FOREWORD

This Final Technical Documentary Report covers all work performed Contract AF33(657)-8798 from July 1962 through 30 June 1964.

This contract with Nuclear Metals, Division of Textron Inc., West Concord, Massachusetts, was initiated under Manufacturing Methods Project 7-946, "Development of Die Materials and Dies". It was administered under the technical direction of Mr. T. S. Felker of the Metallurgical Processing Branch (MATB), Manufacturing Technology Division, AF Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

Messrs. J. G. Hunt, Research Ceramist, and J.L. Klein, Vice President and Manager of Materials Research and Development, were the Nuclear Metals personnel most actively engaged in the program. Others who contributed to the work at Nuclear Metals were Mr. P. Loewenstein, Assistant to the Acting General Manager, Mr. John M. Siergiej, Manager of Mechanical Metallurgy, and Dr. A. R. Kaufmann, Technical Director and Vice President. This report has been given the Nuclear Metals internal report number NMI-9700.24.

This project has been accomplished as a part of the Air Force Manufacturing Methods Program, the primary objective of which is to develop, on a timely basis, manufacturing processes, techniques and equipment for use in economical production of USAF materials and components. The program encompasses the following technical areas:

Metallurgy - Rolling, Forging, Extruding, Casting, Fiber, Powder.
Chemical - Propellant, Coating, Ceramic, Graphite, Nonmetallics.
Electronic - Solid State, Materials and Special Techniques, Thermionics.
Fabrication - Forming, Materials Removal, Joining, Components.

Suggestions concerning additional Manufacturing Methods development required on this or other subjects will be appreciated.

ABSTRACT

The utility of massive ceramic dies for the precision high temperature extrusion of complex shapes of refractory metal alloys has been demonstrated. Precision extrusion of shaped structural steels through ceramic coated dies was gained by refinement and close control of extrusion parameters.

Refractory ceramics of high compression strength like ZrO_2 or TiC were found capable of extruding TZM molybdenum alloy at 3700°F and 75 tsi producing 2.0 by 1.3 by 0.250 inch Tee sections in lengths up to at least fourteen feet with dimensional runout of less than three mils and surface smoothness of 63 rms. Ceramic coated dies, like Al_2O_3 coated tool steel, were found capable of extruding 4340 alloy steel at 2300°F and about 50 tsi, producing 2.0 by 1.5 by 0.062 inch Tee sections in lengths up to at least forty feet with dimensional runout of less than four mils and surface smoothness of 125 rms. The performance of massive ceramic dies in TZM extrusion indicates that massive ceramic dies would produce steel extrusions superior to those obtained with ceramic coated steel dies.

This technical documentary report has been reviewed and is approved.

FOR THE DIRECTOR

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I. INTRODUCTION

A. Background

Advancing technology requires the use of structural shapes of high strength steels and refractory metals. The desired shapes such as T, U, and H, can be made by hot extrusion, in which a cylindrical billet is forced through a shaped die to produce a length of material with desired cross-section. Although conventional materials can be readily fabricated by this technique, the hot extrusion of refractory materials requires high billet temperatures which are beyond the endurance of hardened steel dies.

Development programs, sponsored in recent years by the Air Force,(1) have indicated promising die technology for the extrusion of high temperature billets into rods and structural shapes. This technology, which has become conventional refractory die practice, is employed on several development programs for shaped extrusions, sponsored by the Air Force.(2,3,4)

The research effort in these programs was directed toward decreasing the amount of heat that passed into the die during extrusion. This was approached by (1) decreasing extrusion time by increasing extrusion speed, (2) decreasing the rate of heat flow into the die from the billet by using insulating lubricants of glasses, and (3) insulating the die with ceramic coatings.

Although increased extrusion speed (achieved by specially designed accumulator-driven presses) and improved lubrication were important factors, the major feature of the success of these dies was the use of hard, thermally insulating die coatings which were not only relatively erosion resistant, but also prevented the overheating of the underlying, supporting steel substrate. The coating materials, usually alumina (Al_2O_3) or zirconia (ZrO_2) were applied in thicknesses of about 25 to 50 mils by the flame or arc-spraying processes onto hardened steel substrates with roughened surfaces; the coated die then required grinding for precision and was capable of only one high-temperature extrusion of a rod or a simple shape.

The major failing of the coated dies was the spalling or erosion of the coating surface under the more severe conditions. Die degradation became progressively worse with increasing extruded length, cross-sectional complexity, extrusion force and billet temperature. Coating failures of this type led to poor dimensional uniformity and striated surfaces. The marginal performance of the coated dies was generally attributed to insufficient strength and thermal shock resistance in the coating, as well as insufficient bonding to the substrate aggravated by the difference in coefficient of expansion. Consequently, it was apparent that improved dies were necessary for the precision extrusion of complex shapes of steel and refractory metal alloys.

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Improvement in die performance and thus smoother extrusions with greater dimensional uniformity of complex shapes were presumed by the Air Force and specialists in industry to be gained from development of die designs based on three concepts: improved coated dies, metal-fiber reinforced ceramic dies, and refractory intermetallic dies.

B. Purpose and Objective of Program

1. Purpose

Some die development effort has been included in various prior programs sponsored by the Air Force for the prime purpose of advancing the technology of the hot extrusion of various metals; but, in the effort to produce the required end products, extensive work to develop better or more economical dies has not been possible within the limited scopes of the various programs. Accordingly, the Air Force originated this program, Contract AF 33(657)-8798, in June 1962. The purpose of this program was to develop new and improved dies for the high temperature extrusion of complex structural shapes of steels and refractory metal alloys. These new dies and associated technology would form the basis for the fabrication of a variety of shapes and materials required for Air Force applications. For this reason, specific objectives were chosen which involve development of a die which can endure extrusion conditions which are more severe than is typical for a wide variety of materials.

