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THE EXTRUSION OF TITANIUM

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FOREWORD

This report was prepared by the Battelle Memorial Institute under USAF Contract No. AF 33(038)-3736. The contract was initiated under Project No. 7351, "Metallic Materials," Task No. 73510, "Titanium Metal and Alloys," formerly RDO No. 615-11, "Titanium Metal and Alloys," and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with 1st Lt. J. W. Seeger acting as project engineer. In addition to the authors, other Battelle personnel who contributed to this project were G. H. Schippereit, J. H. Jackson, and C. H. Lorig.

Extrusion facilities for this work were provided by Metal Trims, Inc., Youngstown, Ohio, USAF Contract No. AF 33(600)-9203. Metal Trims' operations were under the supervision of E. Dynner and A. Mitchell, who were assisted by K. Demlow.

This report includes an evaluation of the performance of a number of products for a specific application. Many of the materials tested were not developed or intended by the manufacturer for the conditions to which they have been subjected. Any failure or poor performance of a material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

Acknowledgment is given to the following companies for their cooperation in contributing services and materials for the investigations conducted in this research program.

Fiske Brothers Refining Company, Toledo, Ohio
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Kennametal, Incorporated, Latrobe, Pennsylvania
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Firth Sterling, Inc., Pittsburgh, Pennsylvania
Magnethermic Corporation, Youngstown, Ohio

Data on which this report is based are recorded in BMI Laboratory Record Books Nos. 6722 and 10015.

ABSTRACT

Extrusion tests were conducted on unalloyed titanium and the Ti-3Mn-complex alloy to study the effects of extrusion temperature and die design and to evaluate various lubricants and die materials. Optimum mechanical properties were attained at extrusion temperatures in the alpha-phase region for unalloyed titanium and in the alpha-beta region for the Ti-3Mn-complex alloy.

The surface finish of round bars extruded with flat-face dies was very poor. Improved metal flow and surface finish were obtained with conical dies. The optimum die angle appeared to be about 130 degrees.

Lubricants containing graphite, molybdenum disulfide, and mica produced acceptable surface finishes. The best results were obtained with these materials suspended in a Bentone grease. Titanium carbide, chromium carbide, and cobalt-base alloys showed promise as die materials. The carbides exhibited the least wear and less tendency toward seizing by the titanium. High-quality bars were extruded with these die materials and the Bentone lubricant mixture.

PUBLICATION REVIEW

This report has been reviewed and is approved:

FOR THE COMMANDER:

M. R. WHITMORE
Technical Director
Materials Laboratory
Directorate of Research

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INTRODUCTION

The increased demand for titanium structural shapes by the aircraft industries has emphasized the need for the development of new fabrication techniques for this metal. By its inherent characteristics the extrusion process offers the most advantageous method for forming many of the desired shapes. Consequently, there has been considerable activity in this field by both the Government and private industry.

At the time this research program was initiated, little was known regarding the effects of process variables on the surface quality and mechanical properties of extruded titanium. Although the extrusion process has long been in use for aluminum, copper, and other metals and alloys, little of the information could be applied to titanium. Thus, the objective of this research program was to obtain basic information on the factors involved in extruding titanium. The experimental work comprised three principal investigations, the aims of which were as follows:

(1) Evaluation of the effect of extrusion temperature on the mechanical properties of extruded titanium and a titanium alloy

(2) Evaluation of the effect of conical die design on metal-flow patterns. surface finish of the extruded bars, and extrusion pressure

(3) Evaluation of commercial and experimental lubricants and die materials.

Since this work was essentially of an exploratory nature, the investigations were conducted only on round bars. It is expected that the results of the tests conducted in this program will serve as a useful guide toward the selection of lubricants, die materials, and the proper die design for extruding more complex titanium shapes.

SUMMARY

This report describes the experimental work conducted during the period April 1, 1953, to December 31, 1954, under WPAFB Contracts Nos. AF 33(038)-3736 and AF 33(600)-9203. The primary purpose of this work was to study some of the major factors involved in the extrusion of titanium and titanium alloys.

The extrusion tests were conducted at Metal Trims, Inc., Youngstown, Ohio, using an 850-ton aluminum extrusion press. In these investigations, 4-7/16-inch-diameter billets were extruded to 1-inch-diameter bars, the extrusion ratio being 21:1.

Effect of Extrusion Temperature on Mechanical Properties

The effects of extrusion temperature on the mechanical properties of unalloyed titanium and the Ti-3Mn-1Cr-1Fe-1Mo-1V alloy* were determined. The billets were extruded through flat-face dies, using a graphite-base lubricant.

The unalloyed billets were extruded at 1450, 1600, 1750, and 1900 F. The bars extruded in the alpha-phase region (1450 and 1600 F) exhibited higher yield and ultimate strengths than those extruded in the beta-phase region (1750 and 1900 F), the average ultimate strengths being 85,000 and 70,000 psi, respectively. At 1450 and 1600 F, the bars were only partially recrystallized, whereas the bars extruded in the beta-phase region had completely recrystallized by transformation to alpha on cooling. Annealing for 1 hour at 1500 F and air cooling did not produce any significant change either in tensile properties or in microstructure.

Billets of the Ti-3Mn-complex alloy were extruded at 1350, 1450, 1525, 1600, and 1700 F. In the as-extruded condition, specimens of the bars extruded at 1525 F or higher exhibited brittle fractures. At the lower extrusion temperatures of 1350 and 1450 F, however, this alloy exhibited good combinations of strength and ductility - 183,000 to 200,000-psi ultimate strength and 10.5 to 3.5 per cent elongation in 2 inches. Solution treating at 1300 F, followed by overaging at 800, 900, and 1100 F for various lengths of time, produced strength levels of 180,000, 170,000, and 130,000 psi, respectively. The ductility at these strength levels, however, decreased as the extrusion temperature increased. In each of the conditions tested, the optimum combination of strength and ductility was obtained at an extrusion temperature of 1350 F.

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*This alloy is referred to as the "Ti-3Mn-complex alloy" in this report.

Die Design

In all of the tests with flat-face dies, the surfaces of the extruded bars were scored severely. Examination of the billet skulls revealed the occurrence of a stagnant-metal zone at the die shoulder during extrusion. As a result, the lubricant on the tools became ineffective in preventing the titanium from seizing the die and poor surfaces were obtained.

A considerable improvement in surface finish was attained through the use of conical dies. Four billets of unalloyed titanium were extruded at 1600 F through dies having included angles of 180 (flat face), 160, 140, and 120 degrees. As the included angle decreased, the size of the stagnant-metal zone decreased, until it was completely eliminated with the 120-degree die. The best surface finish, however, was produced with the 140-degree die, which caused only a slight stagnant zone on the lower half of the billet. Also, the 140-degree die required the lowest extrusion pressure, whereas the 120-degree die required the highest pressure. These tests indicated that the optimum die angle, on the basis of metal flow, surface finish, and extrusion pressure, was between 120 and 140 degrees.

Lubricants and Die Materials

Evaluation studies were conducted on a number of lubricants and die materials to determine their comparative performance, with regard to the surface finish of the extruded bars. The extrusion tests were made with 130-degree conical dies.

The Ti-3Mn-complex alloy was extruded at 1600 F to evaluate four experimental lubricants of the following types:

(1) Calcium grease, graphite, and mica

(2) Calcium grease, molybdenum disulfide, and mica

(3) Calcium grease, graphite, molybdenum disulfide, and mica

(4) Bentone, graphite, molybdenum disulfide, and mica.

The Bentone lubricant produced the best surface finish and the least amount of die pickup. Also, the lowest extrusion pressure was required with this lubricant.

The consistency of the lubricant mixture was found to have a marked effect on the surface finish of the extruded bars. In tests with the Bentone mixture in both heavy and medium consistencies, a more uniform distribution of the lubricant on the tool surfaces could be attained with the lighter mixture. Excessive accumulation of the lubricant on the tools was avoided and a smoother surface finish was obtained on the bars than with the heavier mixture.

In the die-evaluation studies, nine materials, comprising titanium carbides, chromium carbide, cobalt-base alloys, a tungsten-base alloy, and a tungsten tool steel, were used to extrude the Ti-3Mn-complex alloy at 1700 F. The carbide materials appeared to be the most satisfactory for extruding titanium in that they exhibited the least wear and the least tendency toward seizing by the titanium. Although the dies were susceptible to heat checking, dimensional tolerances were not affected. In tests with the Bentone lubricant, the best surface finish was attained with the carbide dies, although good surface finishes also were obtained with the cobalt-base-alloy dies.

The typical surface quality obtained in extrusion of the Ti-3Mn-complex alloy when using the better die materials and lubricants evaluated in this research is illustrated in Figure 1. It is believed that, with additional research, this quality can also be achieved in titanium-alloy extrusions of more complex shape.

EXPERIMENTAL PROCEDURES AND EQUIPMENT

The extrusion tests described in this report were conducted at the Metal Trims, Inc., plant in Hubbard, Ohio. An 850-ton Watson-Stillman extrusion press, equipped with a container 4-5/8 inches in diameter by 17 inches long, was used. The ram had an advance speed of 430 inches per minute and a maximum pressing speed of 41 inches per minute. Maximum hydraulic pressure was 3150 psi, which corresponded to an extrusion pressure of 115,000 psi on a billet. A photograph of the press and its auxiliary equipment is shown in Figure 2.

The billets used in the tests were 4-7/16 inches in diameter by 6 inches long and weighed approximately 16 pounds. A 60-cycle, single-phase induction furnace, designed and built for heating titanium by the Magnethermic Corporation, Youngstown, Ohio, was used to heat the billets. A billet could be heated to 1900 F in approximately 13 minutes in this furnace. Induction heating was used because the rapid heating prevents excessive oxidation of the billet surface. The billet temperature was measured with a Brown radiation pyrometer, which was calibrated against readings of thermocouples located at various points near the center and surface of a titanium billet. In the range 1400 to 1900 F, a maximum temperature gradient of 80 F existed between the center and surface of the billet.

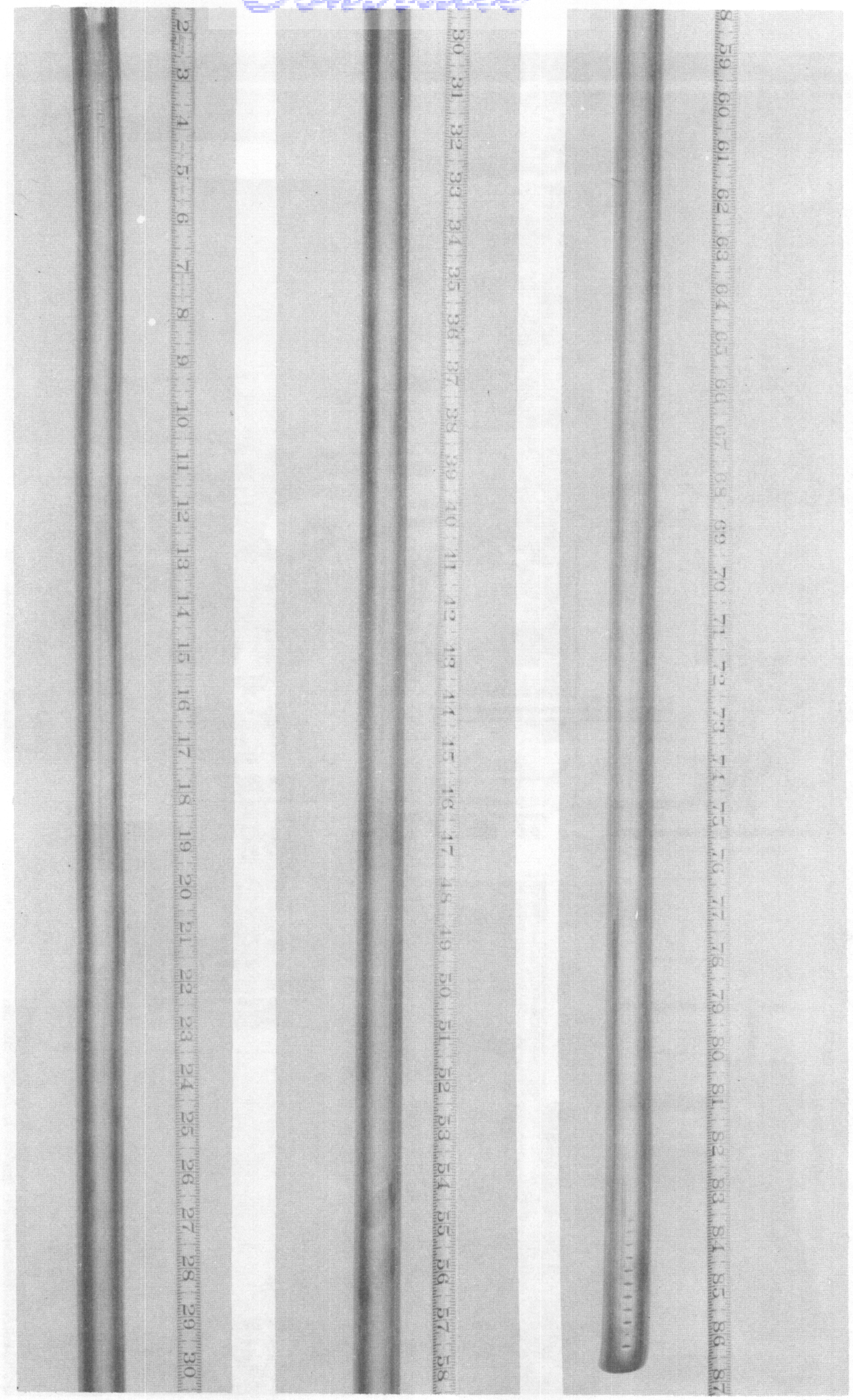

FIGURE 1. ENTIRE LENGTH OF A 1-INCH-DIAMETER BAR OF THE Ti-3Mn-COMPLEX ALLOY, SHOWING THE TYPICAL AS-EXTRUDED SURFACE FINISH OBTAINED WITH THE CARBIDE AND COBALT-BASE DIE MATERIALS AND THE BENTONE-GRAPHITE-MOLYBDENUM DISULFIDE-MICA LUBRICANT

Markings on surface are stains from lubricant and oxidation; no scoring or tearing occurred.

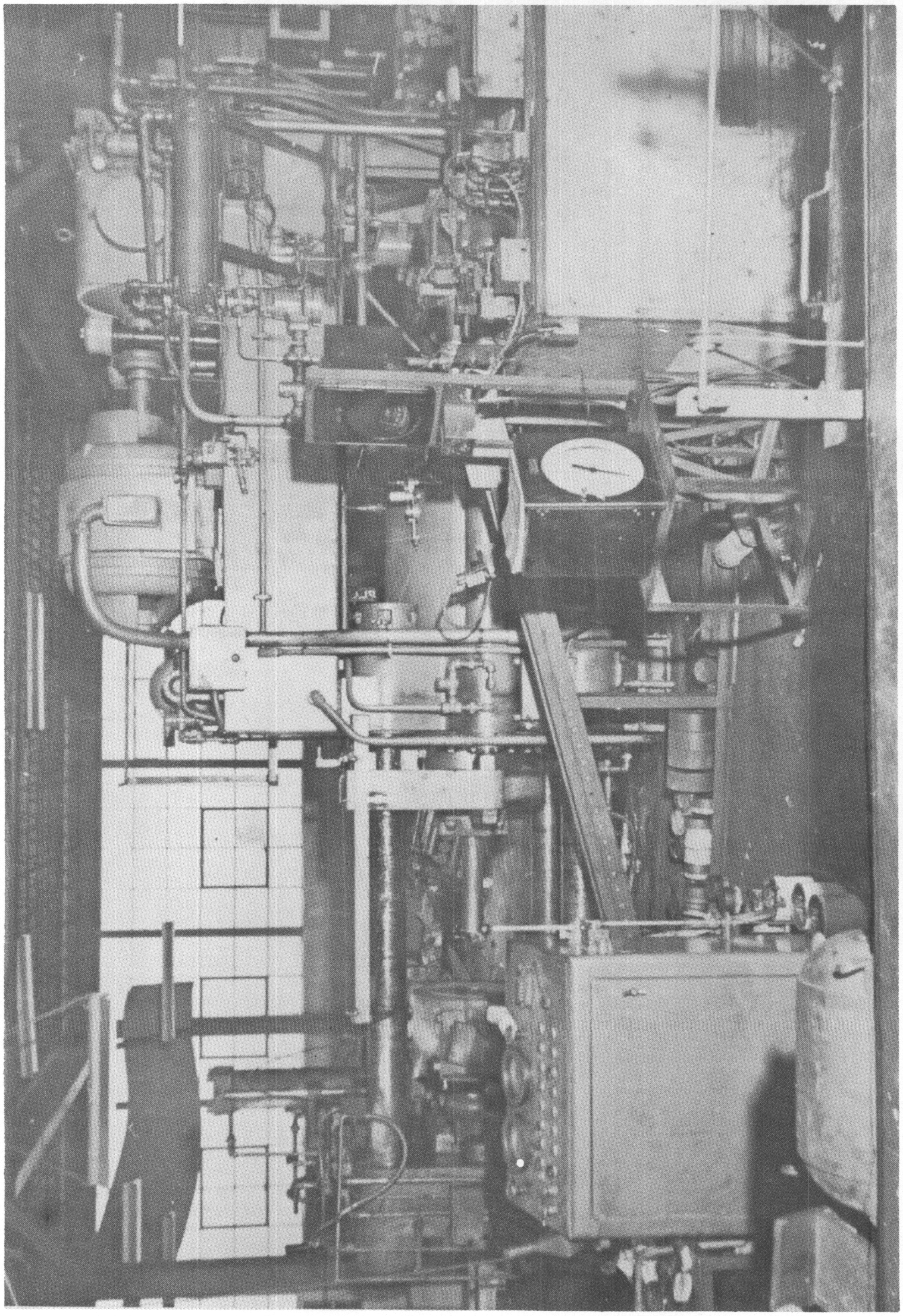

FIGURE 2. PHOTOGRAPH OF THE EXTRUSION PRESS AND AUXILIARY EQUIPMENT AT METAL TRIMS, INC., USED IN THIS RESEARCH

The extrusion-press container was heated to 800 F prior to extrusion. The dies and dummy blocks were preheated to temperatures in the range 500 to 800 F and placed in the holders just before extrusion.

In all of the tests, the billets were extruded to 1-inch-diameter bars. This corresponds to a 21:1 extrusion ratio, or a reduction in area of 95.4 per cent.

Extrusion pressures were measured with a Bacharach hydraulic-pressure indicator. The instrument recorded the hydraulic pressure on the main cylinder as a function of ram travel.

STUDIES ON THE EFFECT OF EXTRUSION TEMPERATURE

As in the case of other alloy systems that undergo allotropic transformation, the mechanical properties of titanium and its alloys are affected by the working temperatures. In commercially pure titanium, allotropic transformation can occur over a range of temperatures between about 1625 and 1700 F, depending upon the amount of impurities present. The effects of hot working in the beta-phase region, however, are not so detrimental for unalloyed titanium as for the alpha-beta alloys. Research at Battelle on titanium-alloy development has shown that the mechanical properties of alpha-beta alloys hot rolled at temperatures in the beta region are much poorer than the properties obtained by rolling in the alpha-beta region. The first series of extrusion tests, therefore, was conducted to determine the effect of extrusion temperature on the mechanical properties of the extruded metal. These studies were conducted on unalloyed titanium and the Ti-3Mn-complex alloy. The latter, having a nominal composition of 3Mn, 1Cr, 1Fe, 1Mo, and 1V, transforms to an all-beta structure at about 1550 F. The billets of this alloy used in the tests were purchased from the Mallory-Sharon Titanium Corporation. Chemical analysis of the billet stock indicated that the total alloy content was about 9 per cent (3.82Mn, 1.39Cr, 1.13Fe, 1.33Mo, 1.29V, bal Ti), or 2 per cent above the nominal composition. As a result, the beta transus for this material was lower than that for the nominal alloy composition.

Conditions of Tests

The unalloyed billets, which were prepared at Battelle, were extruded at four temperatures in the range 1450 to 1900 F. The Ti-3Mn-complex billets were extruded at five temperatures, ranging from 1350 to 1700 F. The extrusion conditions were as follows:

Test	Billet	Billet Temp, F	Heating Time, min	Extrusion Time, sec
		Unalloyed Titanium		
T-1	13-1	1450	7.4	20
T-2	13-2	1600	10.1	15
T-3	24	1750	10.9	12
T-4	16	1900	13.4	9
		Ti-3Mn-Complex Alloy		
T-13	66	1350	7.5	75
T-9	62	1450	9.0	45
T-10	63	1525	10.0	20
T-11	64	1600	10.5	15
T-12	65	1700	11.0	14

Flat-face dies having a 3/16-inch radius at the edge of the die opening and a bearing length of 3/16 inch were used in these tests. The dies were made of Carpenter 883, a 5 per cent chromium hot-work tool steel, heat treated to a hardness of 45 to 50 R_C. This steel, which is commonly used for aluminum extrusion dies, would not be satisfactory for commercial extrusion of titanium, but served the purpose of this phase of the investigation, in which surface quality was not a consideration. The dies were machined and heat treated at the Metal Trims, Inc., plant. A typical die is shown in Figure 3.

Fiske Hot Die Lubricant No. 514-A was used in each of these tests. This lubricant consists of flake graphite, aluminum soap, mineral oil, and inert filler material. Immediately before placing a billet in the container, both the die face and the container were coated with the lubricant.

No difficulties were encountered in extruding the unalloyed billets at any of the temperatures, and die wear was very slight. However, each of the extruded bars exhibited a scored surface over a portion of the back end. As the temperature was increased, however, the surface condition of the bar improved, the scored portion being reduced from one-third the total bar length at 1450 F to about one-sixth at 1900 F.

