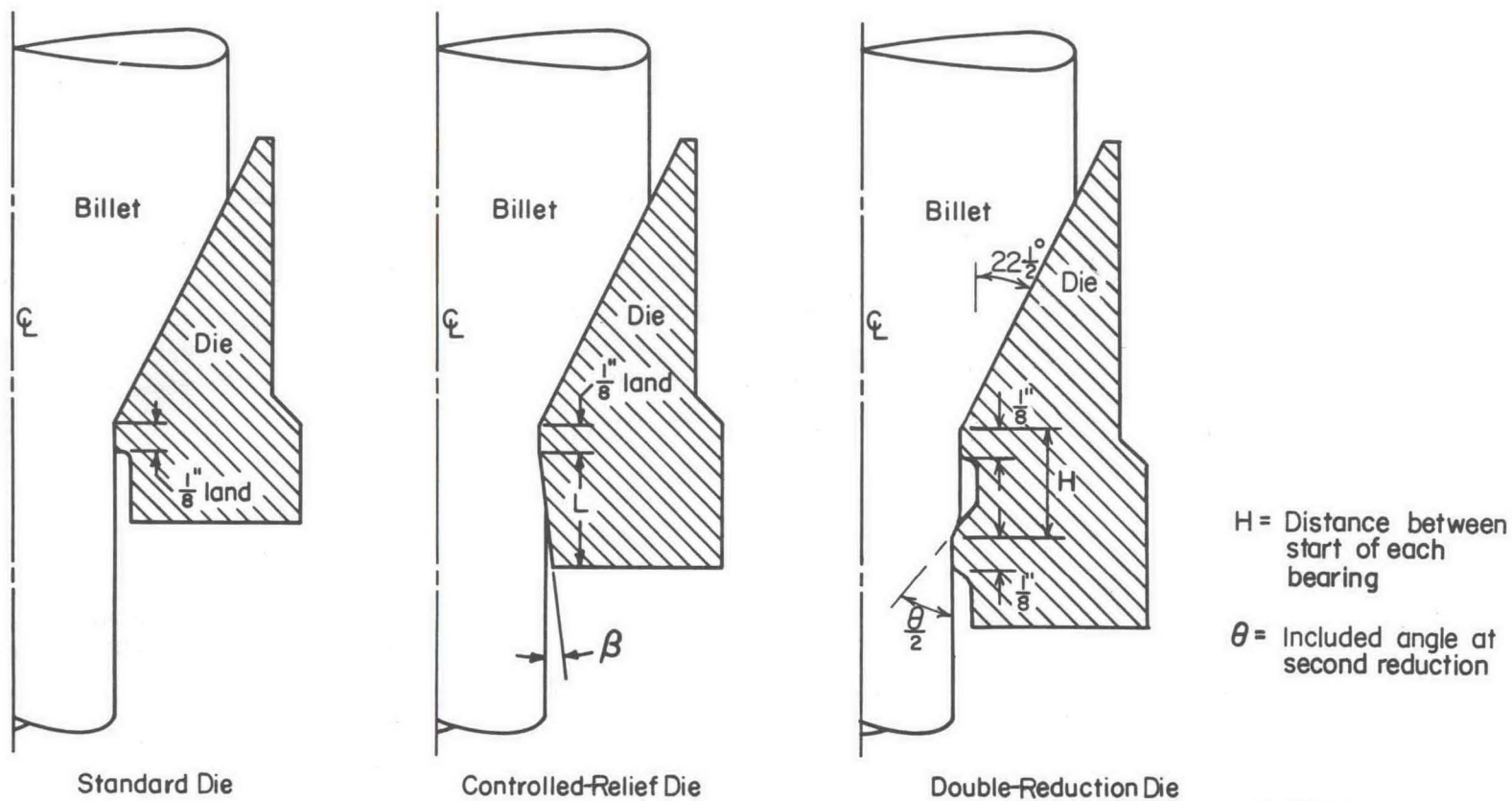
The results obtained with these dies are truly significant. For the first time, beryllium has been cold hydrostatically extruded at a ratio of 4:1 into virtually a crack-free product without the need of fluid back pressure (Trial 495). Similarly, wrought TZM alloy, in both the stress-relieved and recrystallized states, was extruded into crack-free products at a ratio of 4:1. This represents a major breakthrough of utmost importance in deformation of brittle materials.