2. Objective

The specific objective of the proposed program was to develop a die which can convert reference billet materials into specific shapes with greater length, surface smoothness, and dimensional uniformity, than can be achieved by the commercially available zirconia coated steel die, which has been, up to now, the standard high temperature extrusion die.

Prescribed reference alloys for experimental extrusions were AISI-SAE Type 4340 steel (0.40 C, 1.75 Ni, 0.80 Cr, 0.25 Mo) and molybdenum alloy TZM (Mo-0.5Ti-0.08Zr). The reference cross section of the metal to be extruded was a Tee having a flange approximately two inches wide and a stem approximately one inch deep; the maximum thickness of flange and stem were 0.063 inch for the steel and 0.250 inch for the TZM alloy.

Demonstration of success in meeting the program objective was to be the extrusion of twenty or more feet of uniform Tee shape with minimum deterioration of the die. The extrusion equipment was to enable stressing the ram to 90 tsi, and furnaces were to be capable of heating the TZM billets to 3400°F.

In the original program scope, three types of dies were to be investigated consecutively: ceramic-coated metals (Phase I), metal fiber reinforced ceramics (Phase II), and refractory compounds and ceramic based dies (Phase III). As the work progressed, the advantages of concurrent work on all three phases became apparent, and the program was changed accordingly.

Another change in the objective of the program was made involving steel extrusion. Instead of testing experimental dies under standard extrusion conditions, standard coated dies were used in an experiment to determine near optimum extrusion conditions for generating steel Tees. This scope change, in compliance with the request of the program's Project Engineer, Mr. T. Felker, MATB, was based on his judgement, which was proven correct, that the solution to the problem of extruding thin sections of structural steels might also be effected by the optimization of extrusion parameters, even though prior programs in this area were unsuccessful. Section G, pages 111 to 122, reviews this optimization concept in detail.

II. SUMMARY

A. General

The experimental work of the die development was divided into seven parts. These are:

(1) Preliminary plans (Section A)

(2) Preliminary experiments (Section B)

(3) Fabrication of die materials (Section C)

(4) Testing of round dies with steel billets (Section D)

(5) Testing of round dies with TZM billets (Section E)

(6) Testing of shaped dies with TZM billets (Section F)

(7) Testing of shaped dies with steel billets (Section G)

These divisions are made for simplicity in describing the work and do not follow the chronology of experiments and the results obtained. A short chronology is given in Section B below.

B. Chronolcgical Review

Although the period of performance of the program was originally planned for eighteen months, it was extended due to scope changes, and delays in experimental materials from commercial vendors. The program was performed over a two-year period from July 1962 to July 1964. The chronology of planning, experiments, and results are summarized below in segments of quarters or three-month intervals.

1. First Quarter

After a review of extrusion facilities and reported materials properties, work performed during the first quarter (July through September, 1962) was concerned principally with selection of die materials, fabrication techniques, evaluation procedures, and die support systems. For ceramic-coated metal substrate dies, steel and/or refractory metals were chosen with zirconia-based coatings applied by plasma and flame spraying. For fiber-reinforced dies, hot-pressed zirconium silicate, and silicon nitride, each reinforced with tungsten fibers, were chosen. The basis for selection of these materials for fiber-reinforced dies were: (1) tungsten has a high elastic modulus, high strength over a wide temperature range, and is readily available in a form that can be converted into fibers; (2) zirconium silicate and silicon nitride do not react appreciably (if at all) with tungsten under the die fabrication conditions; and (3) the higher expansion coefficient of tungsten results in the desired residual compressive stresses in the ceramic matrix after cooling from the hot-pressing operation used for making the dies. For massive refractory compound dies (ceramic-based dies) various refractory silicides, carbides, borides and nitrides, either self- or otherwise-bonded, and made by hot-pressing or sintering, were selected.

Two procedures were selected for evaluation of die materials: (1) an indentor test, intended to be an economical and simple means of simulating extrusion conditions; and (2) actual extrusion through die materials that showed promise from the indentor test. Initially, materials were to be fabricated and tested first as indentors, then as round dies, and finally as Tee dies. Each type of indentor specimen was to be tested at 2200°F with steel billets and then 3400°F with TZM billets.

Although external die supports are necessary for Tee dies (because of the dimensions of the Tee relative to the extrusion liner), internal supports were planned for round dies for convenience and economy. In addition, since a portion of even an external die sometimes extends into the extrusion container to prevent leakage of metal under high pressures during extrusion, development of supports for internal round dies was anticipated to assist in the development of supports for external Tee dies.

2. Second Quarter

Work performed during the second quarter (October through December, 1962) was concerned primarily with preliminary experiments to verify and delineate further the planning of the program. Preliminary fabrication trials resulted in successful hot-pressing of metal-bonded oxides (tungsten powder plus Al_2O_3 and tungsten powder plus ThO_2). In extrusion tests of AISI 4340 steel through round dies, these metal-bonded oxide dies were superior to uncoated H-21 steel dies. Hot-pressing techniques were investigated for a number of ceramic and metal-ceramic dies. Three types of die supports -- called "soft", "hard", and "isostatic" -- were tried, and none was completely successful.