The scored surface is believed to be caused by the type of metal flow that occurs with flat-faced dies. Examination of the billet skulls indicated that a stagnant zone of metal exists at the front end of the billet during extrusion. Because of the higher frictional forces at the die face than along the container wall, the flow of metal at the die shoulder is retarded. As a result, the flow surface occurs at some angle to the die face, and the surface skin of the billet is enfolded toward the center of the billet during the latter half of the extrusion stroke. Since the billet has a thin oxide skin

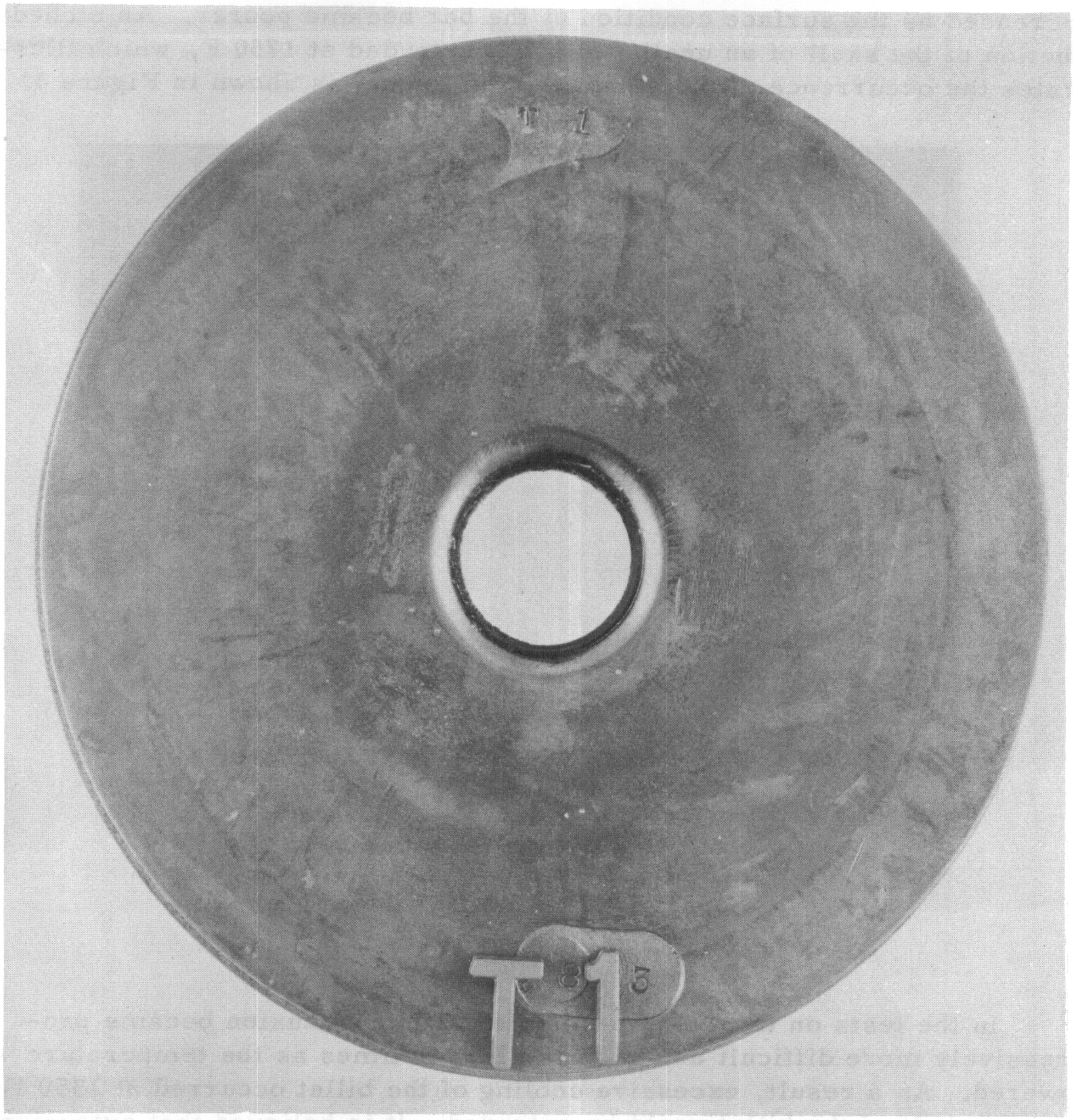

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FIGURE 3. A TYPICAL FLAT-FACE EXTRUSION DIE OF CARPENTER 883 STEEL

Photograph taken after an extrusion of an unalloyed titanium billet and is coated with lubricant from the container wall, a concentric lamination occurs in the bar. The thickness and depth of the laminated ring increased as the surface condition of the bar became poorer. An etched section of the skull of an unalloyed billet extruded at 1750 F, which illustrates the occurrence of the stagnant-metal zone, is shown in Figure 4.

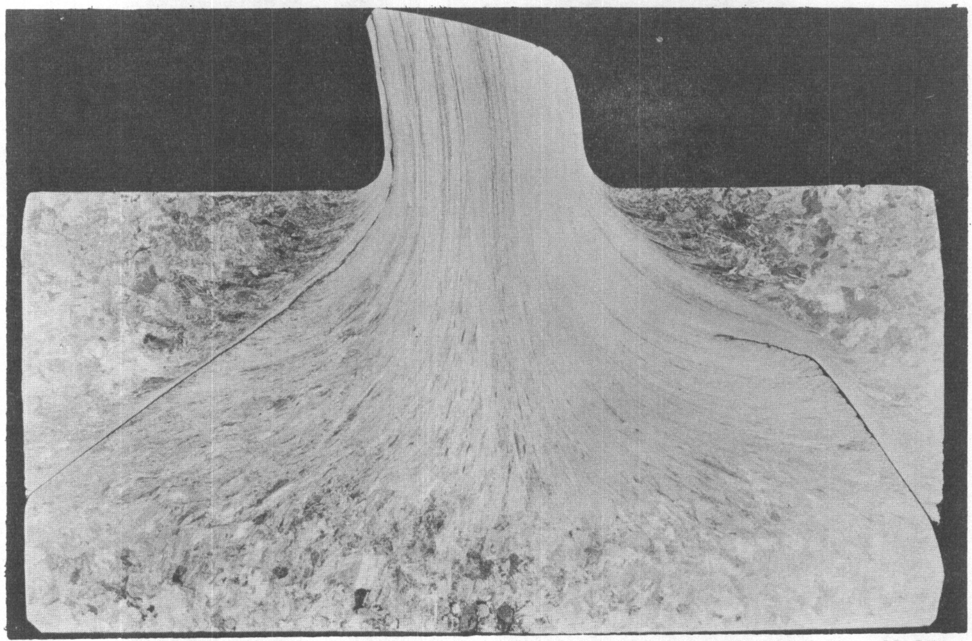


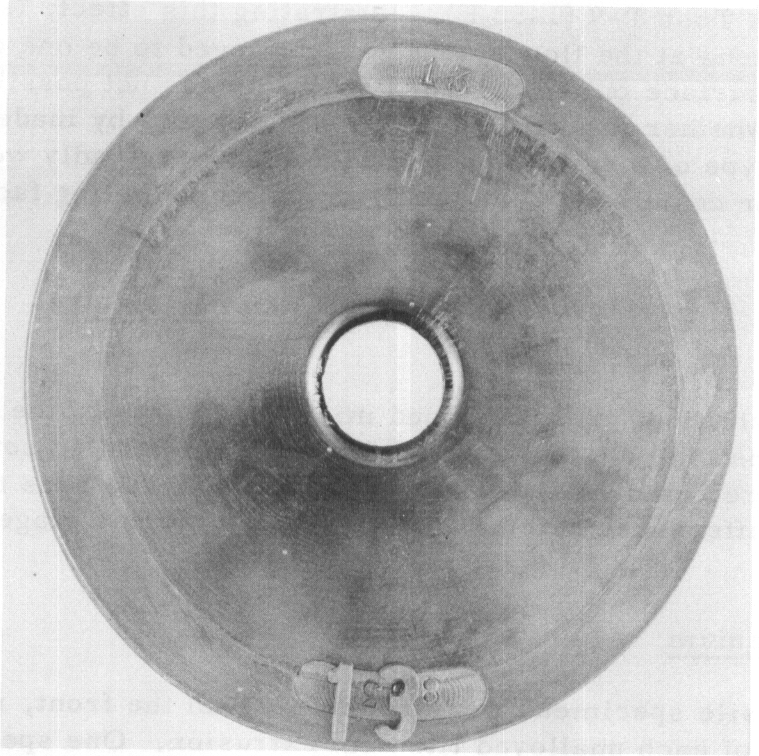
FIGURE 4. ETCHED SECTION OF THE SKULL OF AN UNALLOYED TITANIUM BILLET EXTRUDED AT 1750 F

Note formation of laminated ring caused by stagnant-metal zone.

In the tests on the Ti-3Mn-complex alloy, extrusion became progressively more difficult and required longer times as the temperature was lowered. As a result, excessive cooling of the billet occurred at 1350 F and only half of the billet could be extruded. It is believed that extrusion of the alloy could be accomplished at this temperature on a press having a greater ram speed and larger capacity.

The dies used in the tests at 1450, 1525, 1600, and 1700 F were severely eroded at the die opening, the amount of wear increasing as the temperature was increased. At 1350 F, die wear was very slight; only half of the billet, however, was extruded. The dies used in Tests T-12 and T-13 at 1350 and 1700 F, are shown in Figure 5.

In the tests on the Ti-3Mn-complex alloy, the metal flow was essentially the same as that for the unalloyed titanium billets. The formation of a stagnant metal zone was more pronounced and, in several instances, complete separation of the skull into two pieces occurred at the flow surface.



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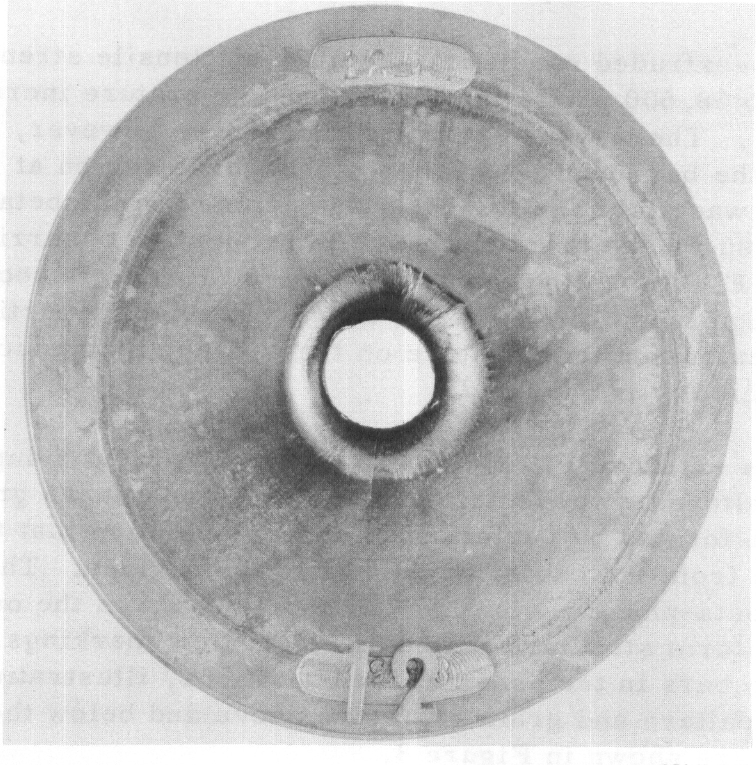


N11230

FIGURE 5. DIES USED TO EXTRUDE Ti-3Mn-COMPLEX BILLETS AT 1350 F (TEST T-13) AND 1700 F (TEST T-12)

The skull from Test T-9 (1450 F), illustrating this effect, is shown in Figure 6. The striations at the flow surface are believed to be one of the causes of the scored surface on the extruded bars. It was not possible, however, to determine whether the poor surfaces were caused by inadequate lubrication or the type of metal flow, since the dies were badly worn. In these tests, die wear could have been the principal contributing factor.

Mechanical Properties and Microstructures

The mechanical properties and microstructures of the extruded bars were determined in both the as-extruded and heat-treated conditions. Specimens were removed from various sections of the bars to determine whether any differences existed in the metal at various stages of extrusion.

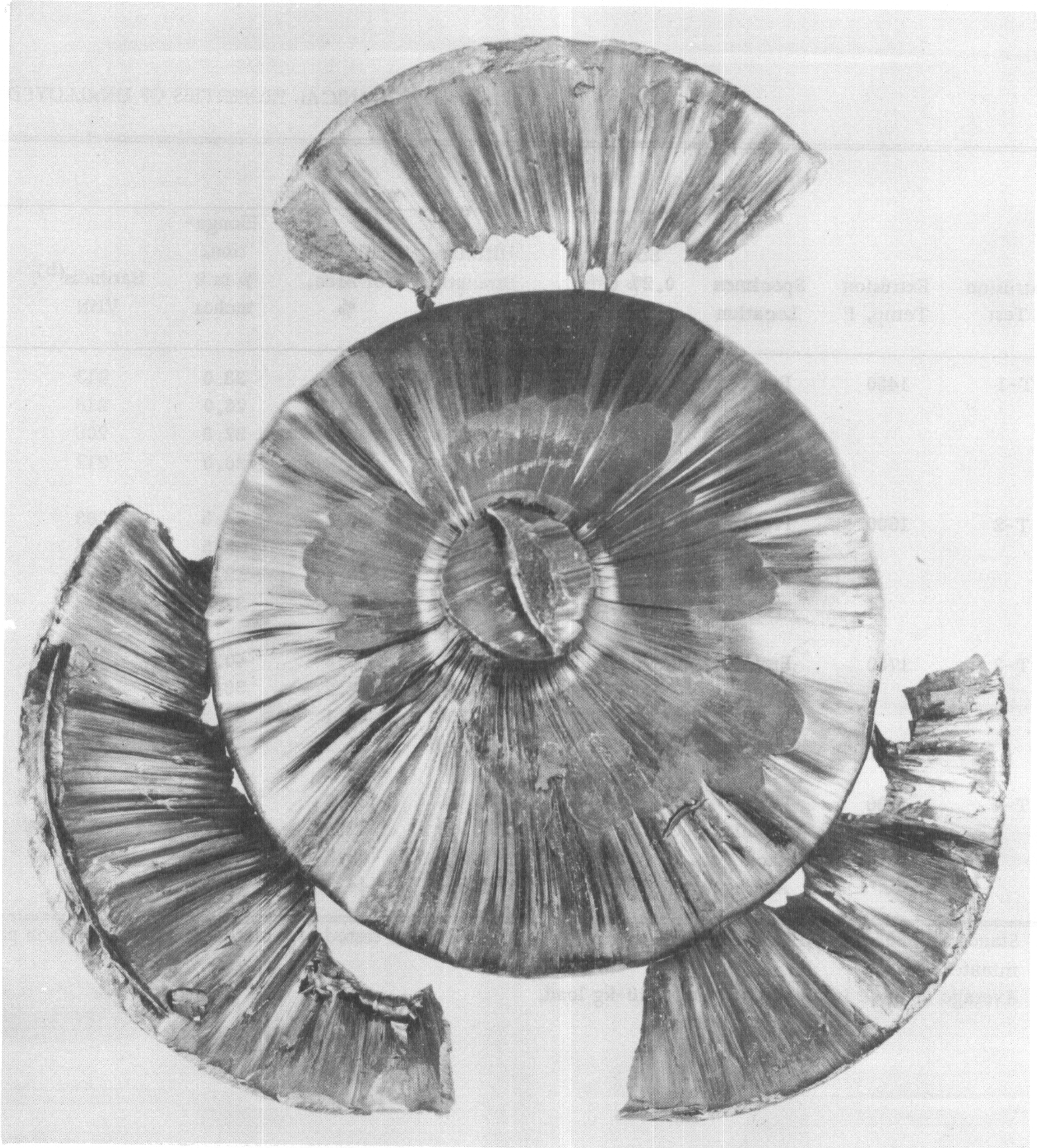
Unalloyed Titanium

Two tensile specimens were removed from the front, middle, and back sections of each unalloyed titanium extrusion. One specimen from each section was tested in the as-extruded condition, and the other was process annealed for 1 hour at 1500 F and air cooled. The mechanical properties of the bars are presented in Table 1.

In the as-extruded condition, the ultimate tensile strength decreased from 87,000 to 68,500 psi as the extrusion temperature increased from 1450 to 1900 F. There was very little difference, however, between the properties of the bars extruded in the alpha-phase region at 1450 and at 1600 F. This was also true for the bars extruded in the beta-phase region at 1750 and 1900 F, the marked change in properties occurring between 1600 and 1750 F. Annealing for 1 hour at 1500 F and air cooling did not produce any significant difference in the mechanical properties of the bars. The effect of extrusion temperature on the average mechanical properties is shown graphically in Figure 7.

The bars extruded at 1450 F and 1600 F exhibited a duplex structure consisting of alternate concentric rings of fine and coarse grains that apparently are formed by variations in the deformation that the layers of metal undergo from the center of the bar to the surface. The bars extruded in the beta-phase region at 1750 and 1900 F, on the other hand, exhibited a uniform structure with only slight flow markings. Macroetched sections of the bars in the as-extruded conditions, illustrating the differences in flow pattern and grain structure above and below the transformation temperature, are shown in Figure 8.

Metallographic examination of the bars indicated that only partial recrystallization occurred at extrusion temperatures of 1450 and 1600 F, whereas complete recrystallization, by virtue of the allotropic



N11246

FIGURE 6. SKULL FROM Ti-3Mn-COMPLEX BILLET EXTRUDED AT 1450 F, SHOWING SEPARATION AT FLOW SURFACE

Note striations on flow surface and at radius of die opening.

TABLE 1. MECHANICAL PROPERTIES OF UNALLOYED

Extrusion Test	Extrusion Temp, F	Specimen Location	As Extruded Yield Strength, 0.2% Offset, psi	Ultimate Strength, psi	Reduction of Area, %	Elongation, % in 2 inches	Hardness$^{(b)}$, VHN
T-1	1450	Front	61,000	82, 500	45.8	33.0	213
		Middle	74,000	94, 500	38.8	26.0	216
		Back	62,500	83, 500	49.8	32.0	206
		Average	66,000	87,000	44.8	30.0	212
T-2	1600	Front	62,000	75,000	50.3	24.5	203
		Middle	64,000	77,000	49.2	38.5	208
		Back	80,500	95, 500	46.0	32.5	226
		Average	69,000	82, 500	45.2	32.0	212
T-3	1750	Front	52,000	65,500	53.3	40.0	174
		Middle	56,500	69, 500	52.2	39.5	182
		Back	55,500	77, 500	48.1	34.5	185
		Average	55,000	71,000	51.2	38.0	180
T-4	1900	Front	57,500	71, 500	36.3	30.5	187
		Middle	55,000	66, 500	42.8	37.5	172
		Back	55,000	67, 500	53.3	39.5	172
		Average	56,000	68, 500	44.1	36.0	177

(a) Standard 0.505-inch-diameter tensile specimens. All specimens tested at a platen speed of 0.04 inch per minute.

(b) Average of three impressions, using a 10-kg load.

PROCESS A TITANIUM EXTRUSIONS(a)

Annealed 1 Hr at 1500 F, Air Cooled				
Yield Strength, 0.2% Offset, psi	Ultimate Strength, psi	Reduction of Area, %	Elongation, % in 2 inches	Hardness(b), VHN
55,500	77,000	44.9	32.5	187
70,000	89,500	44.9	30.0	208
60,500	79,500	42.2	29.0	198
62,000	82,000	44.0	30.5	198
63,500	74,000	49.5	30.5	186
69,000	81,000	46.9	29.0	216
82,500	97,000	44.3	31.0	230
72,000	84,000	46.9	30.0	211
59,000	69,500	28.5	27.0	174
51,000	71,500	47.2	35.5	178
47,500	71,500	40.4	28.5	167
52,500	71,000	38.7	30.5	173
61,000	74,000	37.9	30.5	192
53,000	66,500	52.5	38.0	171
59,000	71,500	39.1	34.0	171
58,000	71,000	43.2	34.0	178

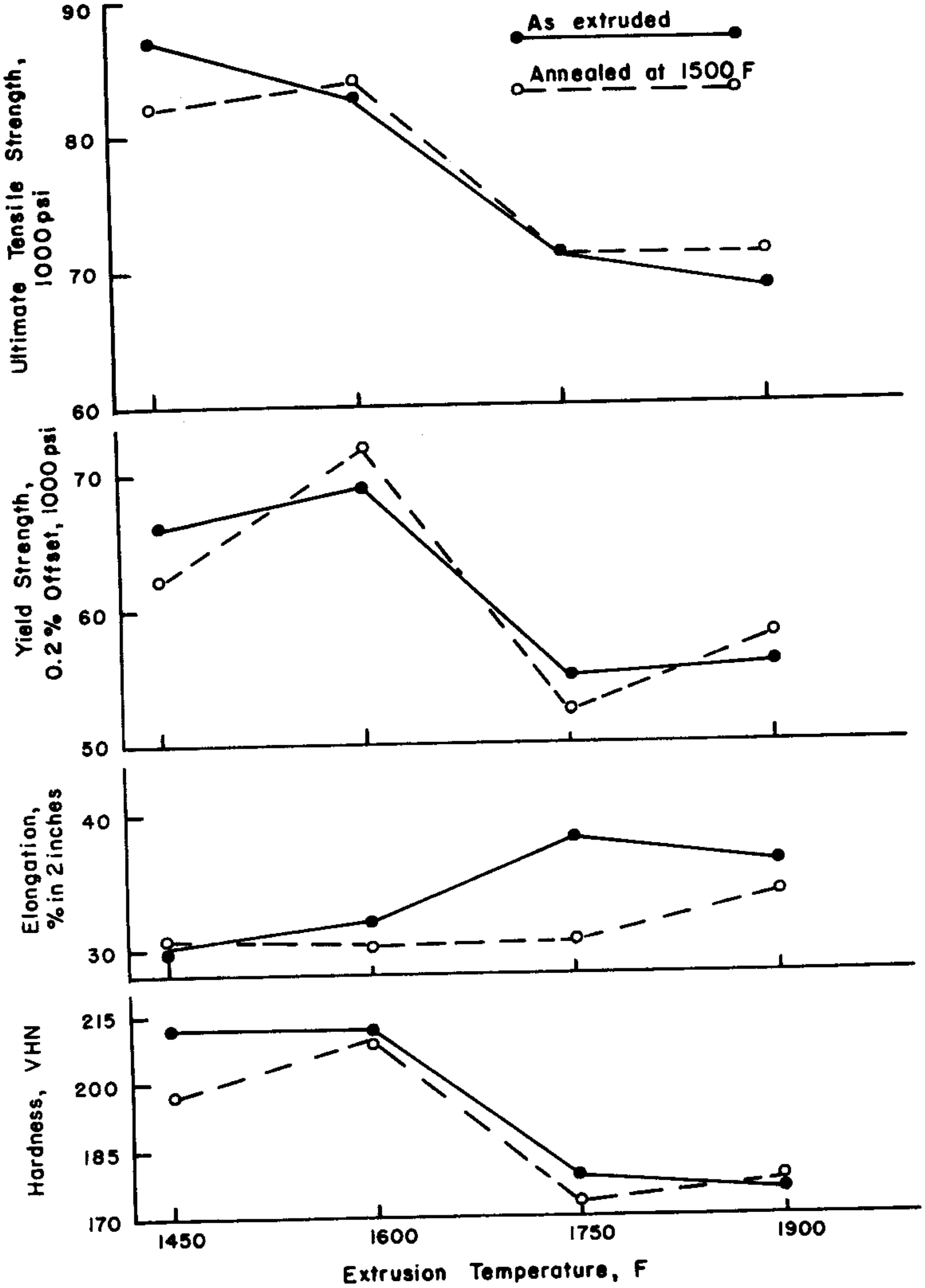
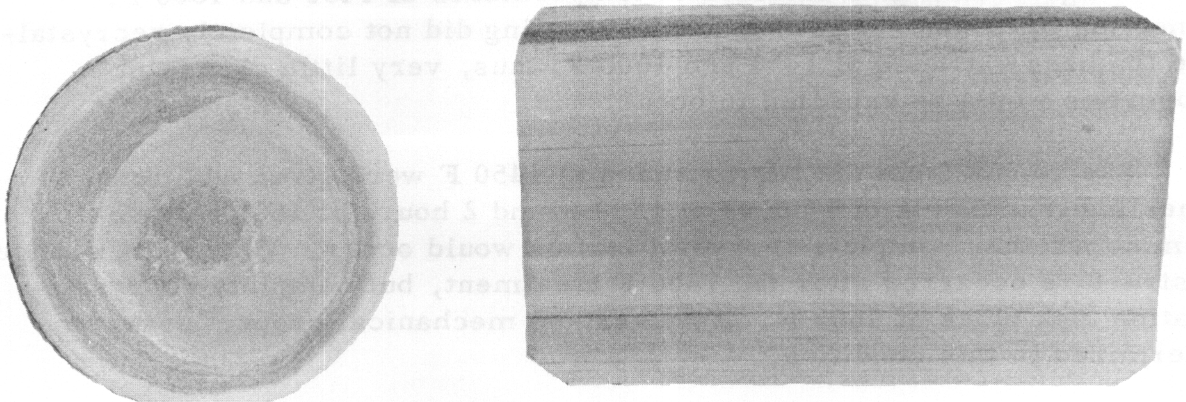


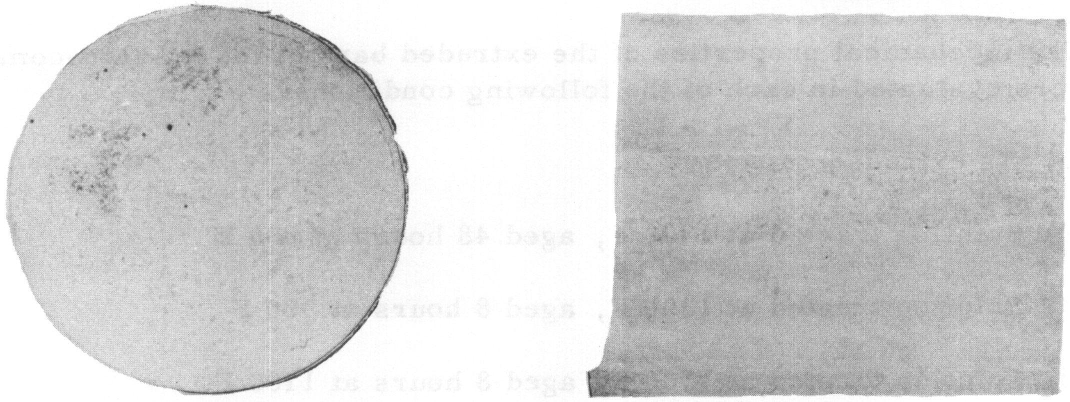
FIGURE 7. EFFECT OF EXTRUSION TEMPERATURE ON THE AVERAGE MECHANICAL PROPERTIES OF AS-EXTRUDED AND ANNEALED UNALLOYED TITANIUM BARS



2X N14710 2X N14711

Cross Section of Bar
Extruded at 1450 F

Longitudinal Section of Bar
Extruded at 1600 F



2X N14987 2X N14988

Cross Section of Bar
Extruded at 1750 F

Longitudinal Section of Bar
Extruded at 1900 F

FIGURE 8. MACROETCHED SECTIONS OF THE UNALLOYED TITANIUM BARS IN THE VARIOUS AS-EXTRUDED CONDITIONS

Note differences in flow markings and grain structures.

transformation, occurred in the bars extruded at 1750 and 1900 F. Thus, residual stresses, strain hardening, or strain aging could be the reason for the higher strengths obtained at temperatures of 1450 and 1600 F. Annealing for 1 hour at 1500 F and air cooling did not completely recrystallize the bars extruded at 1450 and 1600 F; thus, very little change in properties would be expected to occur.