In the previous interim period⁽⁶⁾, only Die Design A had been evaluated on TZM (Trial 469). Transverse cracks were eliminated but longitudinal hairline cracking still occurred. Increasing the second reduction from 1.5 to 3.3 percent (Design B), and the overall extrusion ratio from 2.5 to 4:1 while maintaining the 5/8-inch spacing between lands, permitted the extrusion of a 1-inch length of crack-free product (Trial 478). Only a short extrusion was produced because runout pressures rose rapidly indicating lubrication breakdown and the trial was stopped. The reason for the pressure rise was not clear, but it was felt that a smaller second reduction might improve runout conditions.

In Die C, the reduction in area at the second bearing was reduced to 2.0 percent, the overall reduction remaining nominally 75 percent. This modification was effective in preventing cracks in both recrystallized TZM (Trial 483) and beryllium (Trial 495). Figure 4 shows the crack-free TZM extrusion along with two other extrusions obtained earlier with the standard die profile. The fact that cracking occurred at a higher extrusion ratio (5:1 in Trial 443 versus 4:1 in Trial 483) with 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.⁽⁸⁾
- (2) Die design itself is a very important factor in controlling the conditions which cause cracking.

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



A-54904

FIGURE 3. STANDARD DIE PROFILE AND TWO DIES DESIGNED TO ELIMINATE CRACKING IN BRITTLE MATERIALS

TABLE 4. EXPERIMENTAL DATA FOR HYDROSTATIC EXTRUSION OF TZM MOLYBDENUM ALLOY AND BERYLLIUM

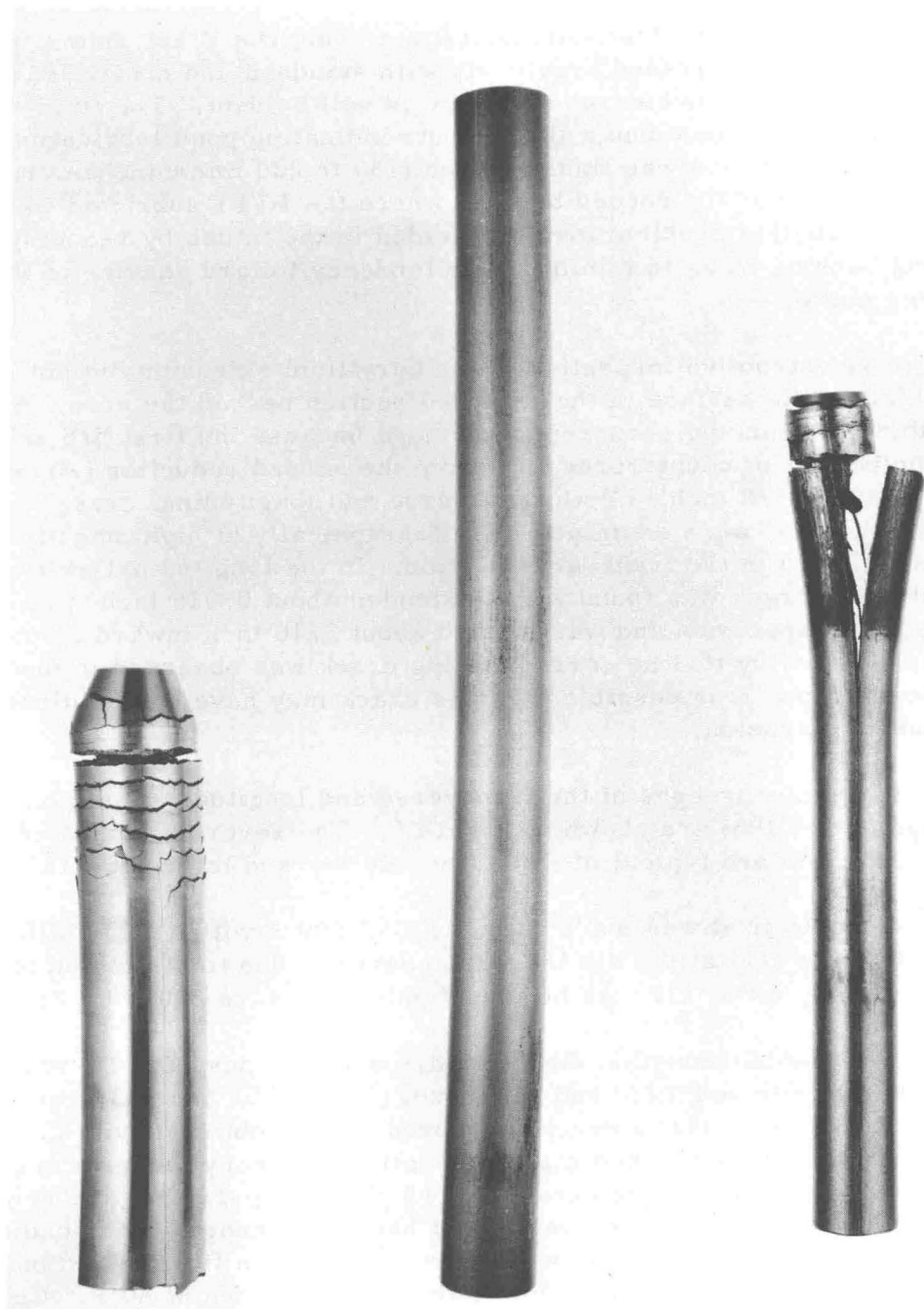
Die angle - 45 degrees (included) Billet lubricant - L38
 Fluid - Castor oil¹