3. Third Quarter

In the third quarter (January through March, 1963) progress was made in the following areas: ceramic-based dies, including oxide-bonded-borides, -carbides, -nitrides, and -silicates, were fabricated by hot-pressing; two alloys, W - 5.5^w/o Ru and Ta - 6^w/o Ru, were made by melting and forging, and a number of round, ceramic-based dies, including TaC-W, TiC-Ni-Mo, TiB_2, ZrC, and ZrO_2, were obtained from outside sources. Most of these materials were ground and prepared for testing. A Tee-shaped die of W (powder) - 33^v/o ThO_2 was successfully fabricated by hot-pressing. In order to have raw materials for fiber reinforced ceramics, two processes were developed to produce tungsten fibers: one by swaging in a steel shroud, and the other by rolling in a steel can. Zirconium silicate and silicon nitride, each reinforced with tungsten fibers, were fabricated into round dies by dry-blending with subsequent hot-pressing. A technique for supporting brittle nibs was developed. The support design depended on having the force from the extrusion billet generate a compressive force on the nib to prevent failure. The value of refractory metals, particularly tungsten, was established as a promising substrate for coated dies. A high-temperature furnace was modified to provide a 2- to 3-second loading time for TZM and steel billets. The indentor test, conceived in the first quarter, was found to be an unsatisfactory means of screening die materials, and this test was abandoned.

4. Fourth Quarter

In the fourth quarter (April through June, 1963) progress was made in the areas of die materials fabrication and die materials testing. The fabrication of tungsten powder and fiber-bonded zircon and silicon nitride Tee dies was carried out by hot-pressing. Metallographic examination of round zircon-tungsten fiber dies indicated no cracking of the ceramic around the tungsten nor any appreciable chemical interaction between the two materials. The Ta-5, 5 Ru and W-6 Ru nibs made earlier (third quarter) were prepared for testing by Eloxing the internal die configuration. Steel supports for Tee dies were prepared, and Tee dies of ZrO_2, TiB_2, TiC, ZrB_2, and ZrC were purchased from commercial sources. In tests with steel billets at 2200°F, steel dies with ZrO_2-based arc sprayed coatings were found to be non-adherent, while massive ceramic and ceramic-based dies, which were dense or contained no low melting constituents, suffered negligible wear. Round, massive, ceramic (ZrO_2) dies withstood at least two TZM extrusions at 3400°F, a result which implies a greater degree of promise than dies with ZrO_2 coatings. Consequently, intensive investigation of coated dies with their inherent bonding problem was held in abeyance.

5. Fifth Quarter

In the fifth quarter (July 1 through October 15, 1963), progress was made in the areas of die materials fabrication, die materials testing, extrusion tooling and extrusion process improvement. Fabrication by hot-pressing of ceramic-based Tee dies was continued. Composites of $ZrSiO_4$-W, Si_3N_4-W, $MoSi_2$-Al_2O_3, ZrB_2-Al_2O_3, and TiB_2-Al_2O_3 were successfully fabricated. Hot press mold design was modified to fabricate the ceramic more nearly to finished size. Improvements in tungsten fiber production were developed. Testing of round, metal-bonded ceramic dies with steel billets, as well as testing of round ceramic-based dies and metallic dies with TZM was completed. Preliminary tests of ceramic-based Tee dies were carried out. Results indicated that: (1) the ceramic-based Tee dies had promise, and (2) greater support for steel casings was necessary.

6. Sixth Quarter

In the sixth quarter (October 15 through December 31, 1963) progress was made in the areas of die materials fabrication and planning of Tee die tests with TZM billets. Fabrication by hot-pressing of ceramic-based dies with .062-inch internal Tee section was attempted. Although successful dies were not obtained, data indicating proper conditions were gained. These include more uniform powder fill, improved alignment of mold parts, and higher strength mandrel materials. Plans were made for evaluating Tee dies of the following die materials: ZrO_2, TiC, TiB_2, ZrC, TaC-W, $ZrSiO_4$-W, Si_3N_4-W, and TiB_2-Al_2O_3.

7. Seventh Quarter

In the seventh quarter (January 1 through March 31, 1964) progress was made in the areas of finishing experimental Tee dies, assembling them into die supports, incorporating a new vacuum furnace and lubrication facility, and performing a preliminary test with modified tooling and new equipment.

8. Eighth Quarter

In the eighth quarter (April 1 through June 30, 1964) progress was made in the areas of testing the remaining ceramic based Tee dies, testing the Al_2O_3 coated steel dies obtained without cost from Republic Aviation by the sponsor, and drafting and submitting the final report. The results of these tests led to the following conclusions:

Refractory ceramics of high compression strength like ZrO_2 or TiC were found capable of extruding TZM molybdenum alloy at 3700°F and 75 tsi producing 2.0 by 1.3 by 0.250 inch Tee sections in lengths up to at least fourteen feet with dimensional runout of less than three mils and surface smoothness of 63 rms. Ceramic coated dies, like Al_2O_3 coated tool steel, were found capable of extruding 4340 alloy steel at 2300°F and about 50 tsi, producing 2.0 by 1.5 by 0.062 in Tee Sections in lengths up to at least 40 feet with dimensional runout of less than four mils and surface smoothness of 125 rms. The performance of massive ceramic dies in TZM extrusion indicates that massive ceramic dies would produce extrusions superior to those obtained in the ceramic coated steel dies.

III. EXPERIMENTAL WORK

A. Preliminary Plans

Since little technology on ceramic materials for hot extrusion dies was available, preliminary plans were formulated to explore the most promising routes of investigation. This early work took the form of brief surveys of present technology and then selection of the most promising routes of development.