Specimens from the bar extruded at 1450 F were given additional annealing treatments of 4 hours at 1500 F and 2 hours at 1600 F to determine whether complete recrystallization would occur. Very little change in structure occurred after the 1500 F treatment, but complete recrystallization took place at 1600 F. However, no mechanical properties were determined in this condition.

Photomicrographs of the bars extruded at 1450 and 1900 F, in the as-extruded and annealed conditions, are shown in Figure 9. These microstructures are typical of the bars extruded at 1600 and 1750 F, respectively, except for slight variations in grain size.

Ti-3Mn-Complex Alloy

The mechanical properties of the extruded bars of the Ti-3Mn-complex alloy were evaluated in each of the following conditions:

(1) As extruded

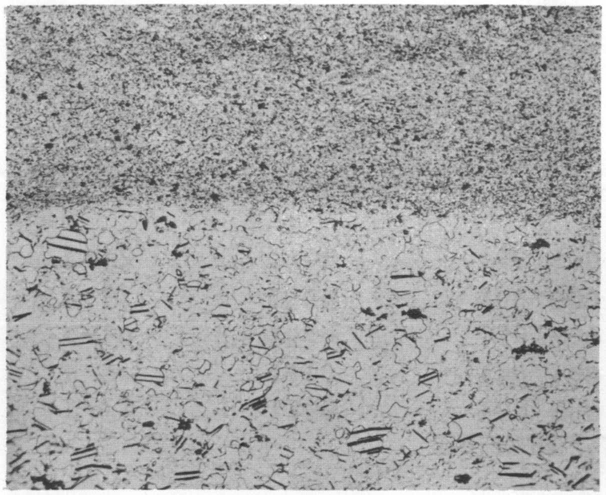
(2) Solution treated at 1300 F, aged 48 hours at 800 F

(3) Solution treated at 1300 F, aged 8 hours at 900 F

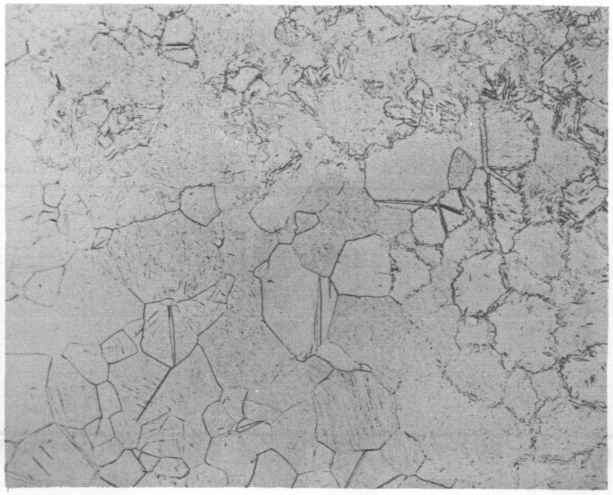
(4) Solution treated at 1300 F, aged 8 hours at 1100 F.

Two specimens were removed from the front end of each bar to determine the as-extruded properties, since this part of the bar suffers the least temperature variation during extrusion. Duplicate specimens from various positions along the length of the bars were used to determine the properties after heat treatment. The mechanical properties of the bars in the various conditions are presented in Table 2.

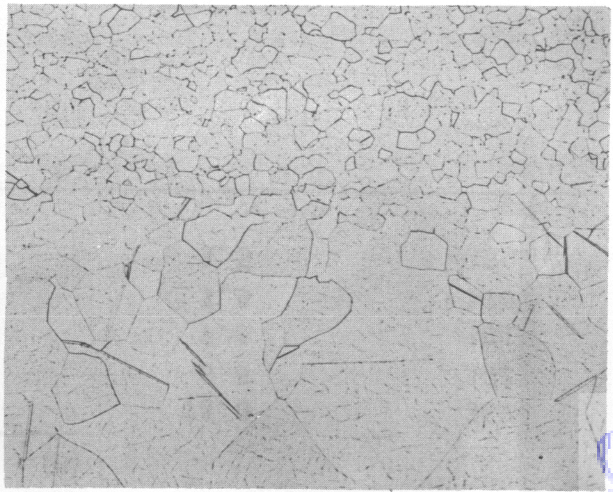
As extruded at 1350 F, the Ti-3Mn-complex alloy exhibited a tensile strength of 183,000 psi, with 10.5 per cent elongation in 2 inches. Increasing the extrusion temperature to 1450 F increased the strength to 200,000 psi, but lowered the elongation to 3.5 per cent. The specimens removed from the bars extruded above 1450 F broke in the threads with a brittle fracture. Metallographic examination of the bars in the as-extruded condition indicated that the alloy was extruded in the beta-phase region at all of the temperatures used. The microstructure of the bars consisted of equiaxed beta grains containing primary alpha in a Widmanstätten



As Extruded at 1450 F

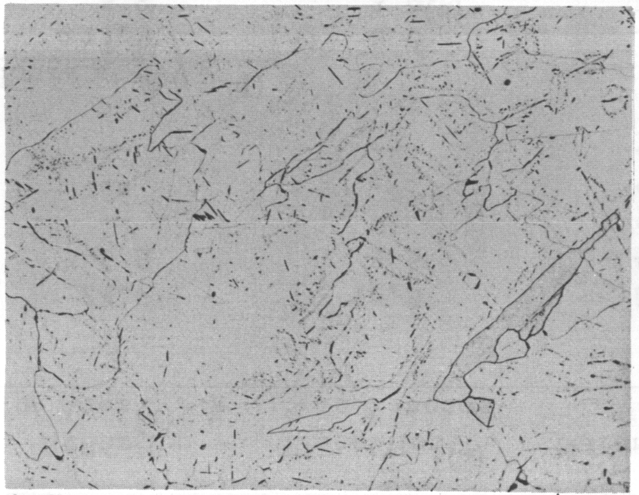


Extruded at 1450 F, Annealed 1 Hour
at 1500 F, and Air Cooled



Extruded at 1450 F, Annealed 2 Hours
at 1600 F, and Air Cooled

As Extruded at 1900 F



Extruded at 1900 F, Annealed for 1 Hour
at 1500 F, and Air Cooled

FIGURE 9. MICROSTRUCTURES OF THE UNALLOYED TITANIUM BARS EXTRUDED AT 1450 AND 1900 F IN THE AS-EXTRUDED AND ANNEALED CONDITIONS

TABLE 2. MECHANICAL PROPERTIES OF THE Ti-3Mn-COMPLEX ALLOY IN THE AS-EXTRUDED AND HEAT-TREATED CONDITIONS

Extrusion Test	Extrusion Temp, F	Yield Strength(a), 0.2% Offset, psi	Ultimate Strength(a), psi	Reduction of Area(a), %	Elongation(a), % in 2 inches	Hardness(b), VHN
			As Extruded			
T-13	1350	169,000(c)	183,000	24.7	10.5	375
T-9	1450	190,500(c)	200,000	5.6	3.5	409
T-10	1525	--	(e)	--	--	417
T-11	1600	--	(e)	--	--	403
T-12	1700	--	(e)	--	--	401
		Solution Treated at 1300 F, Aged 48 Hours at 800 F(d)				
T-13	1350	156,500	182,000	11.9	8.0	387
T-9	1450	--	180,500	5.8	3.0(d)	382
T-10	1525	--	183,000	10.1	7.0	388
T-11	1600	148,500(c)	174,500	9.6	6.5	388
T-12	1700	150,500	178,500	3.7	2.5	402
		Solution Treated at 1300 F, Aged 8 Hours at 900 F(d)				
T-13	1350	150,000	172,000	25.0	13.0	367
T-9	1450	154,500	173,000	17.7	8.5	392
T-10	1525	153,500	173,500	11.5	7.5	381
T-11	1600	150,000	169,500	10.5	7.5	370
T-12	1700	146,000	166,500	4.5	3.5	359

TABLE 2. (Continued)

Extrusion Test	Extrusion Temp, F	Yield Strength[(a)], 0.2% Offset, psi	Ultimate Strength[(a)], psi	Reduction of Area[(a)], %	Elongation[(a)], % in 2 inches	Hardness[(b)], VHN
		Solution Treated at 1300 F, Aged 8 Hours at 1100 F[(d)]				
T-13	1350	120,000	131,000	44.3	25.0	274
T-9	1450	123,000	131,500	37.9	23.5	297
T-10	1525	122,500	130,000	28.4	20.0	295
T-11	1600	120,000	129,000	31.3	21.5	297
T-12	1700	120,000	128,500	23.2	18.5	302

(a) Standard 0.505-inch-diameter tensile specimens. Tested at a platen speed of 0.04 inch per minute. Average of two values, except where noted.

(b) Average of three impressions, using a 10-kg load.

(c) Single values.

(d) Heated at solution temperature for 1 hour and water quenched.

(e) Specimens broke in threads.

configuration, the size of the beta grains increasing as the extrusion temperature was increased. Since the beta transus for this material was lower than normally expected for the Ti-3Mn-complex alloy because of higher alloy content, the heat generated during extrusion was apparently sufficient to raise the temperature of the bars into the beta region. Photomicrographs of the bars extruded at 1350 and 1700 F, illustrating the variation in grain size, are shown in Figure 10. The embrittlement occurring above 1450 F may be caused by the increase in beta-grain size, since the hardnesses of the bars extruded between 1450 and 1700 F were essentially the same.

Solution treating at 1300 F, followed by overaging at 800, 900, and 1100 F for various lengths of time, produced strength levels of 180,000, 170,000, and 130,000 psi, respectively. The elongation and reduction-of-area values obtained in each of the overaged conditions, however, were in inverse proportion to the extrusion temperature. In each condition tested, the best combination of strength and ductility was attained by extruding at 1350 F.

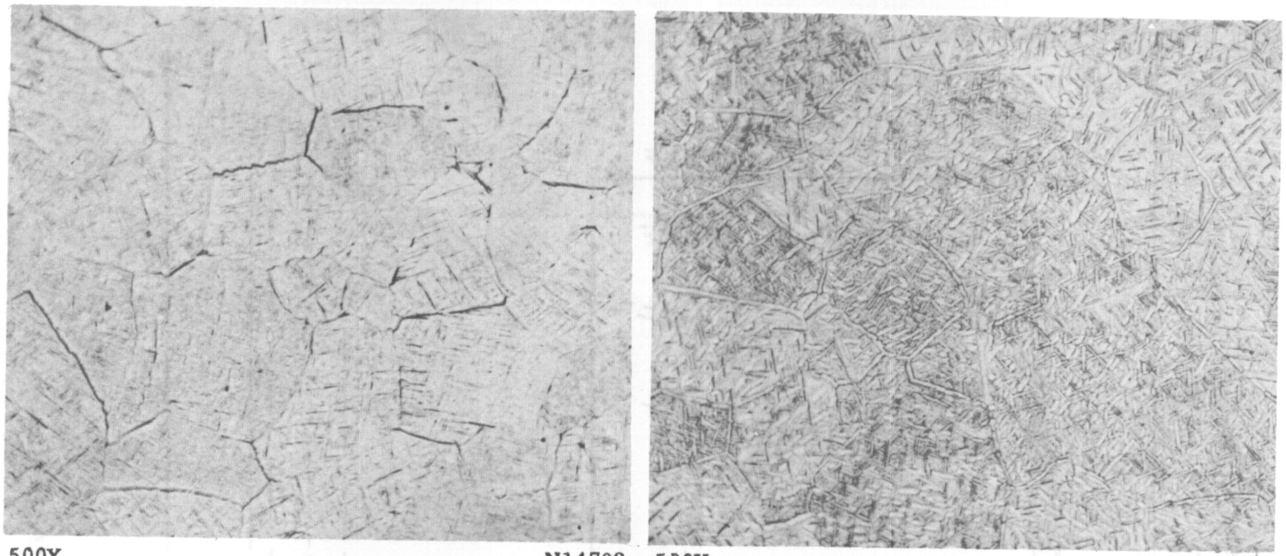
It is interesting to note the effect of overaging on the yield ratio. Overaging for 48 hours at 800 F produced an average ultimate strength of 180,000 psi and an average yield strength of 153,000 psi. Overaging for 8 hours at 900 F lowered the ultimate strength to 170,000 psi, but the yield strength remained the same. This phenomenon has been observed in other work at Battelle on titanium-alloy development, but the cause has not yet been definitely determined.

The microstructures of the bars in the overaged conditions consisted of equiaxed beta grains with primary grain boundary and Widmanstätten alpha. Alpha was also precipitated in a fine dispersion in the beta matrix within the grains. The amount of precipitated alpha increased as the aging temperature increased. Photomicrographs of the bars extruded at 1350 and 1700 F, solution treated at 1300 F, and aged for 8 hours at 1100 F, illustrating the typical structures, also are shown in Figure 10.

Extrusion Pressure and Work of Extrusion

The effect of temperature on the extrusion pressure was determined in the tests on the Ti-3Mn-complex alloy. The hydraulic pressure on the main cylinder was recorded as a function of the ram travel and converted to the pressure acting at the ram. The extrusion-pressure curves are presented in Figure 11.

As the temperature was increased from 1350 to 1700 F, the break-through pressure decreased from 107,000 psi to 71,000 psi. The run-out pressures were in approximately the same order, but increased to about 105,000 psi near the end of the extrusion stroke.

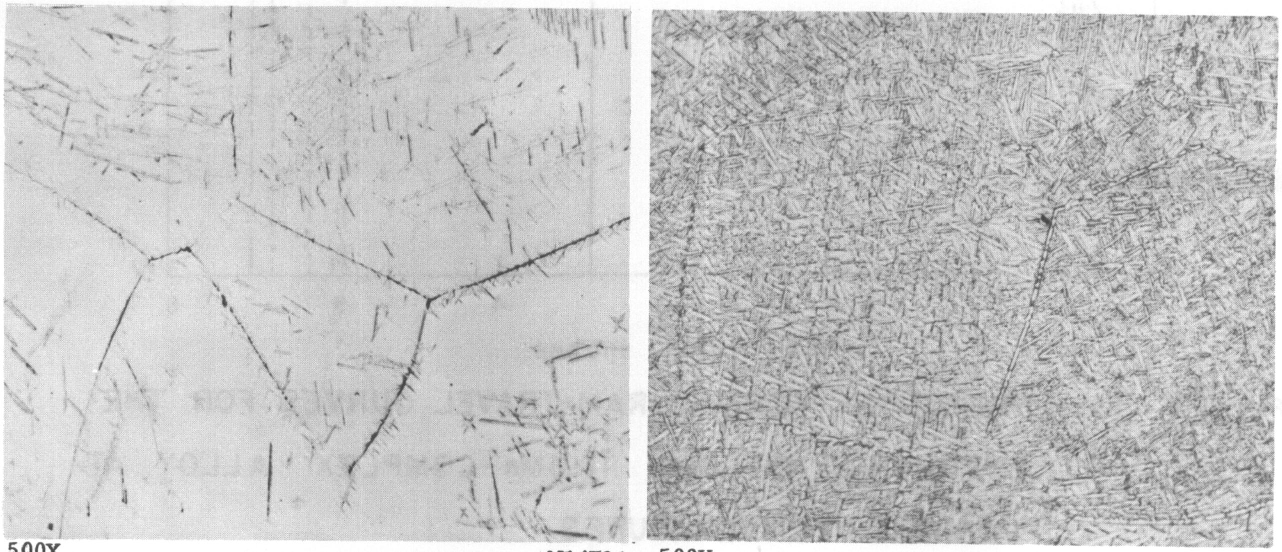


500X N14703

As Extruded at 1350 F

500X N14702

Extruded at 1350 F, Solution Treated at 1300 F. and Aged 8 Hours at 1100 F



500X N14701

As Extruded at 1700 F

500X N14700

Extruded at 1700 F, Solution Treated at 1300 F, and Aged 8 Hours at 1100 F

FIGURE 10. MICROSTRUCTURES OF THE Ti-3Mn-COMPLEX BARS EXTRUDED AT 1350 AND 1700 F IN THE AS-EXTRUDED AND HEAT-TREATED CONDITIONS

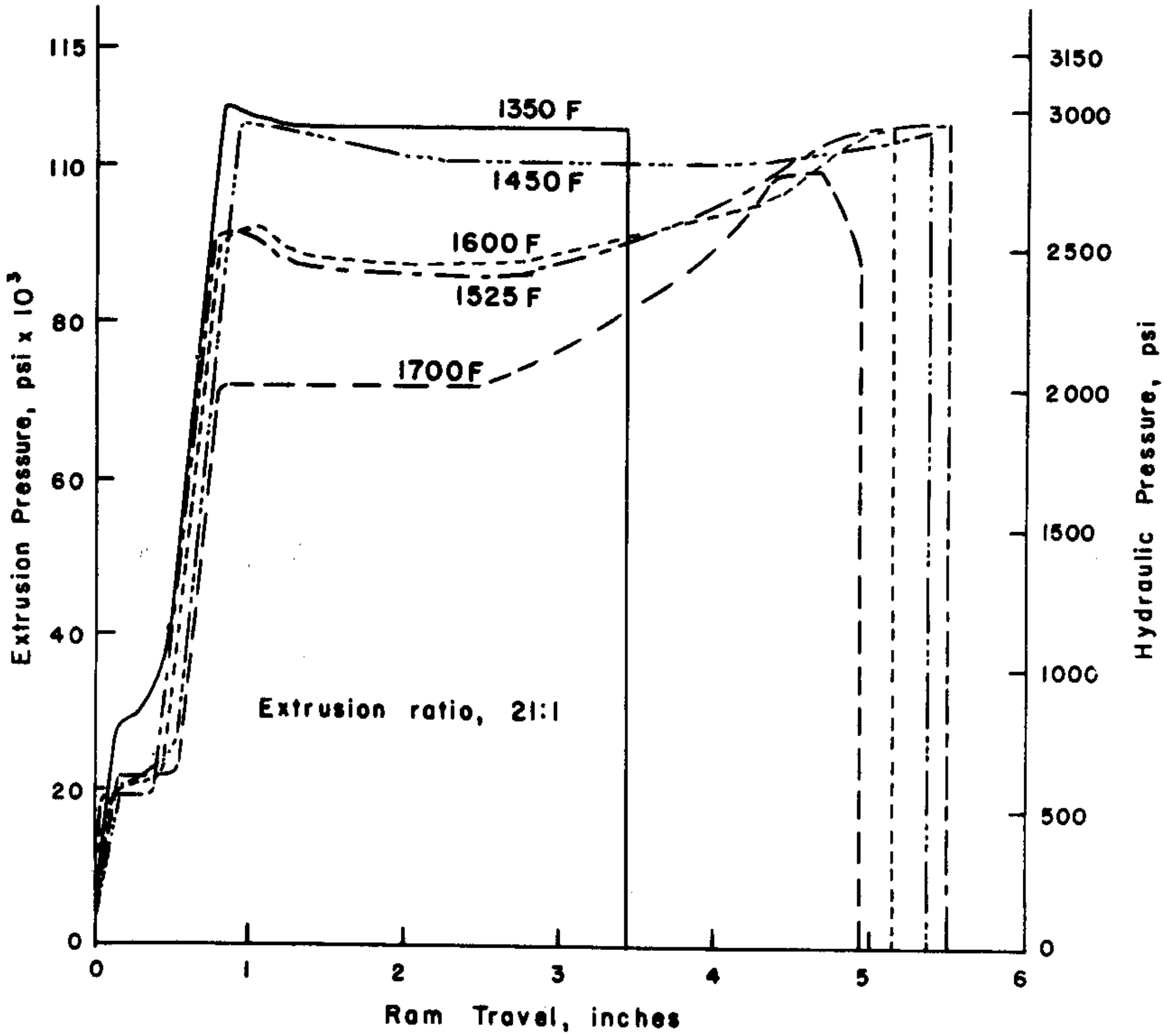


FIGURE 11. PRESSURE VERSUS RAM-TRAVEL CURVES FOR THE EXTRUSION OF THE Ti-3Mn-COMPLEX ALLOY AT VARIOUS TEMPERATURES

The areas under the curves in Figure 11 represent the work required for extrusion at the various temperatures. These values were determined by graphical integration, using a polar planimeter, and converted to work per unit volume. The data for these calculations are presented in Table 3. The effect of temperature on the work of extrusion is shown graphically in Figure 12.

TABLE 3. WORK OF EXTRUDING THE Ti-3Mn-COMPLEX ALLOY AT VARIOUS EXTRUSION TEMPERATURES

Test	Extrusion Temp, F	Work, in-lb	Volume Extruded, $in.^3$	Average Work/Unit Volume, $in\text{-}lb/in.^3$
T-13	1350	265×10^4	52.6	50,300
T-9	1450	415×10^4	85.0	48,900
T-10	1525	394×10^4	86.5	45,400
T-11	1600	348×10^4	78.9	44,100
T-12	1700	299×10^4	77.5	38,500

STUDIES ON THE EFFECT OF DIE DESIGN

The extrusion tests with flat-face dies described in the preceding section show that the flow surface occurs at some angle to the die face, producing a stagnant zone of metal at the shoulder of the die. Hence, laminations and surface defects occur in the extruded bar, and excessive scrap losses result. It was believed that the use of a conical die having the proper included angle would eliminate the stagnant-metal zone and thereby improve the quality of the extruded bar. Consequently, a series of tests was conducted to determine the effect of die angle on the flow of metal during extrusion.