Trial	Objective or Variable	Die ^(a)	Extrusion Ratio	Stem Speed, ipm	Extrusion Pressure, 1000 psi				Type of Curve, p 25	Length of Extrusion, inch	Cracks ^(b)	
					Breakthrough		Runout				Circumferential	Longitudinal
<u>Wrought TZM - Stress Relieved</u>												
469	Reference	A	2.5	6	157	141	142	129	B1	4	None	3
478	Die design	B	4	6	280	242	--	--	B4	1	None	None
505	Die design	D	4	6	252	218	205	183	B1	5	Nose only	4
501(c)	Temperature	C	4	6	--	--	--	--	--	--	Die seal leak	
502(c)	Temperature	D	4	6	178	166	171	159	B2	7	None	None
<u>Wrought TZM - Recrystallized</u>												
483	Die design	C	4	20	198	176	194	168	B1	12	None	None
<u>Beryllium - Powder Origin</u>												
495	Die design	C	4	20	234	205	228	200	B1	10	None	None
503(c)	Temperature	D	4	20	150	140	143	133	B1	14	Numerous	Numerous

(a) See table on p 14 for double reduction die details.

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

(c) 500 F extrusion using polyphenyl ether (PPE) as the fluid medium. Fluid pressures estimated from stem pressures. High-temperature high-pressure gage out of order.



Trial	441	483	443	37022
Extrusion Ratio	2.5:1	4:1	5:1	
Billet Lubricant	L17	L38	L17	
Die	Standard	Double Reduction	Standard	

FIGURE 4. INFLUENCE OF DOUBLE REDUCTION DIE ON CRACKING OF HYDROSTATIC EXTRUSIONS OF WROUGHT TZM MOLYBDENUM ALLOY

The results obtained with beryllium using Die C are shown in Figure 5 along with other specimens extruded previously with standard and controlled-relief dies. The effectiveness of the double-reduction die is self evident. 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. It is believed that this problem may be avoided in the future by reducing the entry angle to the second bearing so as to minimize the tendency toward shaving of the lubricant and extruding metal.

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). Both transverse and longitudinal cross sections of the extrusion beyond the nose were examined metallographically at high magnification. No cracks were observed in the transverse section. In the longitudinal section, a single hairline longitudinal crack was found which extended about 0.015 inch from the transverse-cut plane of the specimen and was located about 3/16 inch inward from the extruded surface. It was noteworthy that no corresponding crack was observed in the mating longitudinal section. Thus, it is possible that this crack may have been a direct result of sectioning and not of extrusion.