1. Survey of Present Technology

a. Commercial Extrusion Practice

Visits were made to two facilities. Both of these facilities were concerned primarily with extruding stainless and other types of steels. Both used glass on the billets but no lubrication in the liner. One was able to extrude through a multi-holed die, while the other did not demonstrate this capability. Both used uncoated dies of cast high-speed steel, and four or five extrusions were obtained from a die before it was discarded or repaired. The cost of a die is somewhere between four and ten dollars.

(It should be noted that both extruders are interested in reducing the cost of dies, even though it is insignificant compared to the cost of dies currently being used for refractory metal shapes in various experimental programs. It should not be inferred from these observations that further development of dies for extruding steel shapes is not necessary. The present program is for Tees with a section thickness of .063 inch; the thinnest section observed in the products of both commercial extruders was about 0.250-inch. The amount of reduction and, consequently, the magnitude of the forces required will be greater for the thinner section and will lead to greater deterioration of dies than is now encountered in current steel practice).

b. Non-Commercial Extrusion Practice

Visits were made to the extrusion facilities at Wright Field, Lewis Research Center of NASA, Tapco Group of Thompson-Ramo-Wooldridge, and to the Brackenridge Laboratory of Allegheny Ludlum Steel Corporation. All of these installations are extruding refractory metals on government programs. In all cases, coated dies were used, but there was no agreement on whether flame or plasma spraying was superior or whether Al_2O_3 coatings were superior to ZrO_2 coatings. Glass lubrication was used except at Tapco where, in low reduction extrusion of tungsten at 3000°F, the interaction of tungsten oxide with the die coating provided the lubrication.

2. Selection of Experimental Die Systems

For the development of dies, completely new materials and combinations of materials were considered. In this section, the factors influencing the choices of materials and combinations of materials selected for each of the three classes of dies are discussed. Some of the materials of the types which were under consideration are listed in Table 1, together with some of their known physical properties.

a. Ceramic-Coated Metallic Materials

Ceramic coatings were to be applied to both steel and refractory metal substrates. Refractory metal substrates chosen for initial study were tungsten and molybdenum-base alloys, both of which are materials of relatively great mechanical strength at high temperature. These substrates also have expansion coefficients that approximate some of the prospective coatings and, therefore, were expected to be more compatible with the coatings than steel. Both wrought and sintered refractory metal substrates were to be studied. In studying the latter, the effect of non-metallic additions on the strength and durability of the substrate was to be explored.

Coatings selected for use on refractory metals were to form good bonds with the substrate, and were to be hard, oxidation resistant, and non-reactive with the substrate or siliceous lubricants at high temperatures. Those considered for evaluation with respect to bondability, oxidation and erosion resistance were SiC, $MoSi_2$, TiB_2, ZrB_2, and their alloys.

Coated dies with steel substrates were tentatively to have three outer layers - a bonding layer, an insulating layer, and a hard facing layer. The bonding layers will be metallic substances such as nichrome alloy; the insulating layer was to be zirconia with various stabilizers, or thoria; the hard facing layer will be hard materials similar to those considered for use on refractory metal substrates. Gradations of one coating into another were to be studied in an attempt to overcome dissimilar and thus disruptive expansion coefficients between the steel and the coatings.

b. Metal-Fiber-Reinforced Ceramic Materials

In this category two systems were chosen for initial investigation. They were silicon nitride (Si_3N_4) reinforced with tungsten, and Zircon ($ZrSiO_4$) with tungsten.

Tungsten was selected as reinforcement for this application because of its high modulus of elasticity, high-temperature tensile strength, availability as filaments, and chemical stability with many ceramic

TABLE 1

SOME PROPERTIES OF REFRACTORY MATERIALS

(All data from the literature)*

Material	% Expansion 0-1800°F	Approx. Melt or Decomp. (°F)	Approx. Use in Air (°F)	Thermal Conductivity, $\frac{BTU/hr/ft^2}{^{\circ}F/ft^2}$		Hardness (Room Temp.)		Elastic Modulus (x10^{-6} psi)	Thermal Shock Resistance	Compressive Strength (x10^{+3} psi)
				100°F	2000°F	Mohs	Vickers			
Graphite	0.20	6900	700	74	24	2		1.3	Excellent	5
Metals:										
Cb	0.81	4400	1100	30	50				Excellent	
Ta	0.69	5400	900	30				27.	Excellent	
Mo	0.56	4800	900	84	50			50.	Excellent	
W	0.46	6200	1500	95	64			60.	Good	
Borides:										
HfB_2	0.54	5900	2000							
TaB_2	0.40	5700	2000	63						
ZrB_2	0.64	5500	2000			8	2200	50.		
CbB_2		5400	2000	10						
TiB_2	0.72	5000	2000	14		9+	3400	60.	Good	97
VB_2		4400								
TaB		4400	2000							
WB		4400	1500							
Nitrides:										
Si_3N_4	0.22	3450	2000	10				32.	Very Good	
HfN		6000	1000							
TaN		5600	1000							
BN	1.30	5300	2000	18.9	16.7	2		18.,15.	Very Good	40
ZrN		5300	1000	15	4					
TiN		5300	1000	25	5	9			Good	141
AlN	0.54	4300	1800			7	1500			

(Table continued on next page).

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* References 5 through 12 inclusive.