Conditions of Tests

Four unalloyed titanium billets were extruded at 1600 F through dies having included angles of 180 (flat face), 160, 140, and 120 degrees. The extrusion conditions were as follows:

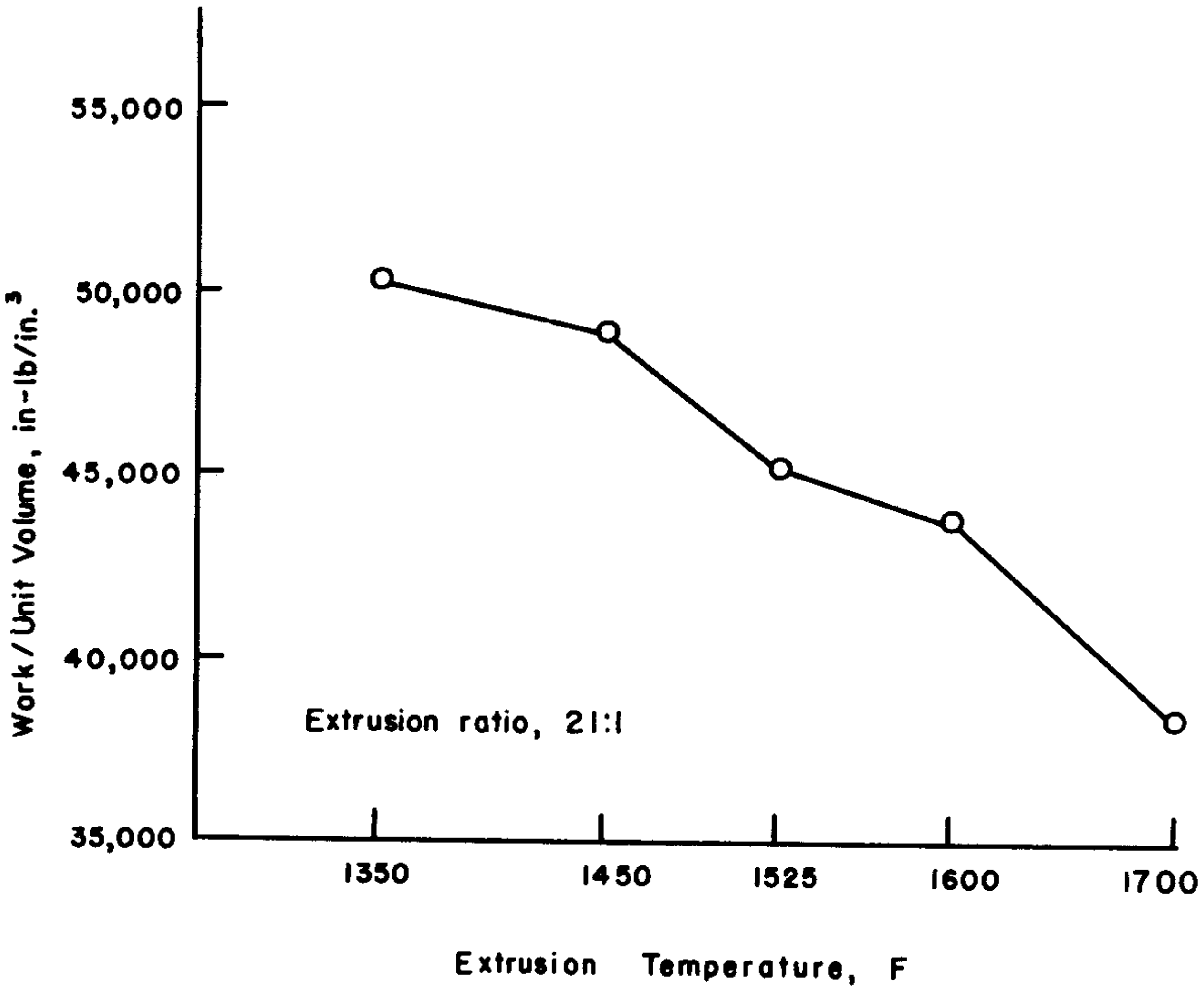


FIGURE 12. EFFECT OF TEMPERATURE ON THE WORK OF EXTRUSION FOR THE Ti-3Mn-COMPLEX ALLOY

Test	Die Angle, degrees	Billet	Extrusion Time, sec
T-14	180	58	13
T-15	160	59	12
T-16	140	60	10
T-17	120	61	15

Carpenter 883 steel dies and Fiske Hot Die Lubricant No. 514-A were also used for these tests. The dies had a 3/16-inch radius at the die opening, and the bearing length was 3/16 inch.

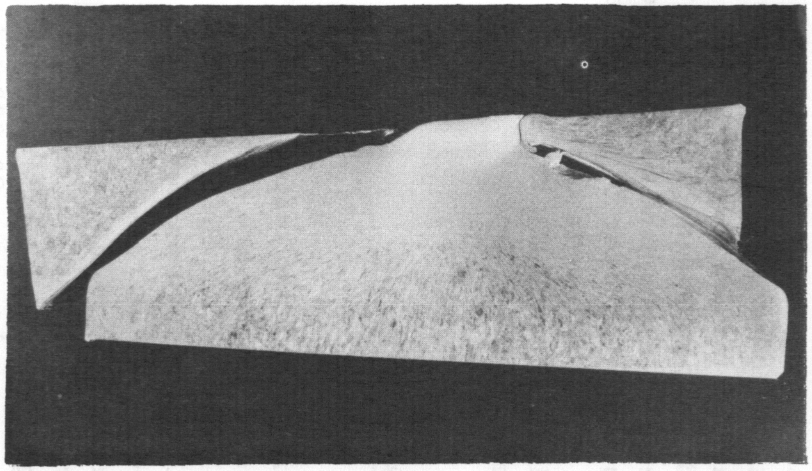
Die wear was very slight in these tests, but a build-up of titanium occurred at the radius of the die opening on the flat-faced die and, to a lesser extent, on the 160-degree die. No build-up occurred on the 140- and 120-degree dies, indicating that better lubrication of the die face was attained with the smaller included angles.

The back third of the bar extruded through the flat-faced die had a badly scored surface. A slight improvement was obtained with the 160- and 120-degree dies, but a good surface over nearly the entire bar length was achieved with the 140-degree die.

The billet skulls were sectioned and macroetched to show the flow pattern that occurred at the various die angles; they are shown in Figure 13. The flat-face die produced a stagnant metal zone about an inch long at the billet wall. Complete separation occurred at the flow surface, which was coated with the graphite lubricant that was carried inward from the container wall. The stagnant zone also occurred with the 160-degree die, but had a reduced thickness. With the 140-degree die, a stagnant zone started to form only on the lower part of the billet, possibly because of inadequate lubrication of the die face or greater cooling at the bottom in contact with the container. The 120-degree die completely eliminated the stagnant-metal zone, the flow of metal being uniform across the billet.

Extrusion Pressure and Work of Extrusion

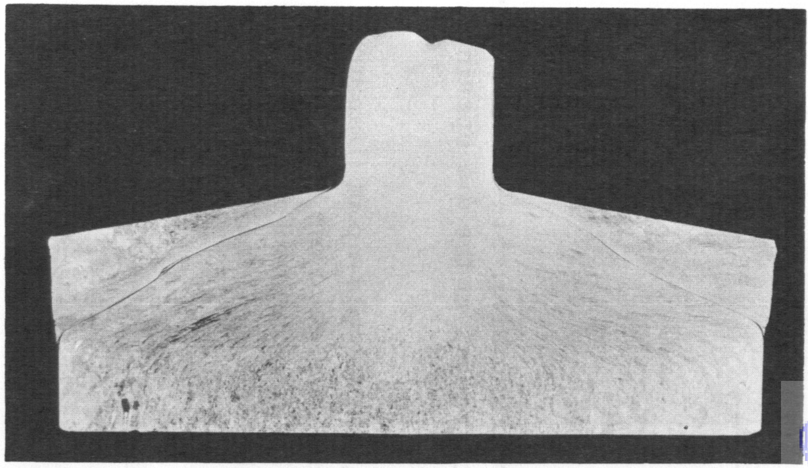
The extrusion pressure was measured in each of the tests. Curves of pressure versus ram travel for the various die angles are shown in Figure 14. Break-through pressures were 73,000 psi for the 180-degree (flat face) die, 62,000 psi for the 160-degree die, 47,000 psi for the 140-degree die, and 65,000 psi for the 120-degree die. The run-out pressures for the 180-, 160-, and 140-degree dies were roughly in the same proportion as the break-through pressures, increasing at the end of the extrusion stroke to about 80,000 psi. During extrusion through the 120-degree die, however, the pressure sharply increased to over 80,000 psi.



N14712

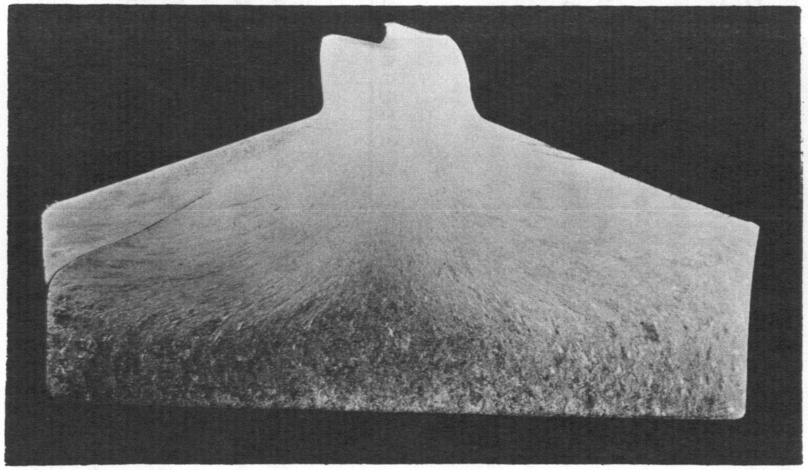
Test 14 Flat-Faced Die

Note separation of skull at flow surface



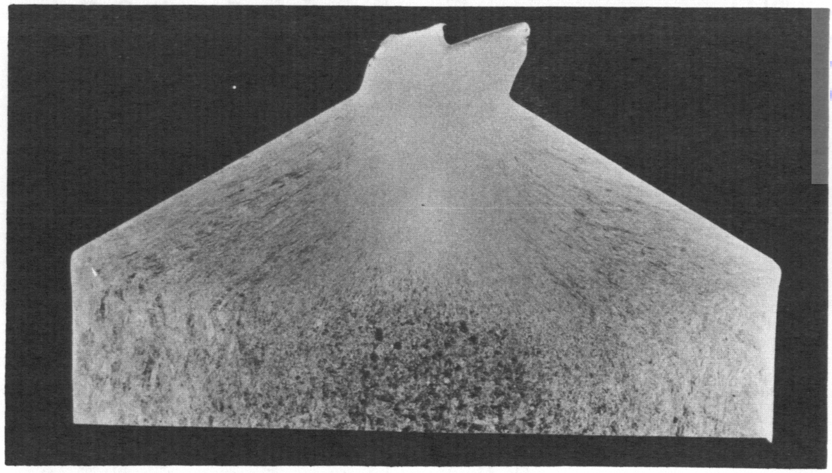
N13526

Test 15 160-Degree Die



N13527

Test 16 140-Degree Die



N13528

Test 17 120-Degree Die

FIGURE 13. ETCHED SECTIONS OF UNALLOYED BILLET SKULLS SHOWING THE EFFECT OF CONICAL DIES ON THE METAL FLOW DURING EXTRUSION

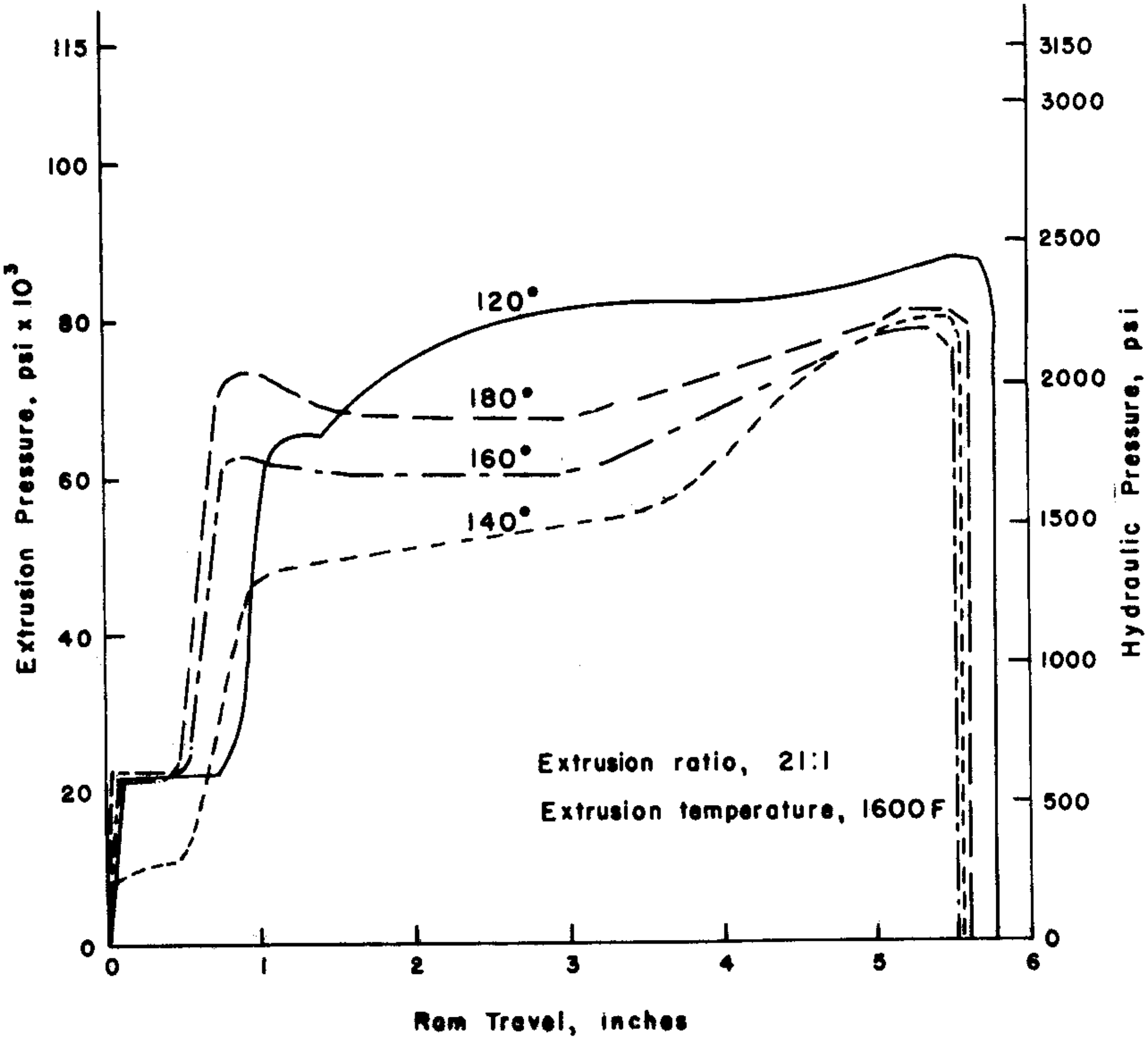


FIGURE 14. PRESSURE VERSUS RAM-TRAVEL CURVES FOR THE EXTRUSION OF UNALLOYED TITANIUM THROUGH FLAT-FACE AND CONICAL DIES

The work of extrusion required for each die angle was calculated from the pressure data obtained in the tests. Data for these calculations are presented in Table 4. The average work per unit volume of extruded metal progressively decreased as the die angle was decreased from 180 to 140 degrees. At a die angle of 120 degrees, however, the work per unit volume was greater than that for the 180-degree (flat-face) die. The effect of die angle on the work per unit volume is shown graphically in Figure 15.

TABLE 4. WORK OF EXTRUDING UNALLOYED TITANIUM AT 1600 F WITH FLAT-FACE AND CONICAL DIES

Test	Die Angle, degrees	Work, in-lb	Volume Extruded, in.^{3}	Average Work/Unit Volume, in-lb/in.^{3}
T-14	180	306×10^4	86.5	35,400
T-15	160	284×10^4	86.5	32,800
T-16	140	245×10^4	86.5	28,400
T-17	120	343×10^4	89.5	38,400

These calculations indicate that the force required to accomplish deformation decreases as the included die angle decreases from 180 degrees (flat-face die) to some critical angle between 140 and 120 degrees. As the die angle decreases, the rate of deformation of the metal moving through the die also decreases, thus reducing the force required. Below this critical angle, the decrease in force required for deformation is more than offset by the increased friction surface in the die.

Mechanical Properties and Microstructures

Tensile specimens were removed from each of the bars for determining the mechanical properties (1) in the as-extruded condition and (2) after annealing for 1 hour at 1500 F and air cooling. The properties of the bars in these conditions are presented in Table 5. The die angle had no significant effect on the strength or ductility of the extruded bars. The approximate values for each test were 90,000-psi ultimate strength, 67,000-psi yield strength, 41 per cent reduction of area, and 29.5 per cent elongation in 2 inches. These values are in close agreement with the properties of the extruded unalloyed titanium billets that were prepared at Battelle and used in Tests T-1 and T-2 described earlier in this report. Annealing for 1 hour at 1500 F and air cooling did not produce any significant change in the properties.

The typical microstructures of the bars in the as-extruded and annealed conditions are shown in Figure 16. No banding or layering effect

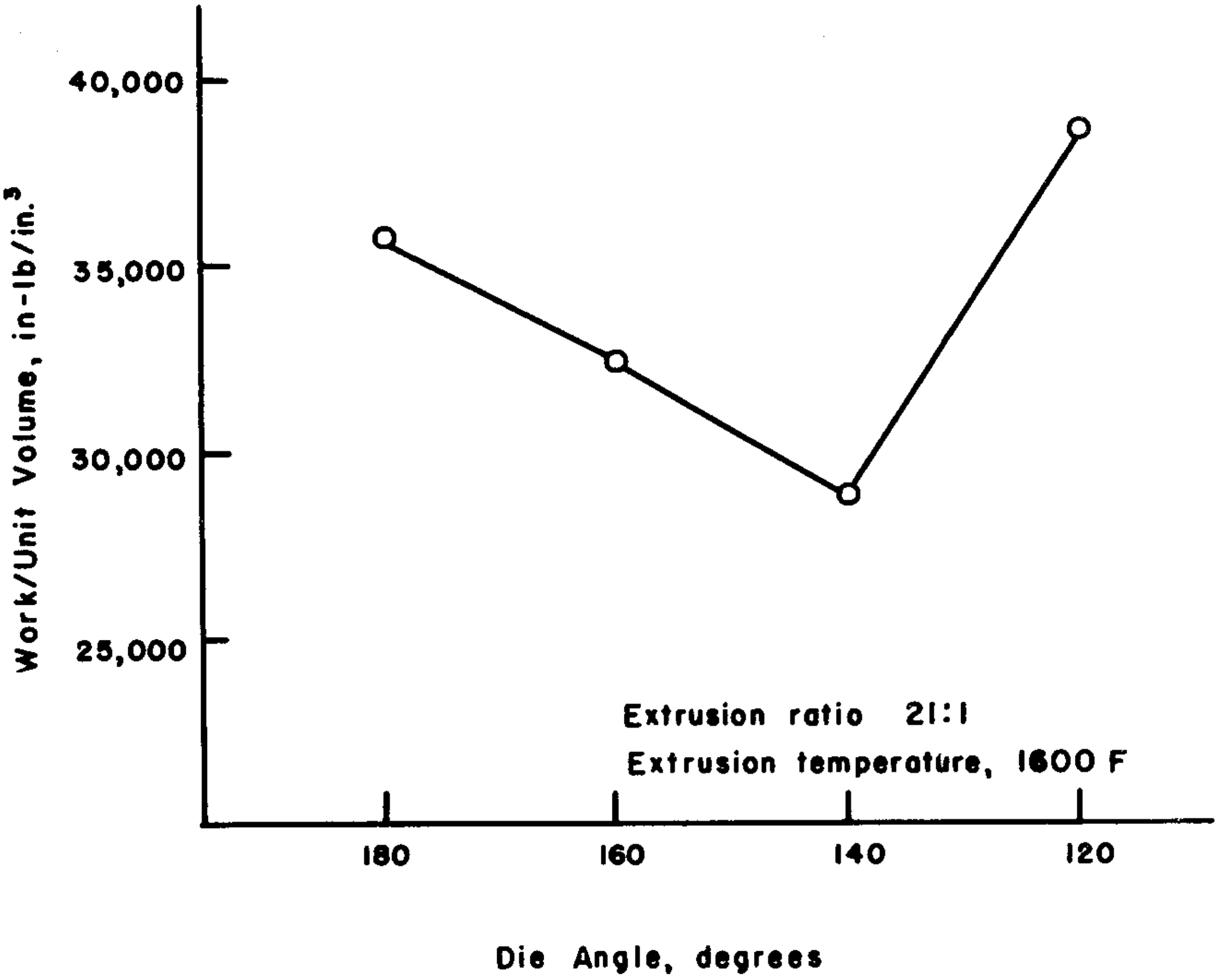


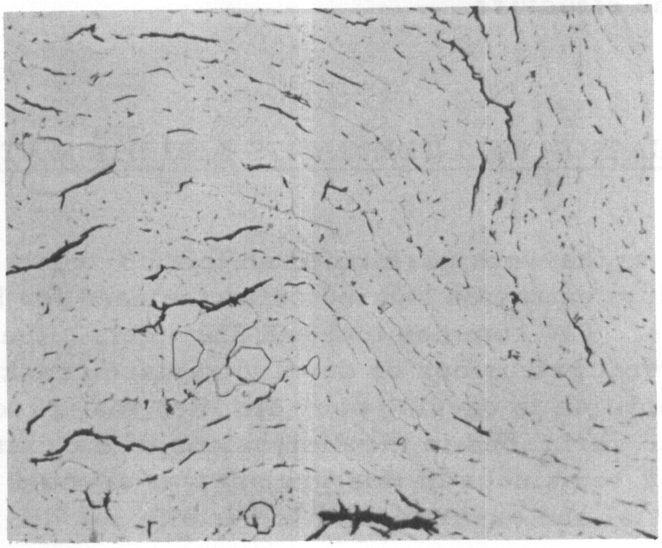
FIGURE 15. EFFECT OF DIE ANGLE ON WORK OF EXTRUSION FOR UNALLOYED TITANIUM

TABLE 5. MECHANICAL PROPERTIES OF THE UNALLOYED TITANIUM BARS EXTRUDED AT 1600 F THROUGH FLAT-FACE AND CONICAL DIES

Test	Die Angle, degrees	Yield Strength[a], 0.2% Offset, psi	Ultimate Strength[a], psi	Reduction of Area[a], %	Elongation[a], % in 2 inches	Hardness[b], VHN
			As Extruded			
T-14	180	68,000	90,500	40.7	27.0	205
T-15	160	64,000	89,500	39.1	29.5	227
T-16	140	66,000	87,000	44.7	32.0	213
T-17	120	69,500	92,000	42.3	29.5	205
		Annealed 1 Hour at 1500 F and Air Cooled				
T-14	180	65,500	92,500	41.4	27.0	209
T-15	160	66,500	89,500	42.2	28.0	198
T-16	140	65,500	90,000	37.1	27.5	209
T-17	120	65,500	89,500	41.3	27.5	199

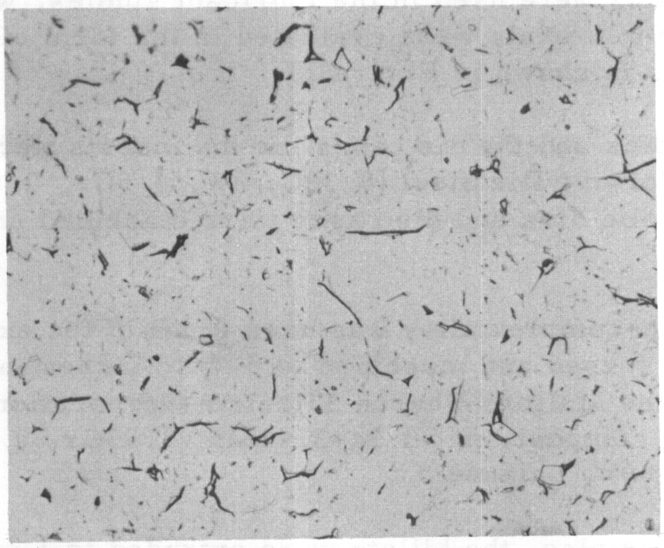
(a) Standard 0.505-inch-diameter tensile specimens. Tested at a platen speed of 0.04 inch per minute. Values are average of two specimes.