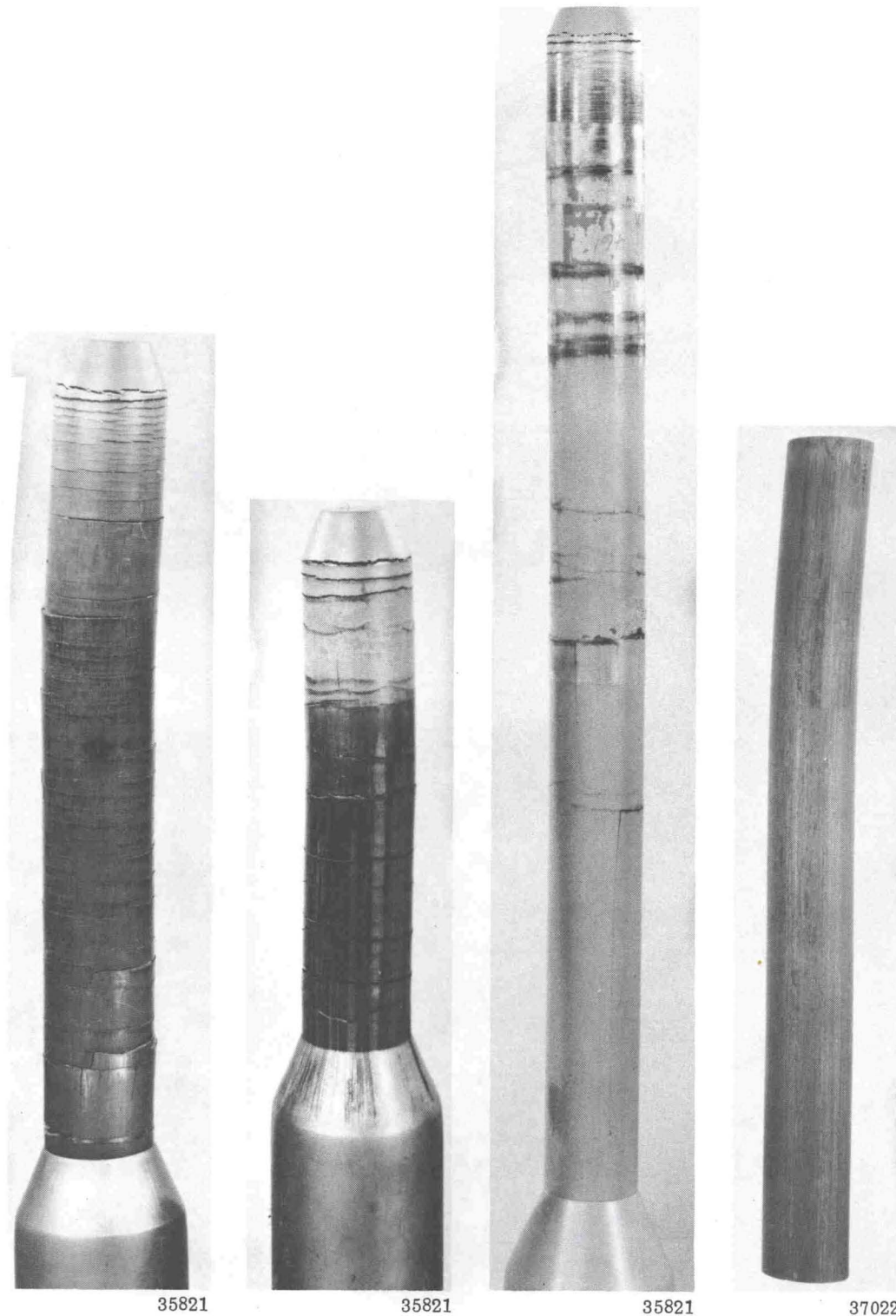
Photomicrographs of the transverse and longitudinal sections of the beryllium extrusion (Trial 495) are shown in Figure 6. The severely elongated grains in the longitudinal section are typical of a heavily cold-worked microstructure.

A single trial was made with Die C at 500 F with a TZM billet (Trial 501). The PTFE O-ring seal at the die leaked, apparently due to distortion of the O-ring at 500 F. Other O-ring materials may be investigated in future 500 F trials.