TABLE 1 (continued)

Material	% Expansion 0-1800°F	Approx. Melt or Decomp. ($^{\circ}$F)	Approx. Use in Air ($^{\circ}$F)	Thermal Conductivity, BTU/hr/ft^2 $^{\circ}$F/ft^2		Hardness (Room Temp.)		Elastic Modulus (x10^{-6} psi)	Thermal Shock Resistance	Compressive Strength (x10^{+3} psi)
				100°F	2000°F	Mohs	Vickers			
Carbides:										
HfC	0.61	7100	1000							
TaC	0.65	7000	1500	13		9+	2000	42.		
ZrC	0.64	6400	1500	12		8.5	2600	45.		238
CbC	0.69	6300	2000	8			2400	49.		
Ta_2C		6100								
TiC	0.75	5900	2000	20	3	9.5	3200	45.	Excellent	110
SiC	0.50	5100	2900	24	12	9.2	3500	68.	Very Good	200
Cr_3C_2		3400	2500				2800	39.		600
Al_4C_3		5100								
W_2C	0.37	5000	1000			9+	3000			
MoC		4800	1000			7.5				
WC	0.48	4700	1000			9+	3000	60.		284
B_4C	0.54	4400	1000	16		9.3	3700	42.		414
Oxides:										
ThO_2	0.92	5500	4900	5	1.5	6.5		21.	Fair-Poor	120
MgO	1.33	5000	4300	20	3.0	6		30.	Fair-Poor	
HfO_2		5000	4300						Poor	
ZrO_2	1.0*	4800	4600	0.9	1.0	6.5		22.	Poor	
CaO	1.28	4700	4300	8	4.5	4.5			Poor	
Al_2O_3	0.83	3600	3500			9	2800	55.	Good	426
BeO	0.88	4600	4400	100	10	9			Very Good	

(Table continued on next page).

* Depends on degree and type of stabilization.

TABLE 1 (continued)

Material	% Expansion 0-1800°F	Approx. Melt or Decomp. (°F)	Approx. Use in Air (°F)	Thermal Conductivity, BTU/hr/ft^2 / °F/ft^2		Hardness (Room Temp.)		Elastic Modulus (x10^{-6} psi)	Thermal Shock Resistance	Compressive Strength (x10^{+3} psi)
				100°F	2000°F	Mohs	Vickers			
<u>Complex Oxides:</u>										
$SrO_2 \cdot ZrO_2$	0.94	5100								
$SiO_2 \cdot ZrO_2$	0.45	4800	3400	3.5	2.2			24.	Good	56
$BeO \cdot SiO_2$	0.62	4800	3400							
$FeO \cdot Zr_2O_3$	0.87	4700								
$BaO \cdot ZrO_2$	0.83	4900								
$CaO \cdot ZrO_2$	1.02	4200								
$SiO_2 \cdot HfO_2$	0.32		3600							
<u>Silicides:</u>										
$MoSi_2$	0.83	3700	3200					59.	Good	250
WSi_2	0.81	3900								
<u>Alloys:</u>										
W-10 Ru		5500	1000				1150			
Mo-10 Hf		4400					495			

materials. The ceramic matrices chosen are: (1) materials of considerable strength in themselves, (2) materials having lower elastic moduli, than the tungsten reinforcement, (3) those having thermal expansion coefficients close to or lower than tungsten, and (4) those chemically compatible with tungsten.

It was believed that the relationship of the thermal expansion coefficients of the components is important in causing the matrix to be placed in compression following cooling after high temperature compaction. The ceramic matrix, if it has a higher expansion coefficient, may crack in tension because it has greater shrinkage than the metallic fiber during cooling. Lower elastic moduli were chosen for the matrix because the total stress that can be borne without rupture by the weaker material of the composite is determined by the relative quantities and elastic moduli of the two materials. Thus, stresses which would fracture the matrix material alone can be withstood by the composite.

The effect of addition of substances with high moduli and hardnesses, e.g., alumina (Al_2O_3) and silicon carbide (SiC), on the strength and wear resistance of the silicon nitride and zircon matrices, was to be studied should it appear desirable to do so. The relative volumes of ceramic and metal and the diameter and length of the metal wires were also to be studied.

c. Refractory Intermetallic Materials

Materials in this group were chosen from substances of great mechanical strength, high hardness, and resistance to high temperature degradation. These included various carbides of boron, silicon, titanium, zirconium, molybdenum, and tungsten, the diborides of titanium and zirconium, the silicides of molybdenum and tungsten, and, perhaps, various alloys such as 9 ZrB_2MoSi_2, which is marketed by the Carborundum Company under the name of Boride-Z. In addition, various tungsten-base alloys such as W-10 Ru, and W-10 Hf were to be evaluated.

Most of these materials are brittle and thus may require modification by the addition of metals such as titanium, zirconium, nickel, molybdenum, tungsten, rhenium or other materials. Two types of metal additions were to be formulated: those expected to bond the non-metallic particles together by some limited mutual solubility, and those expected to react and bond, in the process producing new substances or new phases. Materials of the latter type are represented by Mo and SiC producing Mo_2C and Mo_3Si. Materials of the former type are represented by W and ThO_2.

3. Selection of Fabrication Methods

Techniques for consolidating the die materials into shapes with the desired properties were considered. Coated non-refractory materials will be made primarily by machining the metal substrate and coating it by flame, plasma, or pyrolytic deposition. These techniques were also to be applied to some coated refractory metal substrates, in either the wrought, sintered, or hot pressed condition.