(b) Average of three impressions, using a 10-kg load.


500X N14708

As Extruded at 1600 F

Note orientation of the grains


500X N14707

Extruded at 1600 F, Annealed 1 Hour at 1500 F, and Air Cooled

FIGURE 16. MICROSTRUCTURE OF MST UNALLOYED TITANIUM BAR IN THE AS-EXTRUDED AND ANNEALED CONDITIONS

was observed, as in the unalloyed bars from Tests T-1 and T-2 that were extruded at 1450 and 1600 F. Annealing for 1 hour at 1500 F only partially recrystallized the structure.

EVALUATION OF LUBRICANTS AND DIE MATERIALS

In addition to the work performed on this contract, work by others in the field of titanium extrusion has indicated the need for better lubricants and die materials. The common tool and die steels either are severely eroded after several extrusions or develop surface cracks. Either of these two effects, in addition to causing poor die life, may produce a poor surface finish and variations in the dimensions of the extruded shape. Consequently, the remainder of this project was devoted to evaluating various commercial and experimental lubricants and die materials.

The dies used in these studies had a conical-entry angle of 130 degrees, a 1/2-inch radius at the die opening, and a 3/16-inch bearing length. On the basis of the results obtained earlier with the conical dies, it was believed that this design would produce the optimum flow pattern, require minimum pressure, and provide the best lubrication at the die opening. Solid dies were used in the lubricant studies, whereas the experimental die materials were evaluated in the form of die inserts. The design of the dies is shown in Figure 17.

The solid dies and the die cases for the inserts were made of Firth Sterling L. T. Forging Die Steel (0.33C, 9W, 3.5Cr, 0.50V), hardened to 50 R_C. The solid dies and die cases were machined and heat treated at Battelle.

The Ti-3Mn-complex alloy was used in all of the extrusion tests. The billet stock, forged and machined to 4-7/16 inches in diameter, was purchased from the Mallory-Sharon Titanium Corporation. This material had the following composition: 3.34-3.42Mn, 1.07Cr, 0.93Fe, 0.99-1.01Mo, 1.03-1.09V, balance Ti.

In these tests also, the billets were extruded to 1-inch-diameter bars (21:1 extrusion ratio). Higher extrusion ratios were not employed because of the limited capacity of the press. Billet temperatures higher than would be employed normally for the Ti-3Mn-complex alloy were used to provide a more rigorous test of the lubricants and die materials. As will be shown in the following sections, the hot billet was in contact with the die face for greater periods of time under these test conditions than would occur if a faster press had been used, and the conditions imposed on the die were, therefore, probably more severe.

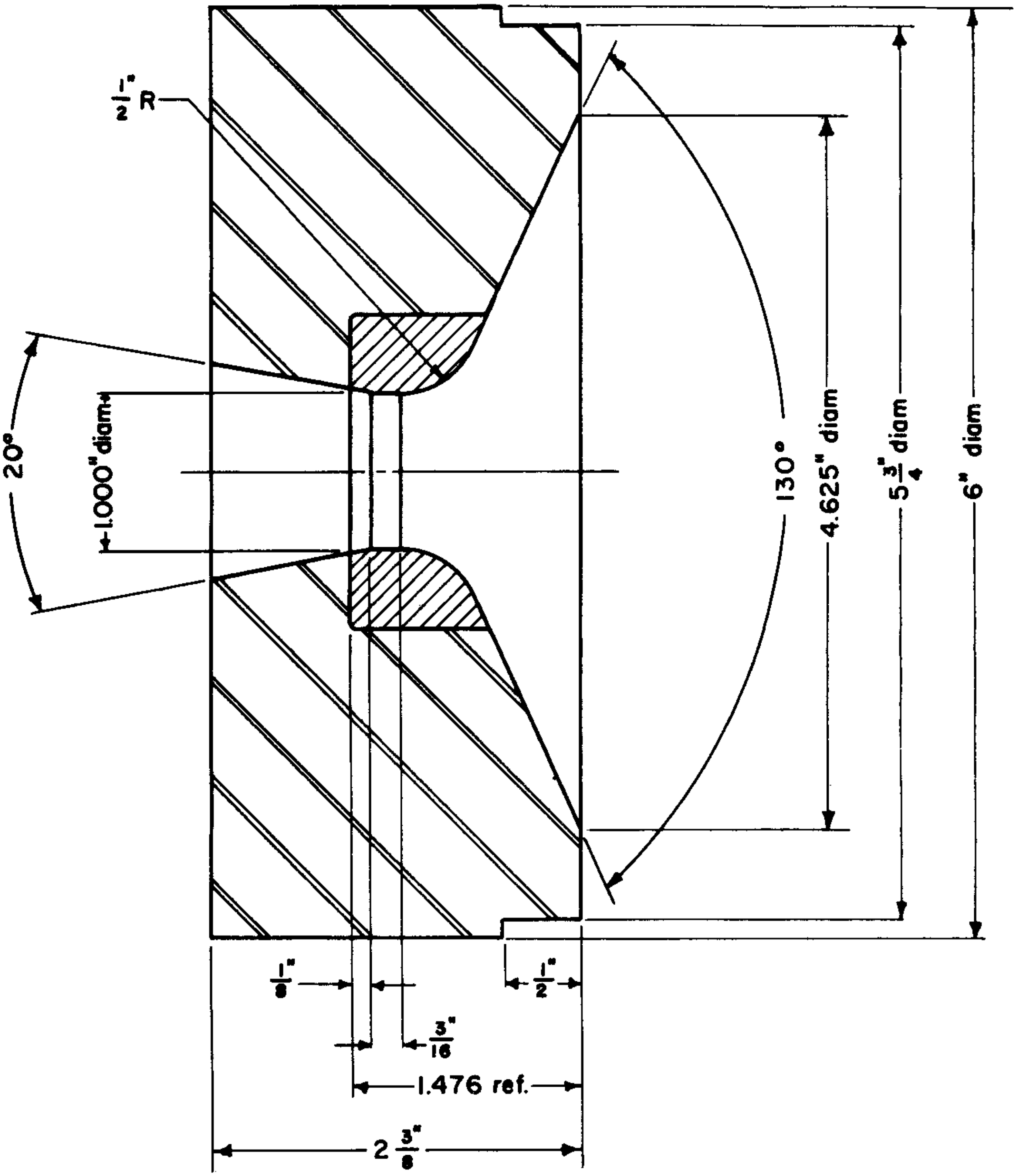


FIGURE 17. DESIGN OF DIES FOR EVALUATING THE EXPERIMENTAL LUBRICANTS AND DIE MATERIALS
Shaded area indicates the die insert.

Experimental Lubricants

A large number of metal-working lubricants for high-temperature applications are produced commercially, but these materials have been developed primarily for deep drawing, wire drawing, forging, and rolling. A limited number have been modified and used for extrusion, but few, if any, have been developed expressly for extruding titanium. Consequently, the Fiske Brothers Refining Company, Toledo, Ohio, was consulted regarding the preparation of experimental lubricant mixtures for evaluation in this work.

Excluding glass, the graphitic lubricants appeared to be the most suitable for hot metal working. Molybdenum disulfide, if protected from excessive oxidation, also has found wide application. Generally, these materials are suspended in an oil or soap-base grease to facilitate their application to the metal-working dies and tools. Another carrier material that has shown good high-temperature properties is Bentonite grease, a soapless lubricant marketed under the trade name "Bentone". Where temperatures in excess of about 600 F are involved, the carrier material burns off, leaving a powdery lubricant film on the tools.

For the purpose of evaluating the relative performance of the solid lubricants and the carrier materials, four experimental lubricant mixtures were compounded. These materials, designated as Fiske-BMI Experimental Lubricants, had the following compositions:

1 - Calcium-base grease containing 35 per cent flake graphite and 5 per cent mica

2 - Calcium-base grease containing 25 per cent molybdenum disulfide and 5 per cent mica

3 - Calcium-base grease containing 25 per cent flake graphite, 15 per cent molybdenum disulfide, and 5 per cent mica

4 - Bentone, containing 25 per cent flake graphite, 15 per cent molybdenum disulfide, and 5 per cent mica.

The calcium-base grease was selected as a base for three of the lubricants because its oxidation products are less abrasive than the other high-temperature soaps. Mica was added to all of the lubricants to improve their insulating properties.

The Fiske No. 514-A lubricant that was used in the earlier tests was a low-viscosity mixture (NLGI No. 0 consistency). Consequently, upon application of the lubricant to the hot tools, it collected on the bottom of the container and on the bottom part of the die. The inability of the lubricant to "stay in place" was reflected by the occurrence of pickup on the die face, which was consistently heavier at the top part of the die. In order to reduce the fluidity of the lubricants at the high temperatures involved, the experimental lubricant mixtures were compounded to a NLGI No. 2 consistency, which corresponds to a high-viscosity grease.

An additional lubricant, marketed under the trade name "Necrolene" by Crawford Emulsions, Inc., Pittsburgh, Pennsylvania, was also tested. This lubricant is prepared in the form of a block grease that is a residual petroleum base containing high-melting-point waxes, lime, and various hardening materials. Upon contact with hot tool surfaces, the Necrolene melts, leaving a solid asphaltic residue.

The Ti-3Mn-complex billets used in the lubricant-evaluation tests were extruded at 1600 F. The lubricants were applied to both the container wall and the die face. Conditions under which the tests were conducted and data obtained are presented in Table 6.

In Test T-18, using Experimental Lubricant No. 1, an accurate evaluation of the lubricant could not be obtained because of the presence of tramp aluminum on the container wall. The die used in this test was resurfaced and another test, T-22, was conducted to evaluate the lubricant. It is interesting to note, however, that the lowest break-through pressure was required in Test T-18.

Effect of Lubricant on Die Pickup and Bar Surface

There was no evidence of die wear in any of the tests. In several instances, however, there was a considerable amount of pickup on the lead-in radius and on the land of the die. The pickup was concentrated mainly on the top part of the die, corresponding to the top part of the container. There appeared to be two factors, other than the actual effectiveness of the lubricants, that influenced the amount and location of the pickup that occurred on the die surfaces. These were as follows:

(1) The distribution of lubricant on the die and the container. Lubricants that are suspended in a grease or oil flash on contact with the hot tool surfaces and become more fluid. If too heavy a coating is applied, the lubricant will flow, collecting at the bottom of the container and on the bottom part of the die. As a result, the amount of lubricant available at the top part of the tools may be insufficient to prevent die pickup.

(2) The clearance between the dummy block and the container. If too great a clearance exists between the dummy block and the container wall, the billet will be scalped, leaving a thin skull of metal on the upper part of the container wall. As a result, the lubricant on this part of the container is trapped, preventing adequate lubrication at the top part of the die and causing the metal flowing into the die to seize on the die surface.

TABLE 6. DATA ON EXTRUSION OF THE Ti-3Mn-COMPLEX ALLOY AT 1600 F USING VARIOUS EXPERIMENTAL LUBRICANTS

Test	Die[(a)]	Billet	Lubricant	Length of Extrusion, in.	Extrusion Time, sec	Average Extrusion Rate, in./sec[(b)]	Extrusion Pressure, 1000 psi		
							Initial	Run-Out	Final
T-18[(c)]	LT-1	67	Fiske-BMI No. 1	72	39	1.9	89.5	89.5	100.5
T-19	LT-2	68	Fiske-BMI No. 2	92	32	2.9	100.5	98.5	100.5
T-20	LT-3	69	Fiske-BMI No. 3	77	34	2.3	100.5	100.5	100.5
T-21	LT-4	70	Fiske-BMI No. 4	97	23	4.2	93.0	86.7	98.5
T-22	LT-1	71	Fiske-BMI No. 1	89	33	2.7	102.0	98.5	102.0
T-23	LT-2	72	Necrolene	91	33	2.8	98.5	86.7	100.5

(a) Die material: Firth-Sterling L. T. Forging Die Steel (0.33C, 9W, 3.5Cr, 0.5V), 50 R_C.

(b) Average speed of extruded bar leaving die.

(c) Tramp aluminum present on container wall prevented accurate evaluation of lubricant in this test. Test repeated as Test T-22.

Increasing the consistency of the lubricants to a NLGI No. 2 grade decreased "running" on the hot tool surfaces and made it possible to obtain a more uniform coating of lubricant on the tools. The dummy block-to-container clearance was too great, however, and this condition is believed to be the principal cause of the pickup that occurred at the top part of the die openings during these tests.

The effectiveness of the lubricants was evaluated by the amount of pickup that occurred on the lower part of the die, which was not affected by the factors heretofore described. The least amount of pickup occurred with Fiske-BMI Experimental Lubricant No. 4 (Bentone containing graphite, molybdenum disulfide, and mica). In Test T-21, in which this lubricant was used, the bottom part of the die retained its original surface condition. The dies used with the other lubricants had varying amounts of pickup on the bottom part of the die opening.

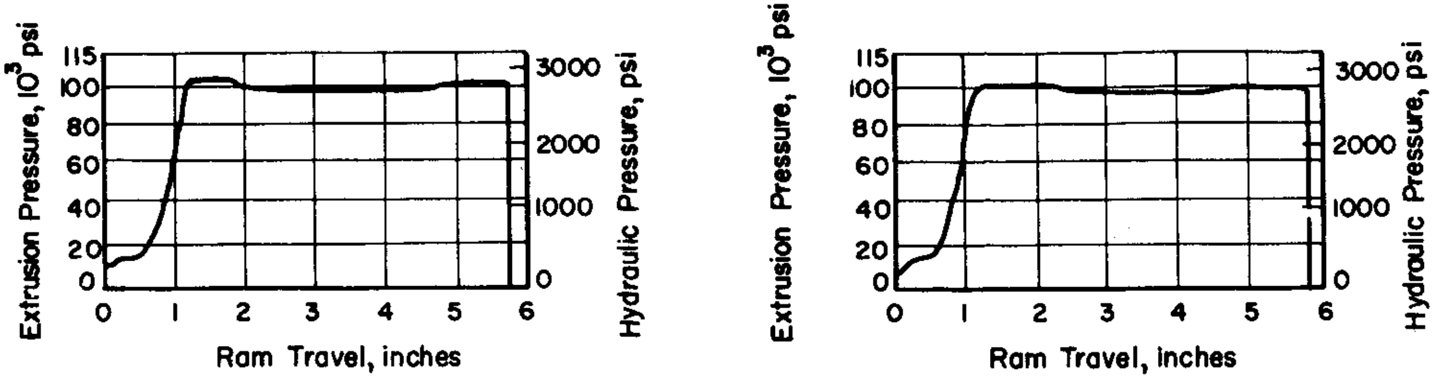
The effect of the amount of titanium pickup on the die was reflected clearly by the surface condition of the extruded bars. The best over-all surface was produced with Experimental Lubricant No. 4. The bottom part of the bar (position relative to die and container) had a smooth finish over its entire length; the top part was slightly scored, but there were no tears or rough zones. The bars extruded with the other lubricants were scored more severely because of the greater amounts of die pickup that occurred.

Effect of Lubricant on Extrusion Pressure

The effectiveness of the lubricants in reducing the friction between the billet and the container wall could not be determined exactly because the top parts of the billets were scalped. The location and size of the skulls, however, were about the same for all of the tests. Therefore, the extrusion pressures recorded are a reasonable indication of the performance of the lubricants. The curves of extrusion pressure versus ram travel for the four experimental lubricants are shown in Figure 18.

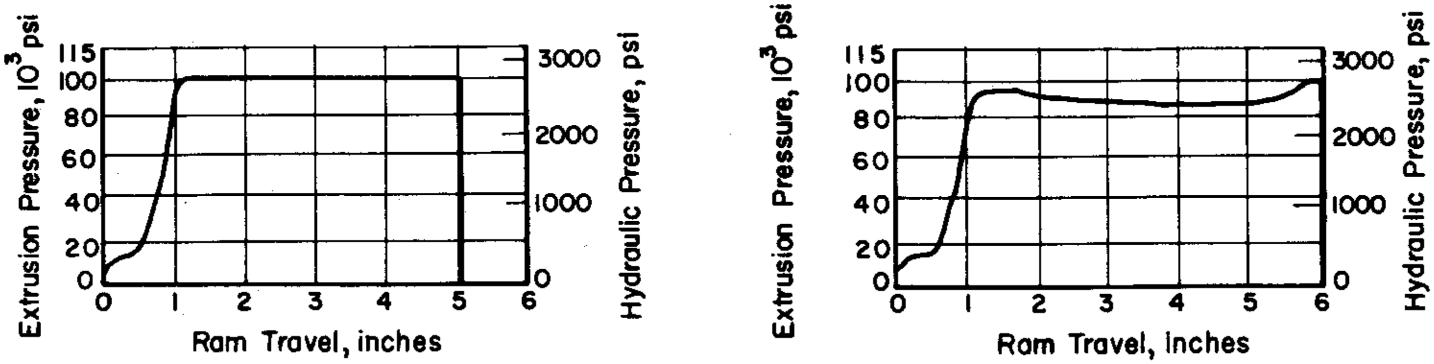
Break-through and run-out pressures with Experimental Lubricant No. 4 were about 10,000 psi lower than with Experimental Lubricants Nos. 1, 2, and 3. Also, the average rate of extrusion with Experimental Lubricant No. 4 was considerably greater than with the other three lubricants, as indicated in Table 6. The lower pressure and higher extrusion speed indicated that a considerable reduction in the friction between the container and billet was attained with this lubricant.

Extrusion pressure with the Necrolene lubricant was about the same as with the Experimental Lubricant No. 4. Although the surface finish was not so good as that obtained with the Bentone lubricant, the Necrolene offers a distinct advantage in that its application to the tools can be controlled more easily. Lubricants of the grease type must be applied by



Test T-22; Experimental Lubricant No. 1: Calcium Grease, 35% Graphite, 5% Mica

Test T-19; Experimental Lubricant No. 2: Calcium Grease, 25% MoS_2, 5% Mica



Test T-20; Experimental Lubricant No 3: Calcium Grease, 25% Graphite, 15% MoS_2, 5% Mica

Test T-21; Experimental Lubricant No 4: Bentone, 25% Graphite, 15% MoS_2, 5% Mica

FIGURE 18. CURVES OF PRESSURE VERSUS RAM TRAVEL FOR THE EXTRUSION OF THE Ti-3Mn-COMPLEX ALLOY WITH THE FISKE-BMI EXPERIMENTAL LUBRICANTS
Note lower pressure required with Experimental Lubricant No. 4.

swabbing the tool surfaces, whereas Necrolene is melted onto the surface by direct contact.

Experimental Die Materials

The destructive effect of high extrusion temperatures on dies made of the common hot-work steels was shown clearly in Tests T-9 through T-13, described earlier in this report, wherein the Ti-3Mn-complex alloy was extruded at temperatures in the range 1350 to 1700 F. Above 1350 F, the dies made of Carpenter 883 steel were severely eroded after one extrusion. In several instances, the dies had been tempered, during extrusion, to less than 20 R_C hardness near the die opening, indicating that the die-face temperature was considerably higher than 1300 F during extrusion. Thus, it is evident that even the high-speed tool steels would fail under continuous use, unless sufficient insulation of the die were provided by the lubricant. Consequently, it was necessary to search the field of high-temperature, abrasion-resistant materials, other than steels, for suitable die materials. Foremost among these materials are the cobalt-base alloys and the cemented carbides.

After consultation with various manufacturers of die materials, nine materials were selected for evaluation as extrusion dies for titanium. They were supplied by the producers for evaluation in the form of inserts for dies of the design shown in Figure 17. The experimental materials, their trade designations, and their sources are as follows:

Type of Material	Trade Designation	Manufacturer
Titanium carbide	Kentanium Grade K153B	Kennametal, Incorporated
Titanium carbide	Kentanium Grade K175B	Kennametal, Incorporated
Titanium carbide	Carboloy Grade X-3532	Carboloy
Chromium carbide	Carboloy Grade 608	Carboloy
Cobalt-base alloy	Haynes Stellite Alloy No. 3	Haynes Stellite Company
Cobalt-base alloy	Haynes Stellite Alloy No. 6	Haynes Stellite Company
Cobalt-base alloy	Haynes Stellite Star J-Metal	Haynes Stellite Company
Tungsten-base alloy	Firth Heavy Metal	Firth Sterling, Inc.
Tungsten tool steel	XDL Steel	Firth Sterling, Inc.

The Kentanium inserts were 2 inches in diameter; the other materials were supplied as 2-1/4-inch-diameter inserts.

It should be understood that the performance of these materials was evaluated for a specific application, other than that for which they were developed originally. Therefore, any failure or poor performance of a material is not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

Description of Tests

The Ti-3Mn-complex billets were extruded at 1700 F to evaluate the die materials. Two series of tests were conducted in this investigation. In the first, the chromium and titanium carbides and the XDL steel were evaluated. The cobalt-base alloys and the Firth Heavy Metal were evaluated in the second series of tests.

Four lubricants were used in these extrusion tests. The designations and compositions of these materials are as follows:

Fiske-BMI Experimental Lubricant No. 4 – Bentone containing 25 per cent flake graphite, 15 per cent molybdenum disulfide, and 5 per cent mica. Two consistencies were used: NLGI No. 4 and NLGI No. 2.

Fiske No. 525 Hot Die Lubricant – Bentone containing 25 per cent flake graphite; NLGI No. 0 consistency.

Fiske-BMI Experimental Lubricant No. 5 – Bentone containing 35 per cent flake graphite and 5 per cent mica; NLGI No. 2 consistency.

Fiske No. 514-A-10AP Lubricant – Aluminum-base grease containing 33 per cent flake graphite and 5 per cent powdered aluminum; NLGI No. 000 consistency.

The majority of the tests were conducted with Fiske-BMI Experimental Lubricant No. 4, which gave the best results in the earlier lubricant studies. The tests with the other three lubricants were of an exploratory nature. A description of the extrusion tests on the experimental die materials, showing the order in which they were conducted, is presented in Table 7.