The double-reduction die with adjacent bearings, Die D, was evaluated at 500 F with stress-relieved TZM and beryllium (Trials 502 and 503, respectively). It is of particular interest that a crack-free product was obtained with the TZM, but the beryllium extrusion was cracked circumferentially in many places along its length. More trials will be necessary to determine whether temperature, the new die design (Die D), or both had an influence on cracking of beryllium under these conditions. The low pressures required for beryllium were particularly significant. Extruding beryllium at 500 F required pressures 1/3 lower than those needed at 80 F. Consequently, much higher extrusion ratios than 4:1 can clearly be obtained at 500 F within the 250,000 psi present capacity of the extrusion tooling.

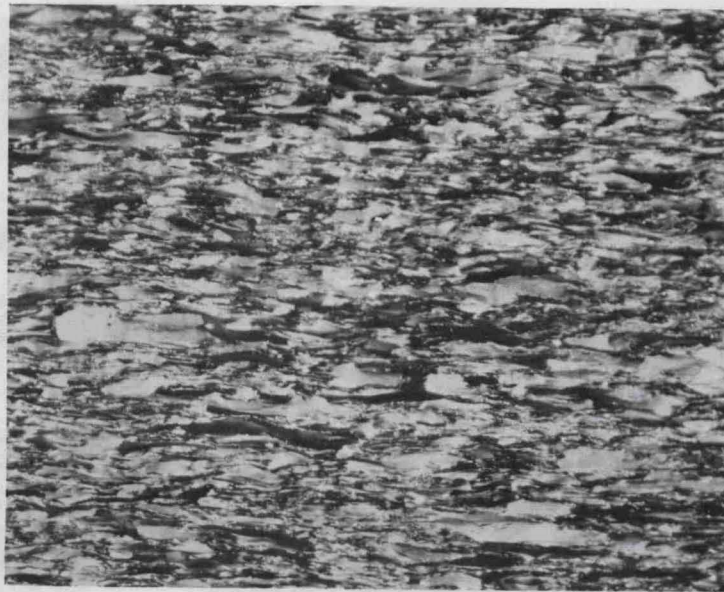
It appears that the crack-free product of TZM obtained with Die D may have been due to the elevated temperature only and not the design of Die D since a repeat of these extrusion conditions at room temperature with TZM (Trial 505) produced a cracked extrusion. Another trial at 500 F but with the standard die design would confirm whether crack elimination in this case was due to temperature, die design, or both factors.

To date, sound hydrostatic extrusions of beryllium and TZM have been obtained by other workers^(8,9) when the product was extruded into a high fluid pressure environment (fluid-to-fluid extrusion). The extrusion ratios achieved here were in the order of 2:1. The provision of a fluid back pressure requires expensive tooling on the exit side of the



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	L38	L38	L38	L38
Die	Standard	Standard	Controlled Relief	Double Reduction (Die C)

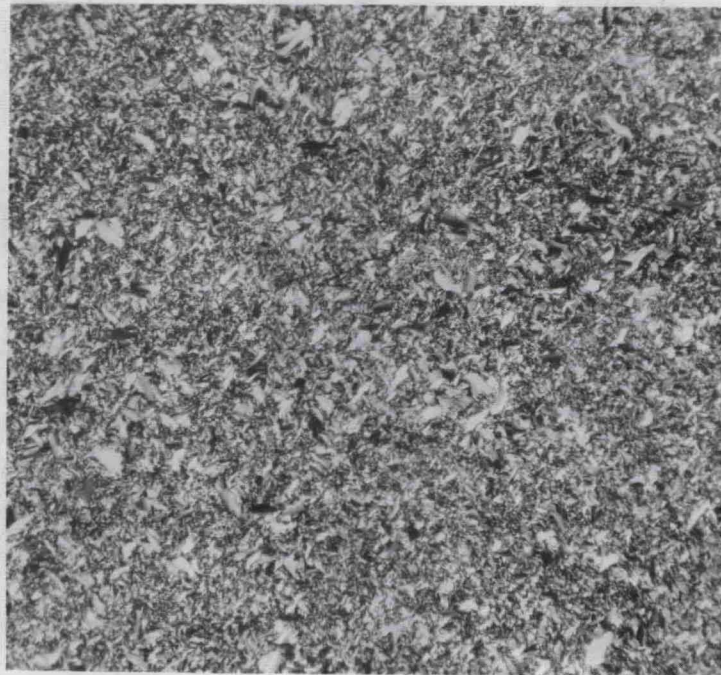
FIGURE 5. INFLUENCE OF DIE DESIGN ON CRACKING IN HYDROSTATIC EXTRUSIONS OF BERYLLIUM



100X

a. Longitudinal

6B551



100X

b. Transverse

6B007

FIGURE 6. PHOTOMICROGRAPHS OF BERYLLIUM COLD EXTRUDED AT A RATIO OF 4:1 BY HYDROSTATIC EXTRUSION WITHOUT FLUID COUNTERPRESSURE

die sufficient in length to accommodate the long extrusion. Also, the main pressure chamber is required to contain pressures in excess of those required in fluid-to-air extrusion by the amount of back pressure.