Hot pressing was selected as the procedure to prepare other groups of specimens. A suitable furnace is shown in Figures 1 and 2. Hot pressing was chosen because of its ability to (1) densify previously mixed, powdered materials of extreme refractoriness with or without the presence of liquid phase, (2) produce shapes that require little or no shaping after pressing, (3) densify composite materials which are exceedingly difficult to sinter, and, (4) obtain nearly theoretical density while retaining small grain size. In many cases non-metallics which have been hot pressed are superior to sintered materials, particularly where little is known of the sintering behavior.

The compositions of the three types of dies, ceramic-coated metals, metal-reinforced ceramics, and refractory intermetallics, were anticipated to be fabricable by hot pressing in graphite molds at temperatures between 2500° and 4500° at pressures up to 2.5 tons/in.^{2} The possibility of deleterious reaction with the molds was to be eliminated entirely or in part by coating the mold with non-reactive barriers such as BeO, Al_2O_3, or BN.

Dies of various compositions could be fabricated by hot pressing thoroughly mixed powders onto an appropriately shaped mandrel by the action of double acting pistons within a tight-fitting sleeve as shown in Figure 3. This figure shows details of the mold assembly in the furnace of Figure 1. If the graphite mold has a lower expansion coefficient than the die material being pressed, splitting of the hot pressed die on cooling could generally be prevented by use of a hollow mandrel. A hole within the mandrel also provided a sight tube for close control of temperature near the specimen. Coatings, or a series of coatings, could be achieved by painting a sticky slurry, or series of slurries, onto the mandrel. The coated mandrel and powder was charged into the mold. The powdered die composition was simultaneously densified and bonded onto the coating during the hot pressing operation. After cooling and removal of the mold from the furnace, the die was pressed out of the mold and the mandrel was machined away to yield a shaped, coated die which required little or no finishing.

4. Selection of Testing Methods

a. Indentor Tests

An indentor test was devised to select the experimental die systems in a meaningful and economical way. Two pieces of equipment were involved: a vertical hydraulic press and a horizontal graphite tube furnace energized by induction. Specimens composed of the experimental

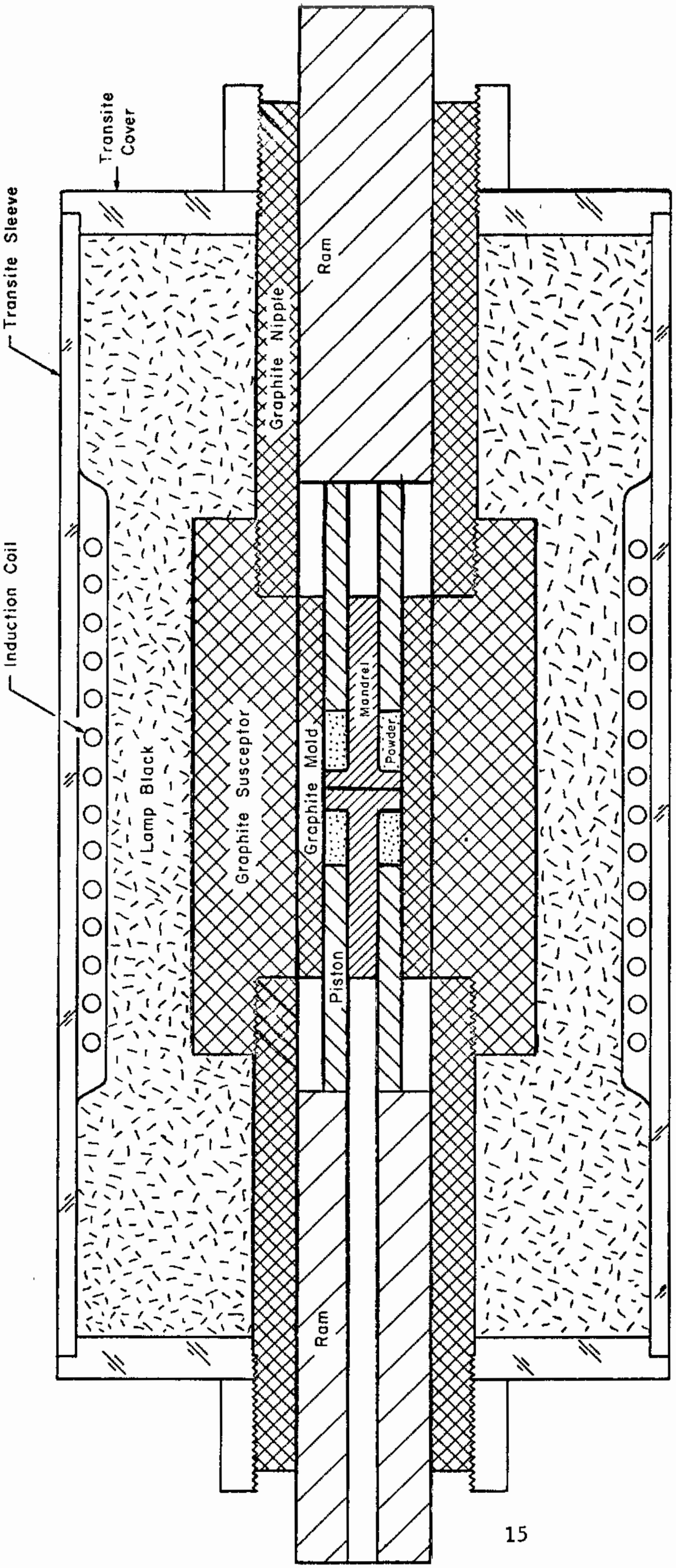

Figure 1. Hot Press Furnace for Die Fabrication.