Prior to the tests, the inside diameter of the container liner was measured, and it was found that severe wear had occurred in the end where the billet is upset and extruded. The wear had caused the liner to become flared at this end, the inside diameter varying from 4.625 inches (the original ID) to 4.680 inches. In order to reduce the clearance between the dummy block and the container wall as much as possible, the diameter of the dummy blocks used in these tests was increased from 4.610 to 4.620 inches. The clearance was still too great, however, and the billets were scalped during extrusion, leaving a thin skull of titanium on the upper part of the container. However, the skulls formed were smaller than those found in earlier tests and appeared to have only a minor effect on die pickup or surface finish. In extruding longer billets, however, this condition probably would result in heavier die pickup and a poorer surface finish, since the lubricant is trapped on the wall of the container. Therefore, in commercial practice, the clearance between the dummy block and the container should be kept as small as possible.

As indicated in Table 7, the dies in each series of tests were used in rotation. After each test, the surface of the die insert was polished to remove any traces of titanium pickup. Measurements were then made to determine the amount of wear.

Test Data and Observations

The results of the extrusion tests on each of the experimental die materials are described in the following paragraphs. For evaluation purposes, the amount of pickup that occurred was rated as follows:

Rating	Symbol	Description
Very light	VL	Traces of titanium on radius of insert; none on die land
Light	L	Several zones on radius and face of insert having a thin build-up of titanium; but none on die land
Medium	M	Thin build-up of titanium around radius and face of insert and on die land
Heavy	H	Heavy build-up of titanium on radius and face of insert and on die land

In general, the surface finish of the extruded bars was very good, being free of tears and seams. Some variation in the finish occurred with the different lubricants and die materials, however, and an arbitrary rating was assigned to the surface finish, as given on page 46.

TABLE 7. DESCRIPTION OF THE EXTRUSION TESTS CONDUCTED

Test	Billet(b)	Die Material(c)	Lubricant(d)
		Test Series 1	
T-24	73	XDL	Fiske-BMI No. 4-(4)
T-25	74	K175B	Ditto
T-26	75	X-3532	"
T-27	76	608	"
T-28	77	K153B	"
T-29	78	XDL	"
T-30	79	K175B	"
T-31	80	X-3532	"
T-32	81	608	"
T-33	82	K153B	"
T-34	83	XDL	"
T-35	84	K175B	"
T-36	85	X-3532	"
T-37	86	608	Fiske-BMI No. 4-(2)
T-38	87	K153B	Ditto
T-39	88	XDL	"
T-40	89	K175B	"
T-41	90	X-3532	"
T-42	91	608	"
T-43	92	K153B	"
T-44	93	XDL	"
T-45	94	K175B	Fiske No. 525-(0)
T-46	95	X-3532	Ditto
T-47	96	608	"
T-48	97	K153B	"

(a) Extrusion temperature 1700 F; extrusion ratio 21:1

(b) Billet size: 4-7/16 inches in diameter by 6 inches long.

(c) Designation of die materials:

XDL - Firth Sterling XDL steel
K175B - Kentanium Grade K175B titanium carbide
X-3532 - Carboloy Grade X-3532 titanium carbide
608 - Carboloy Grade 608 chromium carbide
K153B - Kentanium Grade K153B titanium carbide
H.S. 3 - Haynes Stellite Alloy No. 3
H.S. 6 - Haynes Stellite Alloy No. 6
H.S. J - Haynes Stellite Star J-Metal
WEX 85-10-5 - Firth Heavy Metal

(d) The number in parentheses following the lubricant number is the NLGI grade of consistency.

TO EVALUATE THE EXPERIMENTAL DIE MATERIALS[a]

Test	Billet[b]	Material[c]	Lubricant[d]
		Test Series 2	
T-49	98	H. S. 3	Fiske-BMI No. 4-(2)
T-50	99	H. S. 6	Ditto
T-51	100	H. S. J	"
T-52	101	WEX 85-10-5	"
T-53	102	H. S. 3	"
T-54	103	H. S. 6	"
T-55	104	H. S. J	"
T-56	105	WEX 85-10-5	"
T-57	106	H. S. 3	"
T-58	107	H. S. 6	"
T-59	108	H. S. J	"
T-60	109	WEX 85-10-5	"
T-61	110	H. S. 3	Fiske-BMI No. 5-(2)
T-62	111	H. S. 6	Ditto
T-63	112	H. S. J	"
T-64	113	608	Fiske-BMI No. 4-(2)
T-65	114	X-3532	Fiske No. 514-A-10AP-(000)
T-66	115	608	Ditto

Rating	Symbol	Description
Fair	F	Light scoring over the surfaces, but no tears
Good	G	Light surface scratches, but no scoring or tears
Very good	VG	Smooth surface with few scratches
Excellent	E	Smooth surface free of markings

XDL Steel. Firth Sterling XDL Steel has the following composition: 0.38C, 14.0W, 3.40Cr, 0.50V. The low carbon content in this type of steel is intended to increase its thermal shock resistance without severely affecting the high-temperature abrasion resistance. The XDL insert was heat treated to a hardness of 50 R_C. A 0.012-inch shrink fit was used between the die case and the insert.

Five billets were extruded through this die, using the Fiske-BMI Experimental Lubricant No. 4. Data and observations on these tests are presented in Table 8. The XDL insert withstood the high extrusion temperature, exhibiting no evidence of heat checking. There was a slight amount of die wear, however, the inside diameter of the insert increasing from 0.997 to 1.000 inch. Die pickup became increasingly heavy as the number of tests increased, indicating that possibly the surface of the steel insert had softened. The titanium pickup was very adherent to the steel and had to be removed by grinding. A photograph of the polished XDL die, taken after the last extrusion test, is shown in Figure 19.

Kentanium Grades K153B and K175B Titanium Carbide. The Kentanium compositions used in this work contained nickel as the binder metal. Grade K153B is a commercial grade having a hardness of 82.8 R_A (63 R_C). Grade K175B is an experimental composition having a hardness of 84.1 R_A (65 R_C). Examination of the inserts by Kennametal indicated that the K175B insert had some microporosity after sintering. There was insufficient time to prepare another insert, however, and the material was tested in this condition.

Five billets were extruded through each die, using Fiske-BMI Lubricant No. 4 and Fiske No. 525 Hot Die Lubricant. Data and observations on these tests are presented in Table 9. Cracks developed in the K153B insert after the third test and in the K175B insert after the fourth test. The cracks appeared to be mainly surface checks resulting from thermal shock. Several of the larger cracks, however, were actual fractures. The 0.006-inch shrink fit on the dies was insufficient to maintain compression on the insert at the operating temperature of the

TABLE 8. DATA AND OBSERVATIONS ON THE EXTRUSION TESTS WITH THE XDL STEEL DIE INSERT[a]

Test	Lubricant	Die Temperature, F		Extrusion Time, sec	Length Extruded, in.	Average Extrusion Rate, in/sec[b]	Extrusion Pressure, 1000 psi			Die Pickup[c]	Surface Finish of Bar[c]
		Before	After				Initial	Run-Out	Final		
T-24[d]	Fiske-BMI No. 4[e]	700	850	62.0	88	1.4	104	104	104	L	F
T-29	Ditto	600	750	28.0	115	4.1	93	87	102	L	G
T-34	"	650	750	14.0	84	6.0	82	73	79	L	F
T-39	Fiske-BMI No. 4[f]	600	750	16.5	92	5.6	86	77	93	M	F
T-44	Ditto	500	850	17.0	99	5.8	98	88	104	H	G

(a) Extrusion temperature 1700 F; extrusion ratio 21:1.
(b) Average speed of extruded bar leaving die.
(c) See pages 43 and 46 for an explanation of these symbols.
(d) Graphite back-up used. Delay in extrusion caused severe cooling of the billet.
(e) NLGI No. 4 consistency.
(f) NLGI No. 2 consistency.

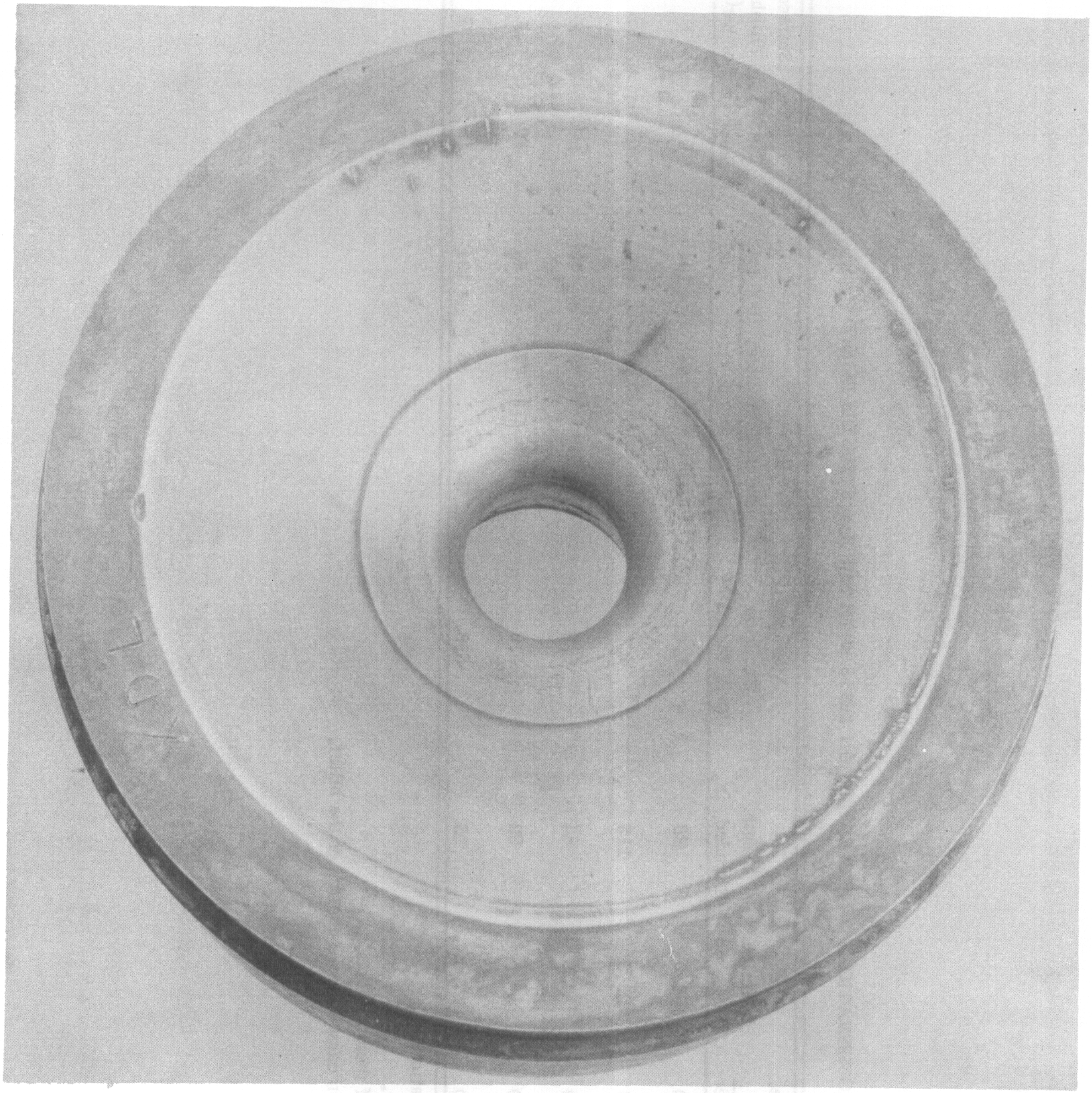


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FIGURE 19. APPEARANCE OF POLISHED DIE WITH XDL STEEL INSERT AFTER EXTRUSION OF FIVE Ti-3Mn-COMPLEX BILLETS AT 1700 F

Markings on face of insert occurred during grinding to remove titanium pickup. Note that no cracks occurred in the insert. Diameter of the die land increased from 0.997 to 1.000 inch during the tests.

TABLE 9. DATA AND OBSERVATIONS ON THE EXTRUSION TESTS WITH THE TITANIUM CARBIDE DIE INSERTS(a)

Test	Lubricant	Die Temperature, F		Extrusion Time, sec	Length Extruded, in.	Average Extrusion Rate, in/sec(b)	Extrusion Pressure, 1000 psi			Die Pickup(c)	Surface Finish of Bar(c)
		Before	After				Initial	Run-Out	Final		
					Kentanium Grade K153B						
T-28	Fiske-BMI No. 4(d)	600	750	28.0	116	4.2	96	86	102	VL	VG
T-33	Ditto	600	750	20.0	116	5.8	83	74	100	L	F
T-38	Fiske-BMI No. 4(e)	500	650	14.5	88	6.1	80	74	95	VL	E
T-43	Ditto	570	900	16.0	91	5.7	97	83	95	L	VG
T-48	Fiske No. 525	500	850	14.7	98	6.7	87	76	95	L	VG
					Kentanium Grade K175B						
T-25	Fiske-BMI No. 4(d)	700	800	36.0	96	2.7	102	102	102	L	F
T-30	Ditto	700	750	20.0	103	5.2	82	75	93	M	G
T-35	"	800	900	19.0	101	5.3	84	76	86	H	G
T-40	Fiske-BMI No. 4(e)	550	700	16.0	92	5.8	79	72	91	L	VG
T-45	Fiske No. 525	500	850	14.2	98	6.9	88	78	93	M	VG
					Carboloy Grade X-3532						
T-26	Fiske-BMI No. 4(d)	800	850	37.0	111	3.0	98	93	102	H	F
T-31	Ditto	650	800	19.0	102	5.4	73	68	88	L	G
T-36	"	750	850	14.0	94	6.7	75	66	77	VL	G
T-41	Fiske-BMI No. 4(e)	550	850	15.7	94	6.0	88	80	96	VL	VG
T-46	Fiske No. 525	650	950	14.3	99	6.9	88	79	92	VL	G
T-65	Fiske No. 514A-10AP	600	900	26.0	89	3.4	95	95	102	M	VG

(a) Extrusion temperature 1700 F; extrusion ratio 21:1.
(b) Average speed of extruded bar leaving die.
(c) See Pages 43 and 46 for an explanation of these symbols.
(d) NLGI No. 4 consistency.
(e) NLGI No. 2 consistency.

die. During preheating, the K153B insert became loose and had to be forced back into the case. A greater shrink fit on the insert might have prevented the occurrence of fractures. The cracks did not appear to have any effect on the surface finish of the bars extruded in these tests.

The K153B insert showed no visible signs of wear and maintained its original dimensions throughout the tests. The K175B insert, on the other hand, did exhibit slight wear, probably because of the porosity on the surface of the insert. After the first test, the inside diameter of the insert increased from 0.996 to 0.999 inch, but no further wear occurred after continued extrusion.

Both materials exhibited good resistance to galling by the titanium. Some pickup occurred on the dies, mainly on the lead-in radius, but it was easily removed without damage to the surface of the insert. Pickup was heavier on the K175B insert; again, this may have been due to the porosity. Photographs of the polished K153B and K175B dies, taken after the tests, are shown in Figures 20 and 21. The dies were swabbed lightly with dilute nitric acid to clean their surfaces.

Carboloy Grade X-3532 Titanium Carbide. Carboloy Grade X-3532 is an experimental titanium carbide composition, using nickel as the binder metal. Six billets were extruded through this die, using Fiske-BMI Experimental No. 4, Fiske No. 525, and Fiske No. 514A-10AP lubricants. Data and observations on these tests are also presented in Table 9.

Slight surface cracks occurred during the fourth extrusion, apparently from thermal shock. The dimensions of the insert were not affected, however, and there was no visible evidence of die wear. Varying amounts of titanium pickup occurred during the tests, but it was not very adherent and could be removed easily without damage to the original surface of the insert. A photograph of the unpolished X-3532 die, taken after the sixth extrusion, is shown in Figure 22. The die face was swabbed very lightly with dilute nitric acid to show the titanium pickup more clearly.

Carboloy Grade 608 Chromium Carbide. Carboloy Grade 608 has the following composition: $83Cr_3C_2$, 2WC, 15Ni. It has a nominal hardness of 88 R_A (70 R_C). The oxidation resistance of Carboloy Grade 608 is reported to be excellent at temperatures up to 1800 F.

Seven billets were extruded through this die, using Fiske-BMI Experimental No. 4, Fiske No. 525, and Fiske No. 514A-10AP lubricants. Data and observations on these tests are presented in Table 10. Slight surface cracks developed in the die land during the fourth test, but dimensional tolerance was maintained to within less than 0.001 inch after the seventh test.

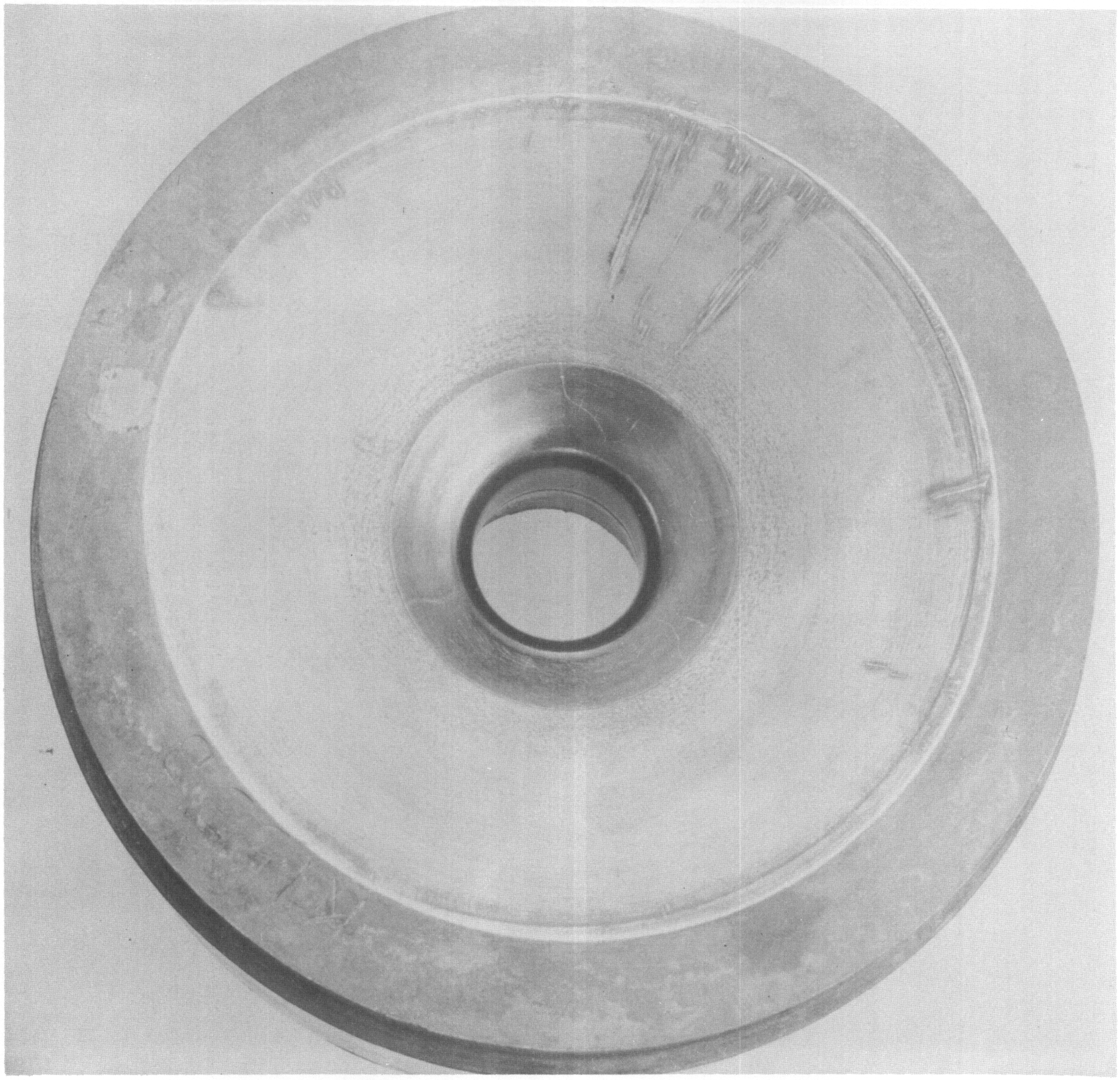

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FIGURE 20. APPEARANCE OF POLISHED DIE WITH KENTANIUM GRADE K153B TITANIUM CARBIDE INSERT AFTER EXTRUSION OF FIVE Ti-3Mn-COMPLEX BILLETS AT 1700 F

Cracks are believed to have resulted partially from thermal shock, but mainly from insufficient shrink fit on insert. Surface finish of insert retained; no change in dimensions.

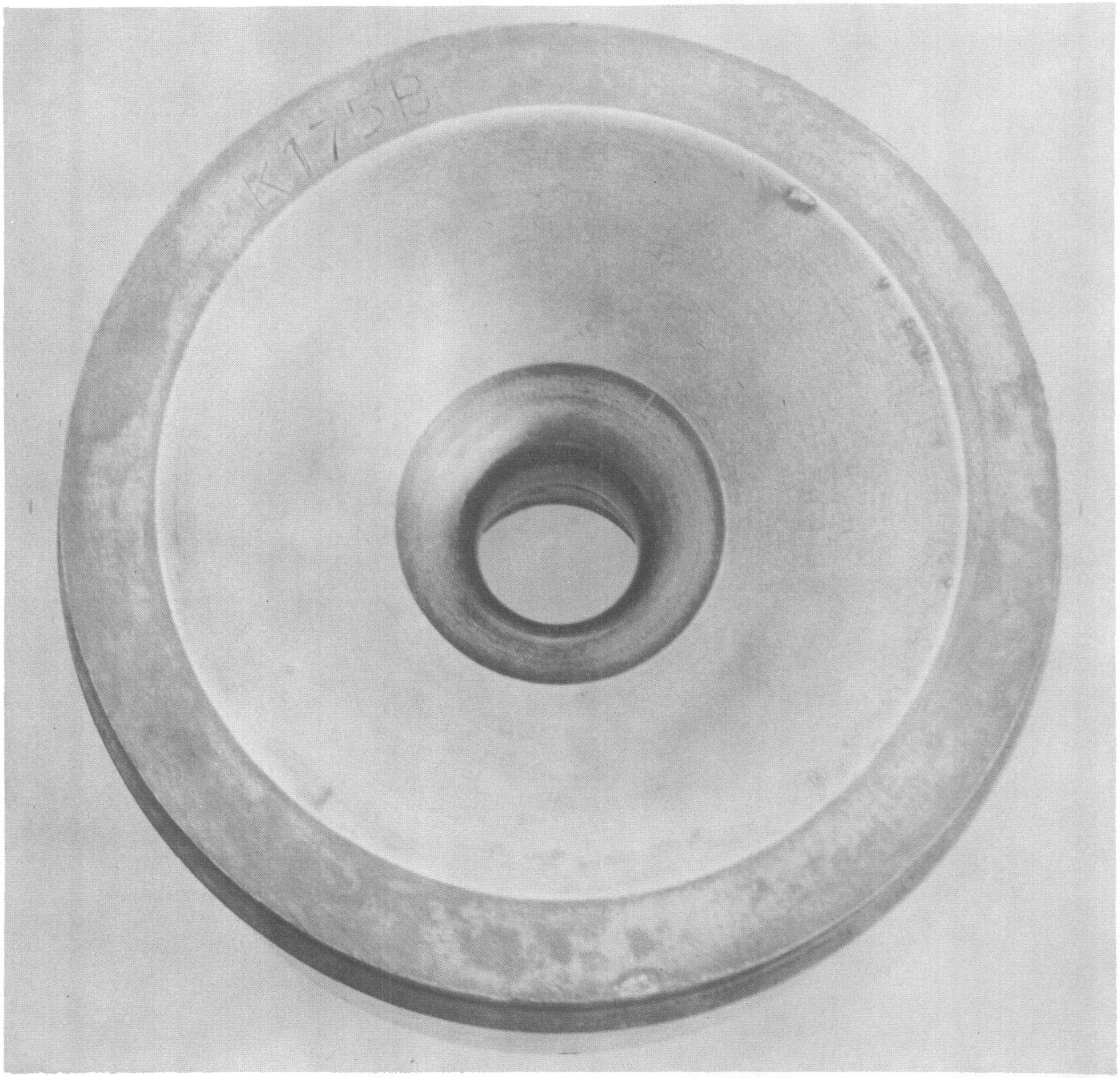

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FIGURE 21. APPEARANCE OF POLISHED DIE WITH KENTANIUM GRADE K175B TITANIUM CARBIDE INSERT AFTER EXTRUSION OF FIVE Ti-3Mn-COMPLEX BILLETS AT 1700 F

Cracks are believed to have resulted partially from thermal shock, but mainly from insufficient shrink fit on insert.