With the double-reduction die a compressive stress is transmitted to a narrow circumferential region of the extrusion only, and the results obtained so far indicate that the small second reduction does not require any appreciable extra fluid pressure over that required for the first reduction. Thus, much of the back pressure in fluid-to-fluid extrusion would appear to be superfluous. The double-reduction die apparently avoids the need for back pressure tooling and the associated higher fluid pressure containment in the main chamber.

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.

HYDROSTATIC EXTRUSION OF SUPERALLOYS ALLOY 718 AND A286

The objective of this series of trials was to determine the extrudability of the superalloys A286 (iron-base) and Alloy 718 (nickel-base). The results obtained are shown on Table 5.

A286 and Alloy 718 billets were received in the solution-treated condition. The initial hardnesses were $<10 R_C$ and $16 R_C$, respectively. All billets were lubricated with L 38 (PTFE) and extruded at a stem speed of 20 ipm through standard-profile dies of 45-degree included angle.

The maximum extrusion ratios that were achieved at room temperature within the 250,000 psi pressure capacity of the tooling were:

A286	5:1
Alloy 718	3.3:1

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.

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-drawing. In this Battelle-developed process, the wire is subjected to hydrostatic fluid pressure on the entry side of the die and controlled draw stress on the exit

TABLE 5. EXPERIMENTAL DATA FOR HYDROSTATIC EXTRUSION OF SUPERALLOYS

Die angle - 45 degrees (included) Billet surface finish - 60-100 microinches (RMS)
 Fluid - Castor oil at 80 F Billet lubricant - L38
 Polyphenyl ether (PPE) at 500 F Stem speed - 20 ipm

Trial	Extrusion Ratio	Extrusion Temperature, F	Extrusion Pressure				Type of Curve, p 25	Length of Extrusion, inch	Comments
			Breakthrough		Runout				
			Stem	Fluid	Stem	Fluid			
<u>Superalloy - A-286 (iron-based)</u>									
479	3.3:1	80	198	173	190	165	B-1	13	
480	5:1	80	280	234	258	217	B-1	19	
500	5:1	500	235	--	217	--	B-3	5	Lubrication broke down
<u>Superalloy - Alloy 718 (nickel-based)</u>									
481	3.3:1	80	273	225	258	217	B-1	15	
484	3.3:1	80	285	238.5	270	226.5	B-1	11	
499	3.3:1	500	245	--	228	--	B-3	3	Lubrication broke down

side. The equipment for exerting and monitoring the draw stress on the wire was described in Interim Report VII⁽⁵⁾ and details of the starting wire are given in Interim Report VIII⁽⁶⁾.

Two trials were conducted at a nominal temperature of 500 F with 0.0196-inch-diameter beryllium wire of ingot origin. The wire was lubricated with L38 (PTFE) and was reduced to 0.0124-inch diameter in one pass (60 percent reduction). The die used was made from tungsten carbide, its entry angle was 45 degrees included, and its bearing length was approximately 0.003 inch. In both trials, about 15 inches of good-quality wire was produced.

The fluid (polyphenyl ether) and tooling were heated to 500 F, but, because of a time lapse during loading of the wire, some heat was apparently conducted away from the area of the die to the support tooling. Consequently, during extrusion, the pressure plus draw stress (P + D) requirements to produce the wire gradually increased until they became excessive. However, the P + D requirements at the beginning of extrusion were in the order of 100,000 psi. In future trials at elevated temperature, procedures will be modified to maintain temperature during loading and extrusion. Such steps are not necessary in the 500 F extrusion of the large-diameter billets because much more rapid handling and extrusion of the billets is possible.