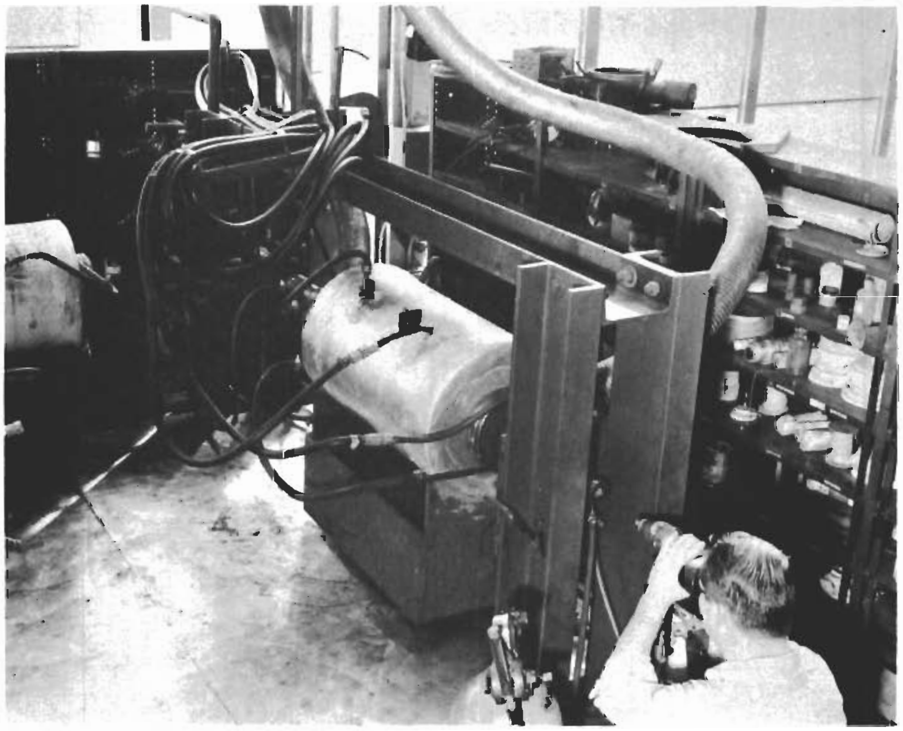

Figure 2. Photo of Hot Press Furnace for Dies.

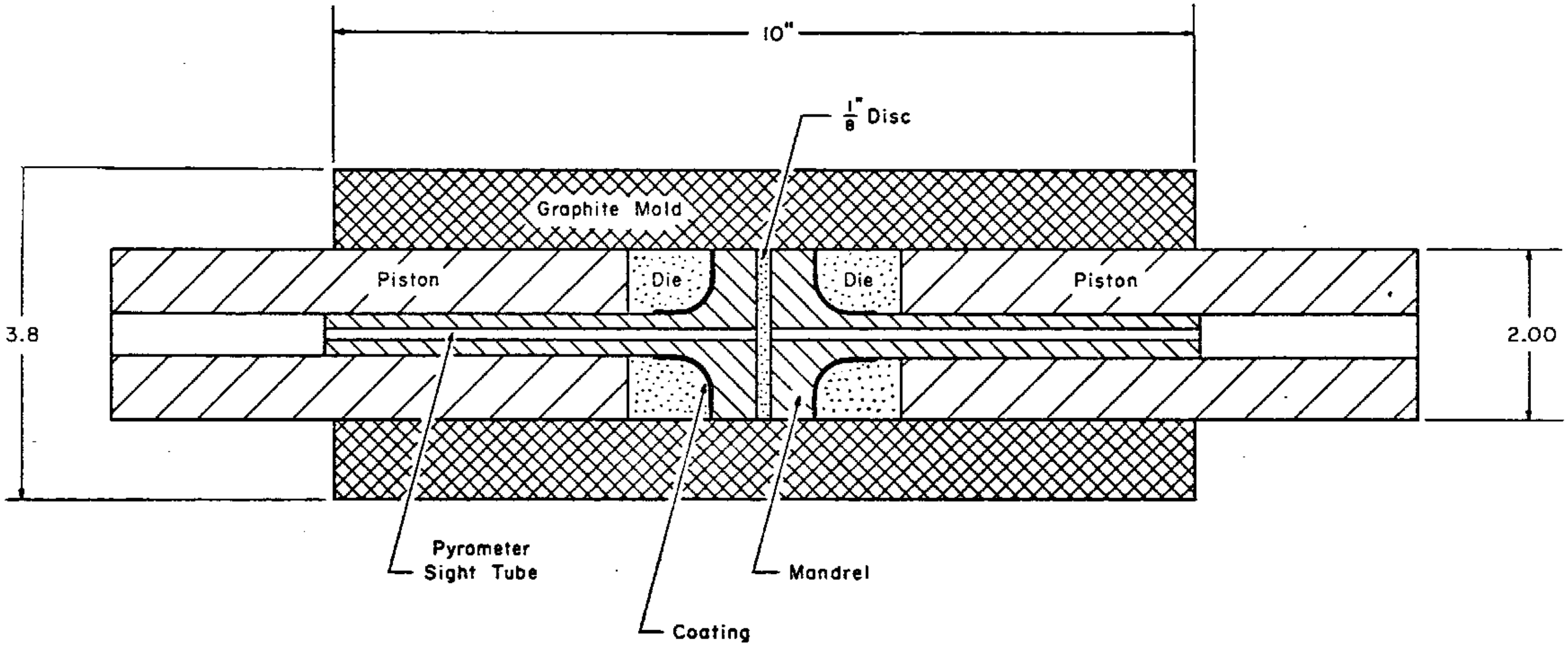

Figure 3. Hot Press Mold for Fabricating Two Coated Dies.