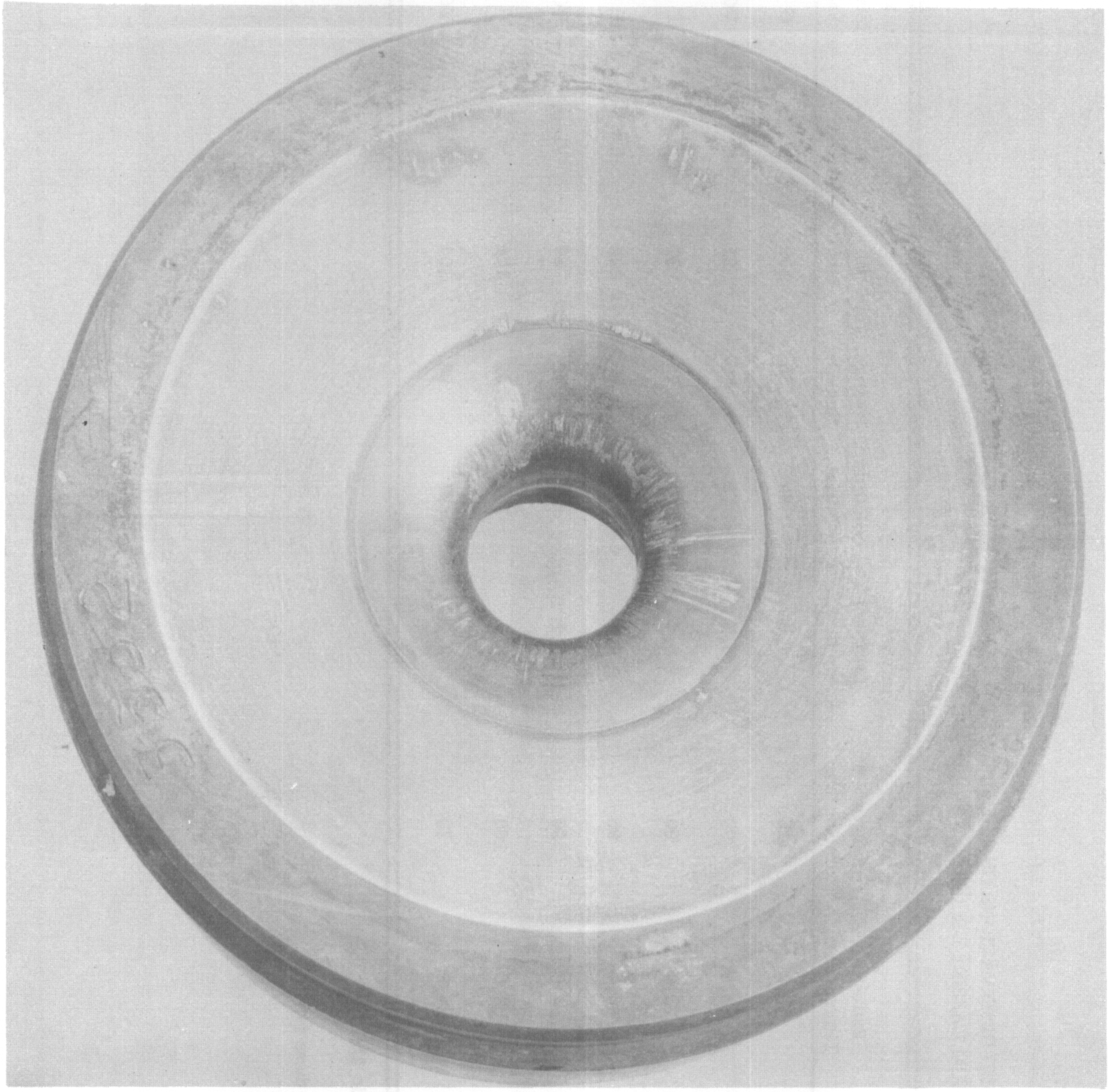

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FIGURE 22. APPEARANCE OF UNPOLISHED DIE WITH CARBOLOY GRADE X-3532 TITANIUM CARBIDE INSERT AFTER EXTRUSION OF SIX Ti-3Mn-COMPLEX BILLETS AT 1700 F

Titanium buildup on insert from last test, using Fiske No. 514A-10AP lubricant, is shown to illustrate "medium" die-pickup rating. Original insert surface retained, with no change in dimensions. Note light surface cracks on die land.

TABLE 10. DATA AND OBSERVATIONS ON THE EXTRUSION TESTS WITH THE CARBOLOY GRADE 608 CHROMIUM CARBIDE DIE INSERT(a)

Test	Lubricant	Die Temperature, F		Extrusion Time, sec	Length Extruded, in.	Average Extrusion Rate, in/sec(b)	Extrusion Pressure, 1000 psi			Die Pickup(c)	Surface Finish of Bar(c)
		Before	After				Initial	Run-Out	Final		
T-27	Fiske-BMI No. 4(e)	600	850	29.0	116	4.0	90	86	102	L	G
T-32	Ditto	500	700	18.0	99	5.5	79	75	91	L	VG
T-37	Fiske-BMI No. 4(f)	700	850	18.0	90	5.0	82	75	91	L	E
T-42	Ditto	600	900	16.4	95	5.8	93	85	102	L	E
T-47	Fiske No. 525	600	850	14.6	96	6.7	87	79	89	VL	VG
T-64(d)	Fiske-BMI No. 4(f)	700	950	33.6	100	3.0	102	102	104	M	G
T-66	Fiske No. 514A-10AP	500	850	24.3	79	3.2	97	93	104	VL	VG

(a) Extrusion temperature 1700 F; extrusion ratio 21:1.
(b) Average speed of extruded bar leaving die.
(c) See pages 43 and 46 for an explanation of these symbols.
(d) Lubricant applied to die face only.
(e) NLGI No. 4 consistency.
(f) NLGI No. 2 consistency.

A slight amount of pickup occurred in all of the tests. In Test T-64, only the die face was lubricated, and pickup was slightly heavier than in the other tests. In all of the tests, however, the pickup was not adherent, and the insert retained its smooth surface finish. A photograph of the unpolished Carboloy Grade 608 die, taken after the last extrusion, is shown in Figure 23. The die face was swabbed lightly with dilute nitric acid.

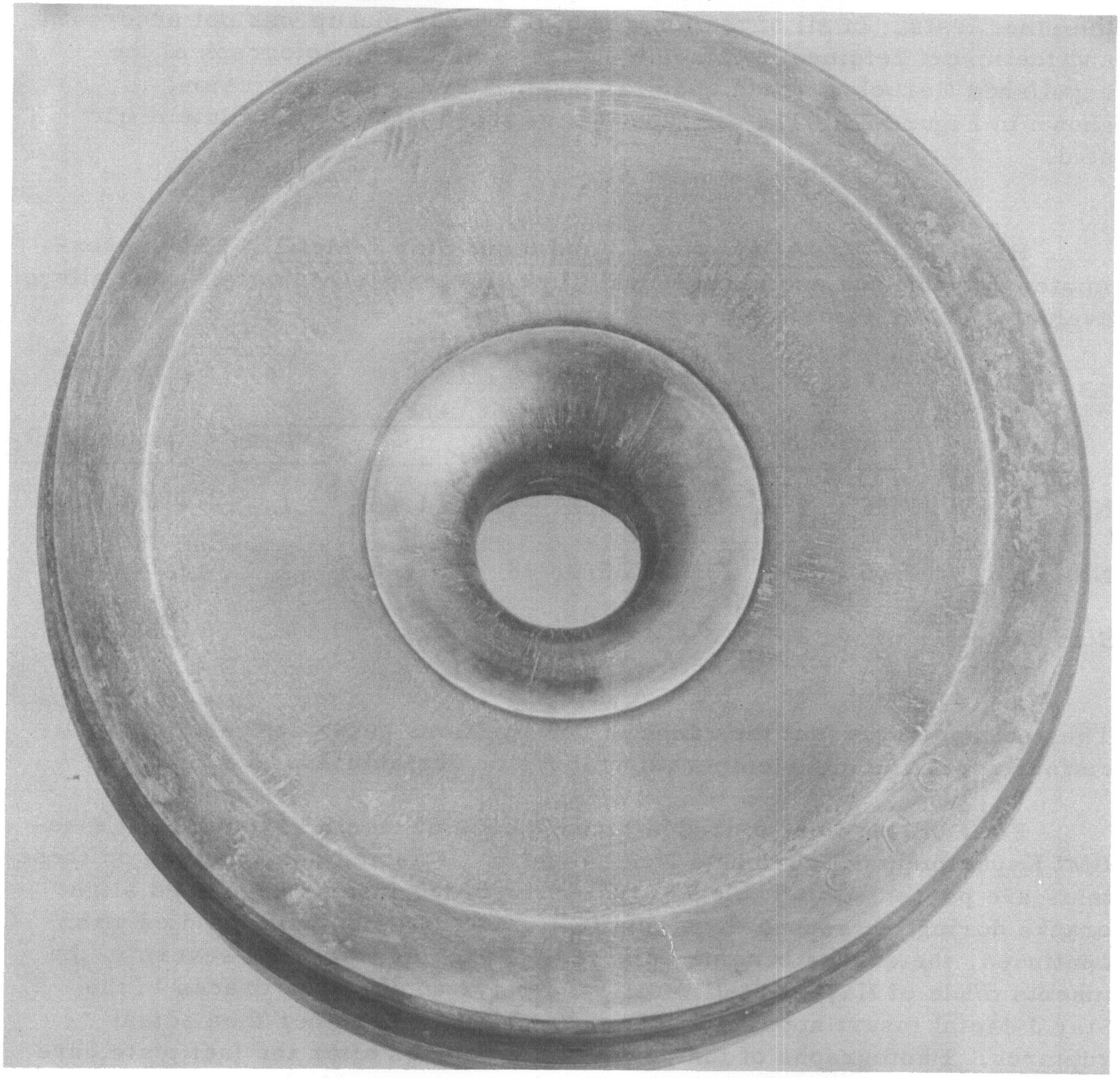
<u>Haynes Stellite Alloys Nos. 3 and 6 and Star J-Metal.</u> The approximate compositions and hardnesses of the Haynes Stellite cobalt-base alloys evaluated as die inserts were as follows:

	Composition, per cent							
Alloy	Co	Ni	Fe	Cr	W	C	Others	Hardness, R_C
No. 6	56	3	2.5	27.5	4.5	1.25	1	44-48
No. 3	46	3	3	30.5	13	2.45	1	53-57
Star J-Metal	41	2.5	3	32	17	2.5	2	56-59

These alloys represent the range of compositions generally used for applications requiring high-temperature abrasion resistance.

Four billets were extruded through each of the dies, using the Fiske-BMI Experimental Lubricants Nos. 4 and 5. Data and observations on these tests are presented in Table 11. All three of the inserts developed slight cracks during the second extrusion test. As extrusion with the dies was continued, the cracks became enlarged. Cracking was most severe in the inserts made of Haynes Stellite Alloys Nos. 3 and 6. The cracks in the Star J-Metal insert appeared to be surface checks, rather than actual ruptures. Photographs of the polished dies, taken after the last tests, are shown in Figures 24, 25, and 26. The dies were swabbed lightly with dilute nitric acid to clean the surfaces.

Die pickup occurred in each test with these inserts, varying from a thin build-up of titanium on the radius to a heavy build-up on the face and land of the inserts. In most instances, the pickup was very adherent and difficult to remove. Slight erosion marks were present after the pickup was removed, and it was necessary to polish the dies after each test. As a result, the inside diameters of the inserts increased, as given on page 61.



N19487

FIGURE 23. APPEARANCE OF UNPOLISHED DIE WITH CARBOLOY GRADE 608 CHROMIUM CARBIDE INSERT AFTER EXTRUSION OF SEVEN Ti-3Mn-COMPLEX BILLETS AT 1700 F

Titanium buildup on insert from last test, using Fiske No. 514A-10AP lubricant, is shown to illustrate "very light" die-pickup rating. Light surface cracks in die land, but original surface of insert retained; no change in dimensions.

TABLE 11. DATA AND OBSERVATIONS ON THE EXTRUSION TESTS WITH THE COBALT-BASE-ALLOY DIE INSERTS[a]

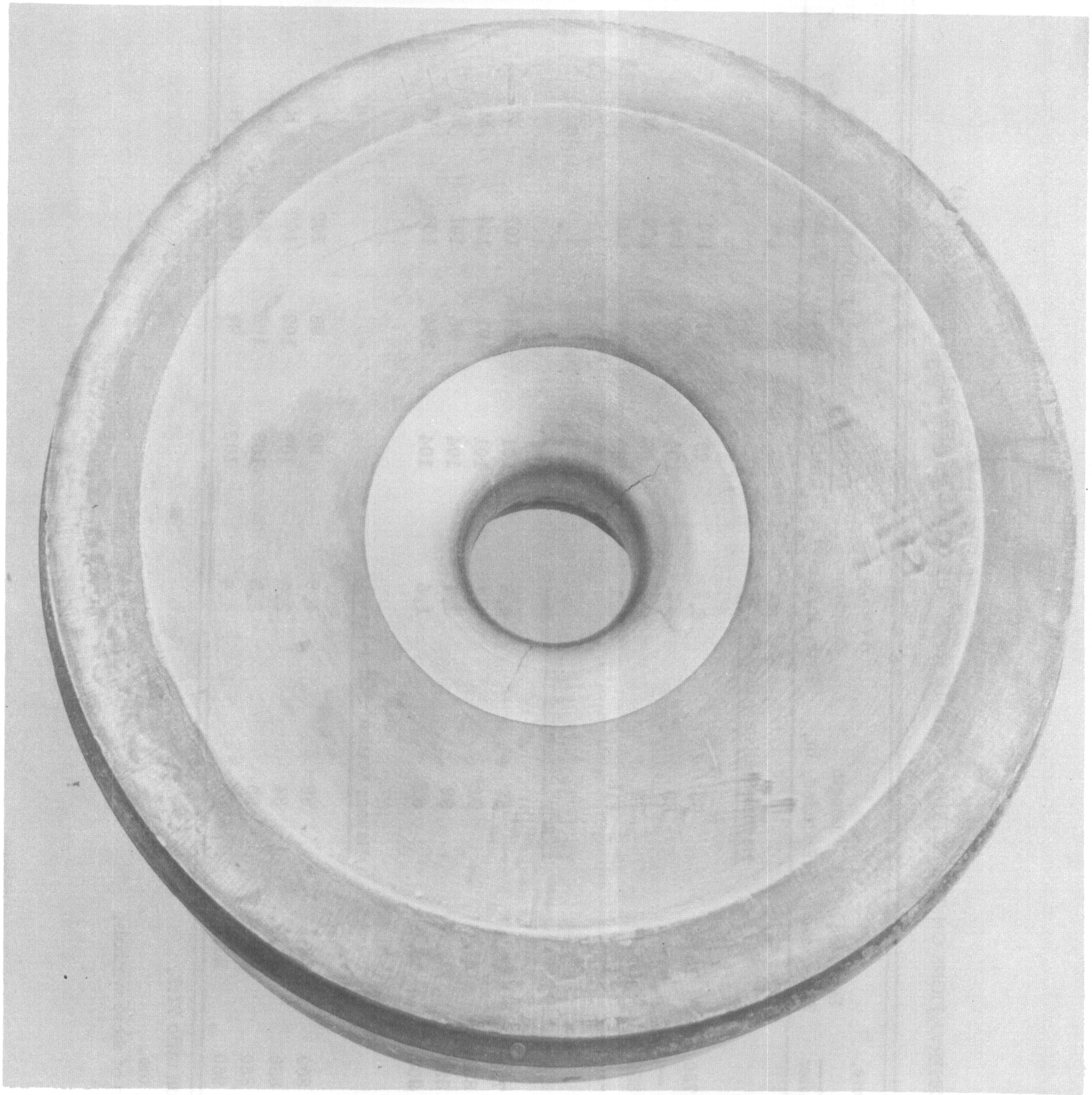
Test	Lubricant	Die Temperature, F		Extrusion Time, sec	Length Extruded, in.	Average Extrusion Rate, in/sec[b]	Extrusion Pressure, 1000 psi			Die Pickup[c]	Surface Finish of Bar[c]
		Before	After				Initial	Run-Out	Final		
					Haynes Stellite Alloy No. 3						
T-49	Fiske-BMI No. 4[d]	650	750	13.8	90	6.5	77	77	101	H	G
T-53	Ditto	650	--	--	89	--	104	93	104	M	VG
T-57	"	550	850	17.5	93	5.3	86	86	102	M	G
T-61	Fiske-BMI No. 5	600	900	26.4	81	3.1	103	103	103	L	VG
					Haynes Stellite Alloy No. 6						
T-50	Fiske-BMI No. 4[d]	550	750	18.4	89	4.8	101	95	102	M	VG
T-54	Ditto	600	850	18.0	91	5.1	101	101	104	M	VG
T-59	"	600	850	26.5	98	3.7	104	95	102	H	VG
T-63	Fiske-BMI No. 5	650	900	40.0	62	1.5	104	104	104	VL	G
					Haynes Stellite Star J-Metal						
T-51	Fiske-BMI No. 4[d]	600	800	18.5	89	4.8	99	98	102	L	F
T-55	Ditto	650	850	36.8	84	2.3	102	102	102	H	F
T-59	"	600	850	27.2	90	3.3	103	103	103	M	VG
T-64	Fiske-BMI No. 5	700	950	23.0	102	4.4	103	94	102	H	G

(a) Extrusion temperature 1700 F; extrusion ratio 21:1.

(b) Average speed of extruded bar leaving die.

(c) See pages 43 and 46 for an explanation of these symbols.

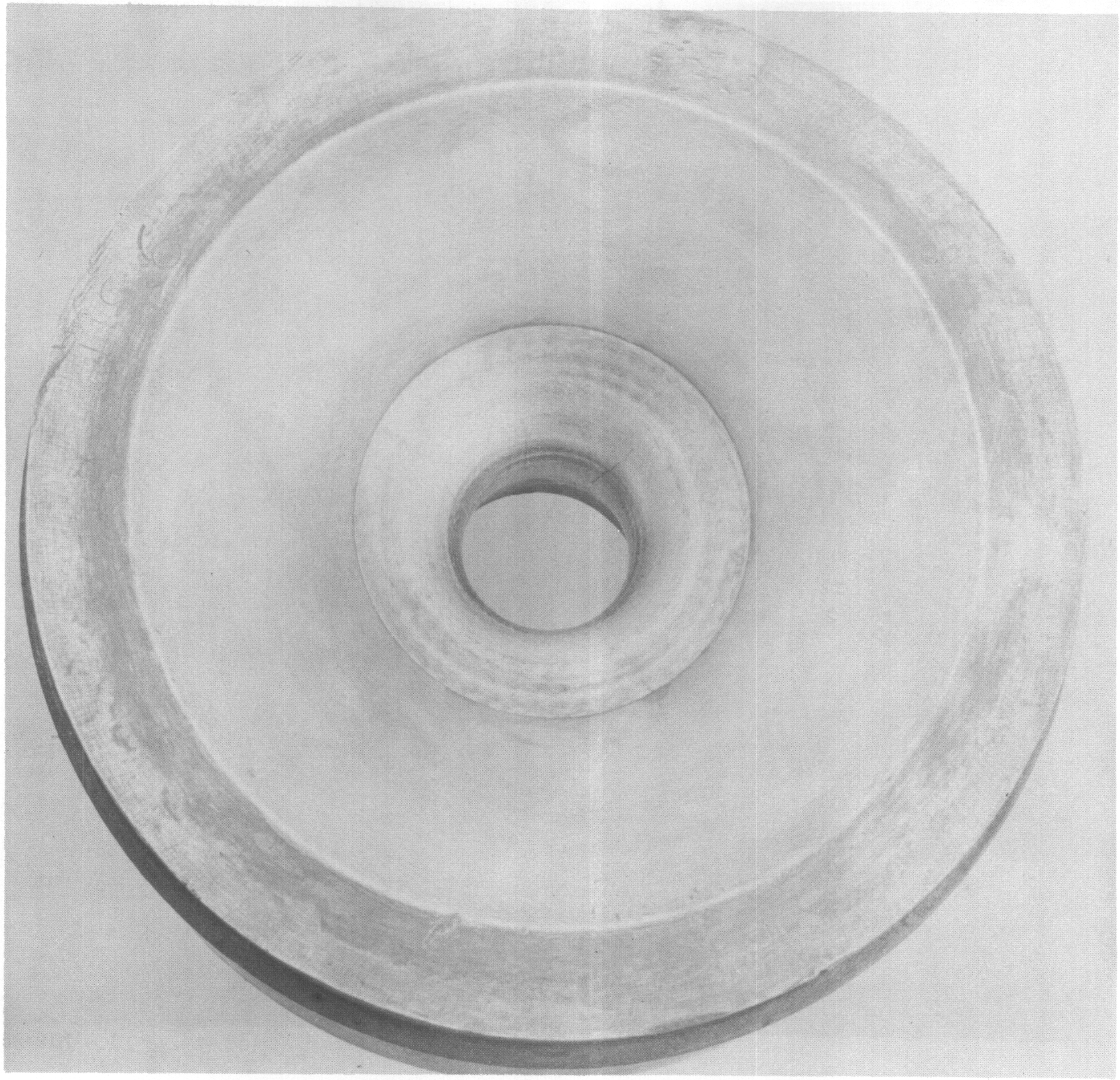
(d) NLGI No. 2 consistency.