The starting wire had an irregular surface finish and this was carried through onto the extruded wire, although the surface irregularities were smoothed somewhat.

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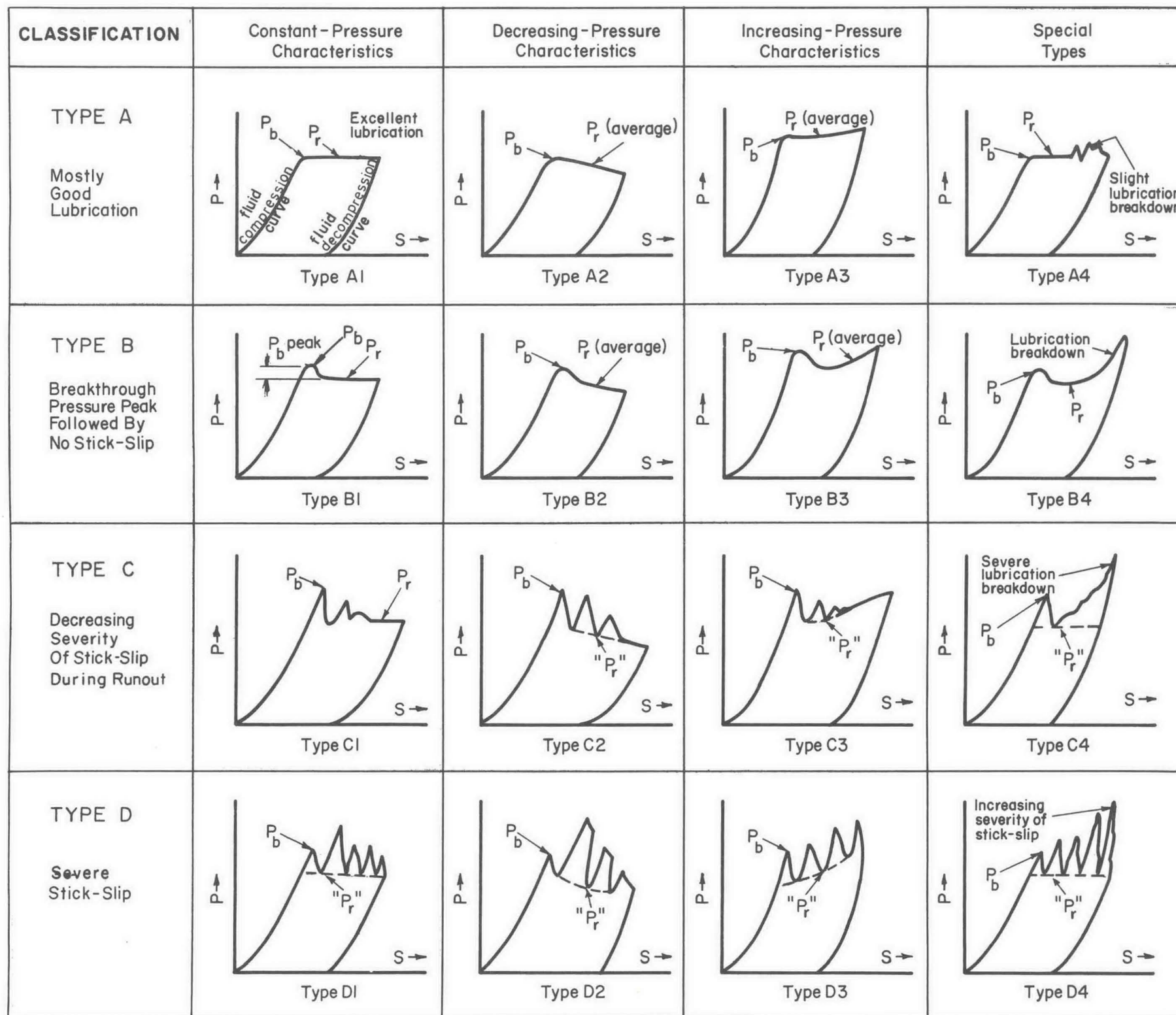
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25 and 26

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FIGURE 1. CLASSIFICATION OF PRESSURE - DISPLACEMENT CURVES OBTAINED IN HYDROSTATIC EXTRUSION

P_b = breakthrough pressure

P_r = runout pressure

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