N19486

FIGURE 24. APPEARANCE OF POLISHED DIE WITH HAYNES STELLITE ALLOY NO. 3 INSERT AFTER EXTRUSION OF FOUR Ti-3Mn-COMPLEX BILLETS AT 1700 F

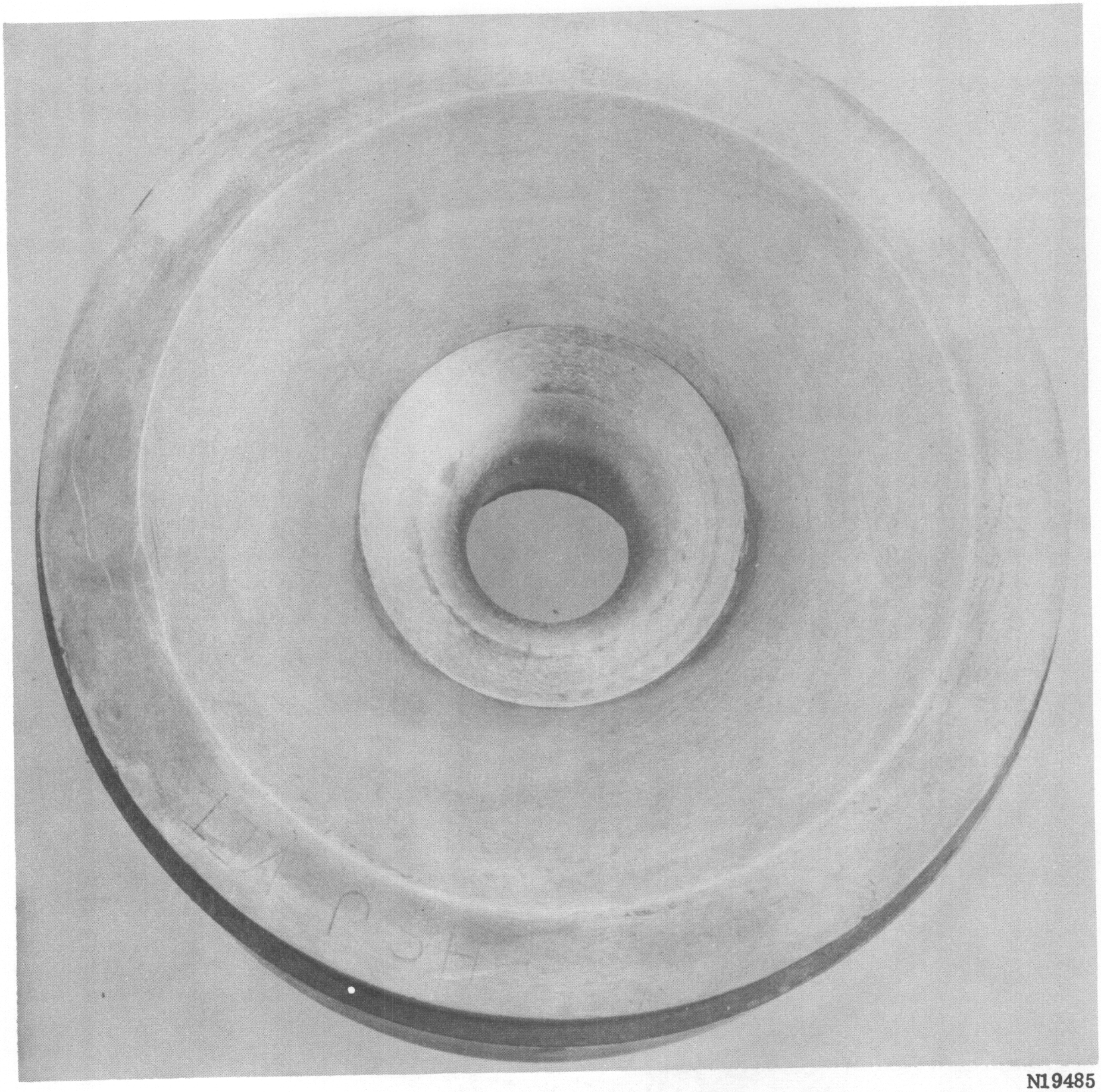
Cracks occurred in second test and increased in size as extrusion was continued. Diameter of die land increased from 0.996 to 1.000 inch during tests.



N19492

FIGURE 25. APPEARANCE OF POLISHED DIE WITH HAYNES STELLITE ALLOY NO. 6 INSERT AFTER EXTRUSION OF FOUR Ti-3Mn-COMPLEX BILLETS AT 1700 F

Cracks occurred during second test. Diameter of die land increased from 0.997 to 1.001 inch during tests.



N19485

FIGURE 26. APPEARANCE OF POLISHED DIE WITH HAYNES STELLITE STAR J-METAL INSERT AFTER EXTRUSION OF FOUR Ti-3Mn-COMPLEX BILLETS AT 1700 F

Cracks occurred during second test but were not so severe as those in the other cobalt-base alloys. Diameter of die land increased from 0.997 to 0.999 inch during tests.

Haynes Stellite Insert	Initial Diameter, in.	Final Diameter, in.
Alloy No. 3	0.996	1.000
Alloy No. 6	0.997	1.001
Star J-Metal	0.997	0.999

Firth Heavy Metal, WEX. Firth Heavy Metal is a powder-metallurgy product whose principal constituent is tungsten. Small percentages of nickel and copper powders are added to improve the machinability of the sintered product. The hardness of Firth Heavy Metal is about 300 BHN (32 R_C).

The Firth Heavy Metal used in making the die insert for this work was the WEX 85-10-5 composition. The machined insert was carburized to increase the wear resistance of the surface.

Three billets were extruded through the Firth Heavy Metal die, using Fiske-BMI Experimental Lubricant No. 4. Data and observations on these tests are presented in Table 12. The Firth Heavy Metal exhibited better thermal-shock resistance than the carbides or cobalt-base materials, no surface cracks occurring during the extrusion tests. Also, this material apparently has good resistance to seizing by the titanium. Die pickup was very slight in each of the tests and was not adherent.

Although the insert showed no signs of wear, the dimensions of the die opening could not be maintained. Initially, the inside diameter of the insert at the die land was 0.989 inch. After the third test, the diameter of the die land varied from 0.991 inch at the front to 0.979 inch at the die exit, indicating that the insert was upsetting. As the die land became increasingly tapered, its length was insufficient to even out the slight differences in metal flow that occurred within the billet. The unevenness of the metal flow through the die caused the bar to "chatter" as it extruded. The bars extruded in Tests T-56 and T-60 had good surface finishes, but dimensional tolerance was destroyed because of the die chatter.

A photograph of the Firth Heavy Metal die, taken after the third test, is shown in Figure 27. The die was not polished, but was swabbed lightly with dilute nitric acid to clean the surface.

Effect of Lubricant on Surface Finish

The surface finish of the bars extruded with Fiske-BMI Experimental Lubricant No. 4 was generally very good. Variations existed in the surface

TABLE 12. DATA AND OBSERVATIONS ON THE EXTRUSION TESTS WITH THE FIRTH HEAVY METAL DIE INSERT(a)

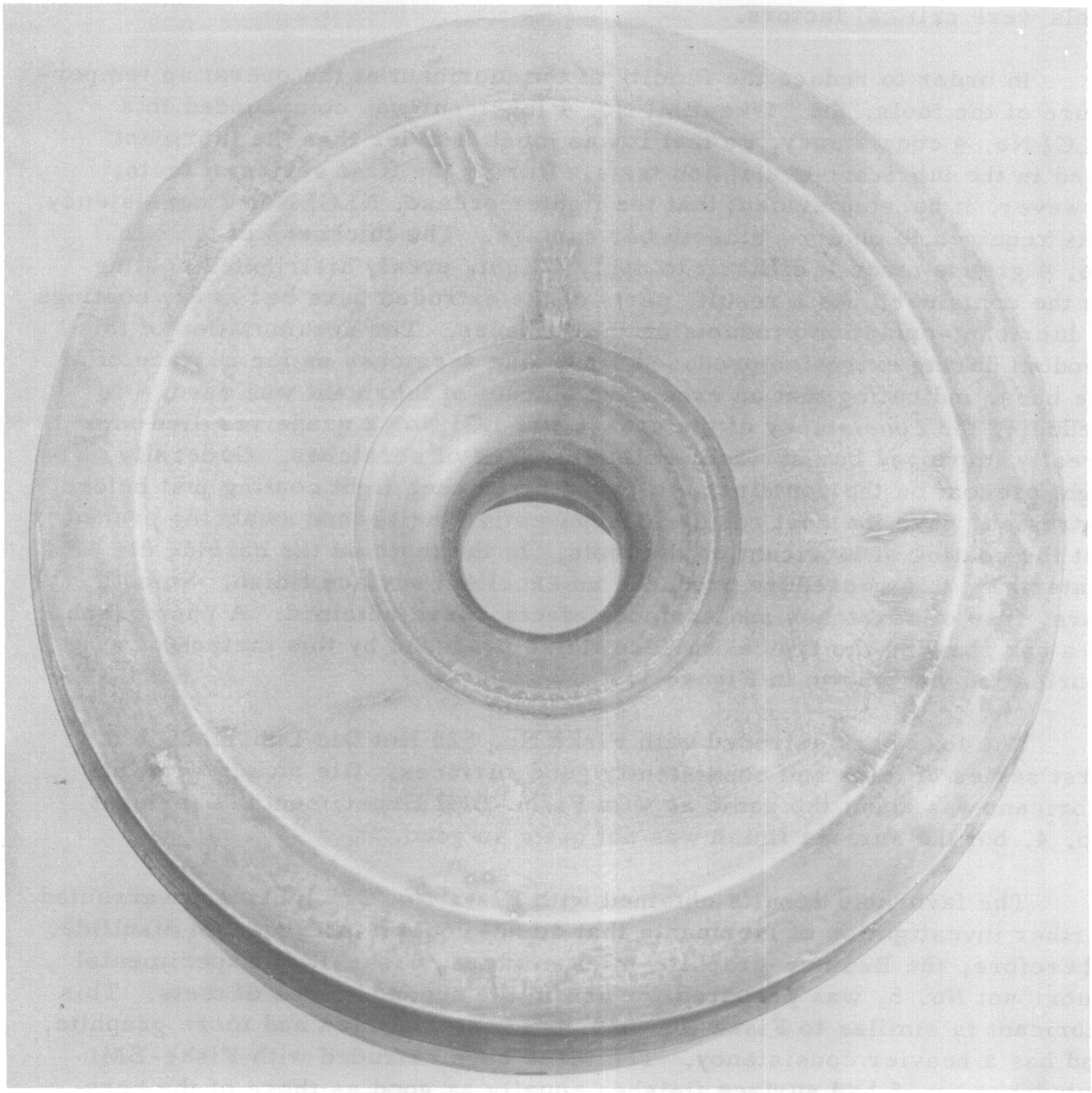
Test	Lubricant	Die Temperature, F		Extrusion Time, sec	Length Extruded, in.	Average Extrusion Rate, in./sec	Extrusion Pressure, 1000 psi			Die Pickup(c)	Surface Finish of Bar(c)
		Before	After				Initial	Run-Out	Final		
T-52	Fiske-BMI No. 4(d)	650	900	17.6	92	5.2	100	97	102	VL	F
T-56	Ditto	600	850	25.0	87	3.5	103	103	104	VL	G
T-60	"	600	950	27.0	71	2.6	102	102	103	VL	G

(a) Extrusion temperature 1700 F; extrusion ratio 21:1.

(b) Average speed of extruded bar leaving die.

(c) See pages 43 and 46 for an explanation of these symbols.

(d) NLGI No. 2 consistency.



N19490

FIGURE 27. APPEARANCE OF POLISHED DIE WITH FIRTH HEAVY METAL INSERT AFTER EXTRUSION OF THREE Ti-3Mn-COMPLEX BILLETS AT 1700 F

Firth Heavy Metal had good resistance to thermal shock and to seizing by the titanium. Insert was deformed during extrusion. Diameter of the die land, initially 0.989 inch, varied from 0.991 inch at entry to 0.979 inch at exit after third test.

finish of the bars extruded with a given die, however, and it was evident that the consistency and the manner of application of the lubricant to the tools were critical factors.

In order to reduce the fluidity of the lubricant at the operating temperature of the tools, the Fiske-BMI No. 4 lubricant was compounded to a NLGI No. 4 consistency, so that it was much heavier than the lubricant used in the lubricant-evaluation tests. During the first series of tests, however, it became evident that the lighter grease, NLGI No. 2 consistency, was required to obtain a smooth bar surface. The thickness of the NLGI No. 4 grease made it difficult to apply a light, evenly distributed coating on the container. As a result, parts of the extruded bars had heavy coatings of lubricant-oxidation products on the surfaces. The accumulation of this product during extrusion produced many fine scratches on the surface of the bars, indicating that an excessive amount of lubricant was used. Reducing the consistency of the grease to NLGI No. 2 grade resulted in a greatly improved bar surface, relatively free of scratches. Generally, a light precoat on the container, followed by another light coating just before extrusion, gave the best results. Apparently, the second swabbing evened out the coating of lubricant on the tools. In the tests on the carbide die materials, this procedure produced an excellent surface finish. Smooth bars, free of scratches and surface defects, were obtained. A photograph of a bar showing the typical surface finish produced by this method of lubrication was shown in Figure 1.

The four bars extruded with Fiske No. 525 Hot Die Lubricant in the first series of tests had consistently good surfaces. Die pickup with this lubricant was about the same as with Fiske-BMI Experimental Lubricant No. 4, but the surface finish was not quite so good.

The favorable results obtained with Fiske No. 525 lubricant warranted further investigation of lubricants that do not contain molybdenum disulfide. Therefore, the Bentone-graphite-mica mixture, Fiske-BMI Experimental Lubricant No. 5, was prepared for use in the second series of tests. This lubricant is similar to Fiske No. 525, but contains mica and more graphite, and has a heavier consistency. The three bars extruded with Fiske-BMI Lubricant No. 5 had surface finishes equally as good as those of the bars extruded through the same dies with Fiske-BMI Experimental Lubricant No. 4. Die pickup also was about the same with the two lubricants. These tests indicate that it may be possible to eliminate molybdenum disulfide in the lubricants for extruding titanium, but further work would be required to verify these results.

The two bars extruded with the Fiske No. 514A-10AP lubricant, which contained powdered aluminum, had very good surface finishes. The use of powdered metals, such as aluminum, in extrusion lubricants had not been considered prior to these tests. The favorable results obtained, however, indicate that further work should be done with lubricants of this type.

Metal-Flow Patterns

Examination of the billet skulls indicated that no stagnant-metal zone occurred with the 130-degree conical dies. An etched section of the skull of a Ti-3Mn-complex billet, illustrating the typical metal-flow pattern that occurred, is shown in Figure 28. Generally, the flow of metal into the die opening was uniform from all parts of the billet. In several instances, however, nonuniform metal flow occurred, a part of the billet appearing to be restrained from flowing into the die opening. As a result, the outer skin of the billet was enfolded toward the center of the billet and a layer of lubricant and oxide was trapped within the billet, as is shown in Figure 29. This flow condition can be caused by (1) insufficient lubrication on part of the die, (2) poor alignment between the conical die and the container, or (3) large temperature gradients within the billet. Although this condition did not have any noticeable effect on the surface or soundness of the bars extruded in this work, it would produce seams in the surface and laminations in the interior of extrusions produced from longer billets. A photograph showing the typical appearance of a billet skull in which this type of flow occurred is shown in Figure 30. It is obvious from this photograph that the quality of the extrusion would be influenced by such a condition.

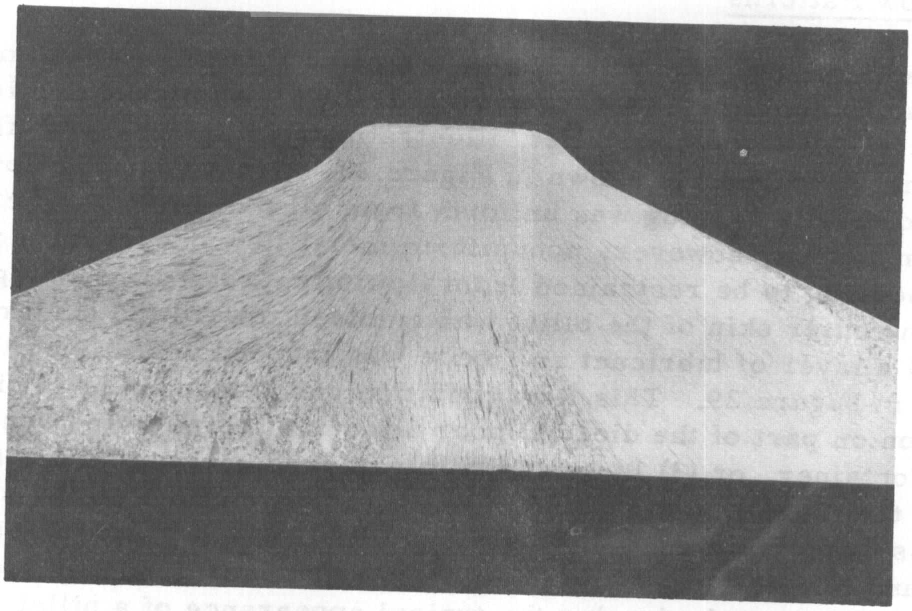
Discussion on Die Materials

The tests conducted in this work were not intended to provide data indicative of the relative lifetimes of the various die materials, since this would be beyond the scope of the research program. Rather, the materials were tested to determine the conditions necessary to obtain titanium extrusions with a high-quality surface finish and good dimensional tolerance. Although several lubricants were used in this work, the dies were evaluated on the basis of their performance with Fiske-BMI Experimental Lubricant No. 4.

Generally, the best surface finish was obtained with the carbide dies. These materials exhibited the best high-temperature abrasion resistance and the least tendency toward seizing by the titanium. Although cracks occurred in the carbide inserts, there was no change in the dimensions of the dies. Varying amounts of pickup occurred in all of the tests with these inserts, but the titanium was not very adherent and could be removed easily without damage to the die surface.

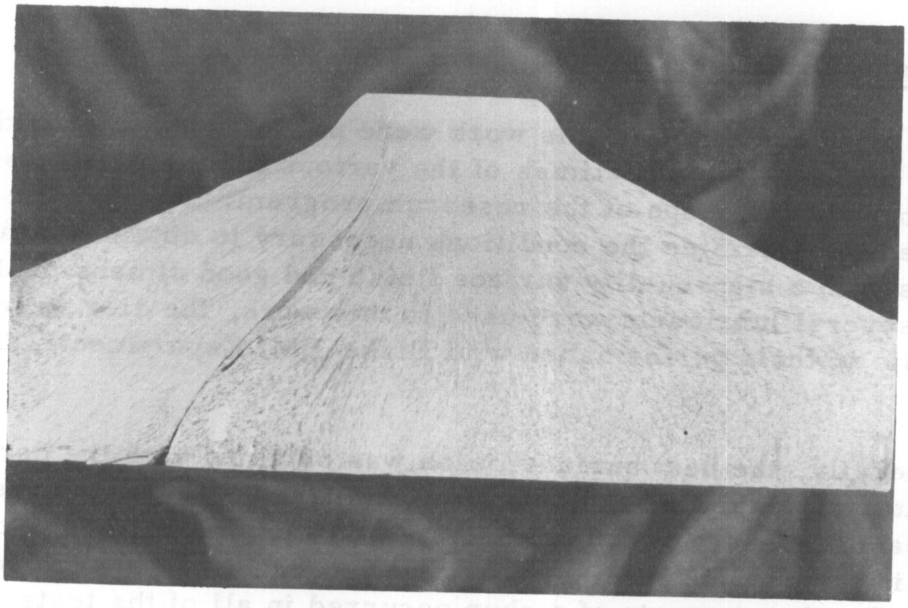
Lower extrusion pressures, under the same test conditions, were required with the carbide dies than with the other die materials. This is interpreted as being a function of the lesser tendency of the titanium to seize on the carbide dies than on the other die materials.

A good surface finish was obtained with the cobalt-base-alloy dies, also, but slight die wear occurred and the inserts required grinding and



N19308

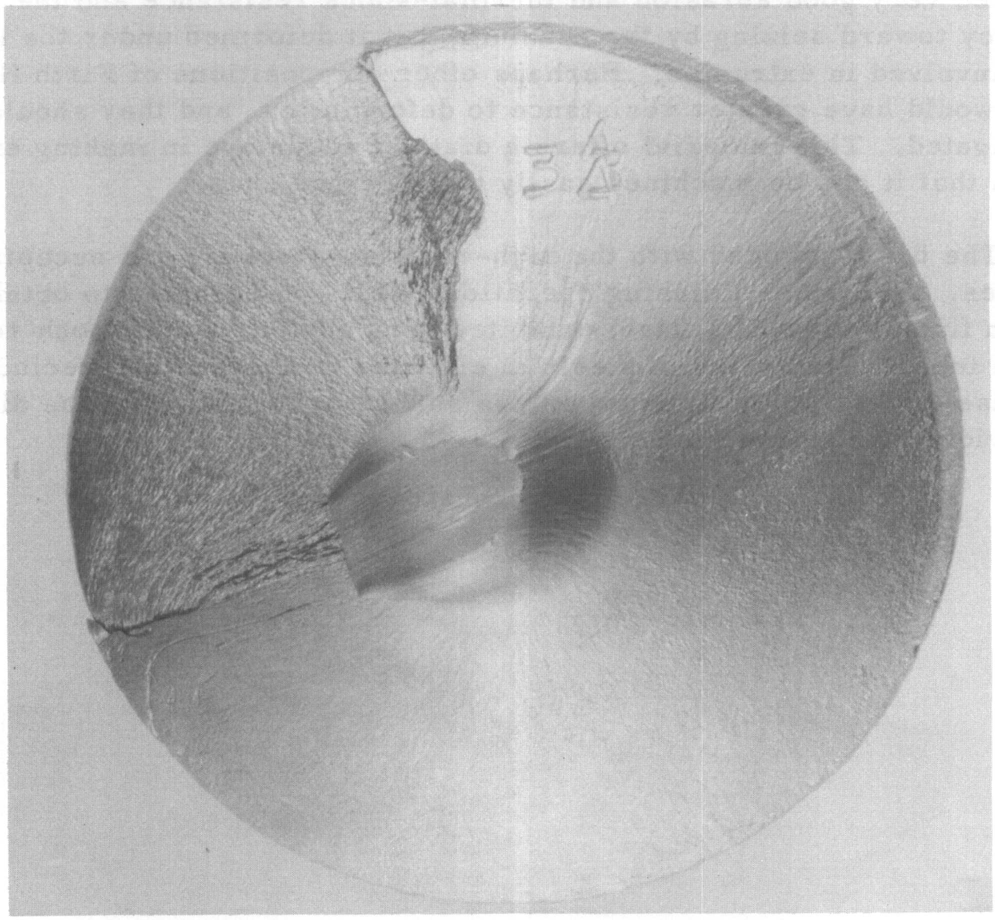
FIGURE 28. ETCHED SECTION OF THE SKULL OF Ti-3Mn-COMPLEX BILLET EXTRUDED THROUGH 130-DEGREE CONICAL DIE



N19307

FIGURE 29. ETCHED SECTION OF THE SKULL OF Ti-3Mn-COMPLEX BILLET SHOWING NONUNIFORM METAL FLOW

Note that lubricant has been folded within billet.



N19510

FIGURE 30. TYPICAL APPEARANCE OF THE SKULL OF A BILLET IN WHICH NONUNIFORM METAL FLOW OCCURRED

polishing after each test. These materials exhibited a greater tendency toward seizing by the titanium than did the carbides. Also, cracking was more severe in the cobalt-base alloys and, as a result, several of the bars extruded with these dies exhibited die markings.

The surface finish obtained with the Firth Heavy Metal die would have been very good, had not the chatter marks occurred. This material exhibited very good abrasion and thermal-shock resistance and the least tendency toward seizing by the titanium, but it deformed under the high loads involved in extrusion. Perhaps other compositions of Firth Heavy Metal would have greater resistance to deformation, and they should be investigated. This material offers a distinct advantage in making extrusion dies in that it can be machined easily to a desired shape.

The bars extruded with the high-tungsten-steel die had acceptable surfaces, but further finishing operations would be required to obtain a smooth finish. The XDL steel exhibited very good thermal-shock resistance, but the inherent softening of steels at elevated temperatures precludes their use for extruding titanium unless sufficient insulation of the die face is provided by the lubricant.