materials were mounted in a support on the end of the stem of the hydraulic press as shown schematically in Figure 4. After a miniature billet was heated in the tube furnace to the appropriate temperature, ($\approx 3400^\circ F$), it was expelled by a push rod into a support on the press bed. The press was actuated and the specimen was forced onto the billet at pressures of about 100 tons/in.^{2}, which was anticipated to cause plastic flow of the billet over the face of the specimen. Siliceous lubricants were added to the billet and/or the specimen. A photograph of this equipment is shown in Figure 5.

b. Extrusion Tests

For materials or die systems that appeared to be most satisfactory in the preliminary round die tests, T-shapes would be made and evaluated with billets of 4340 alloy steel and TZM alloy at $2200^\circ F$ and $3400^\circ F$, respectively, with appropriate siliceous lubricants. A new 1400-ton fast-acting press (up to 1100 inches per minute) was to be used.

5. Selection of Supporting Methods

The relative importance of support for dies depends in large measure on the brittleness of the die. Although several types of supports were under consideration and investigation, those which comprised hardened steel casings supported within the liner, as shown in Figure 6, received emphasis initially. The insert in this type relies on compressive support, either by a thermal shrink as shown in Figure 6 or by the action of a taper shown in Figure 7. Some ductility in the insert may be required for this method of support because of the slight expansion of the casing and the liner under high pressure.

Soft supports were investigated as alternatives. One type, shown in Figure 8, was to rely on the action of the billet to stress the soft support, which, in turn, was to compress the nib or insert within it. Another type, shown in Figure 9, was to depend on the approach or entry shape of the die to cause a soft material, preceding the billet, to flow not only through the die but laterally as well, thus providing approximately isostatic support on the outside, as well as within the inside of the die before the billet extruded. In this way the die was to be nearly equally stressed on the inside and out. When the metal started through the die, the softer material was to be trapped, and continued to exert pressures comparable to those pressures within the die.

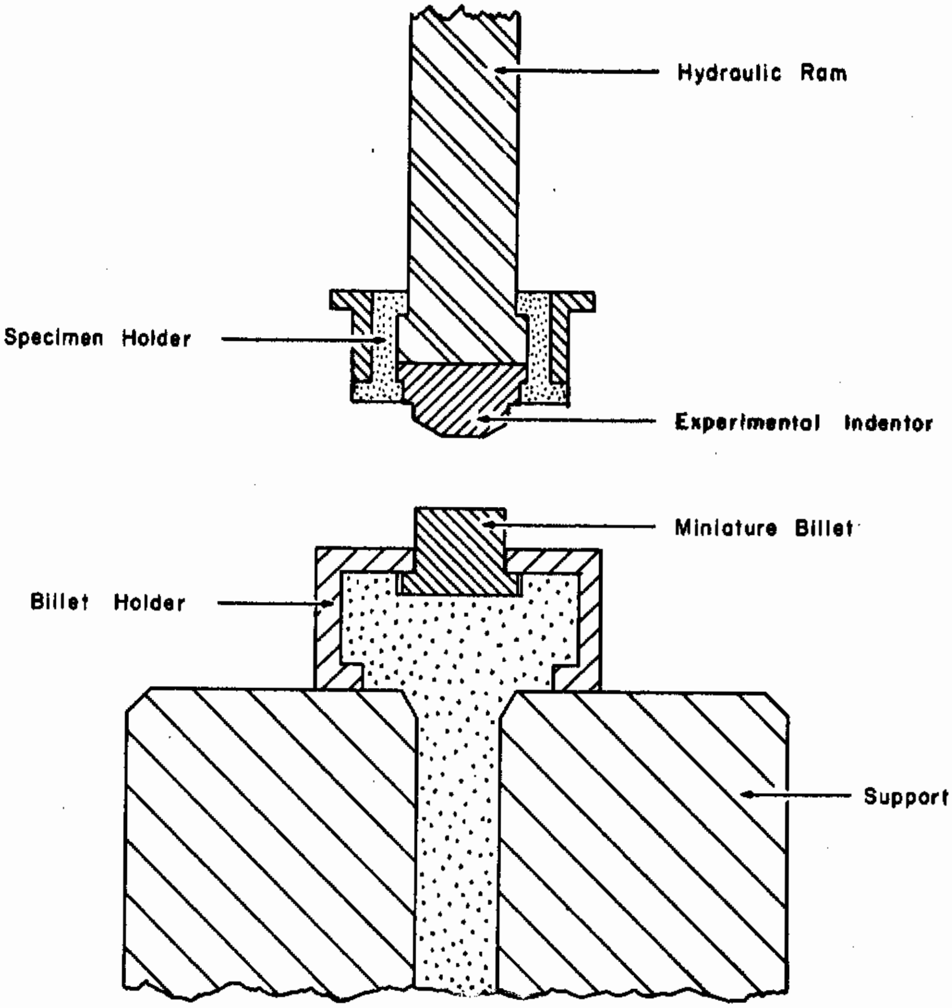

Figure 4. Diagram of Indentor Test Assembly.

Experimental indentor materials are compressed at 100 tons/in^2 into the minature billet at 3400°F.

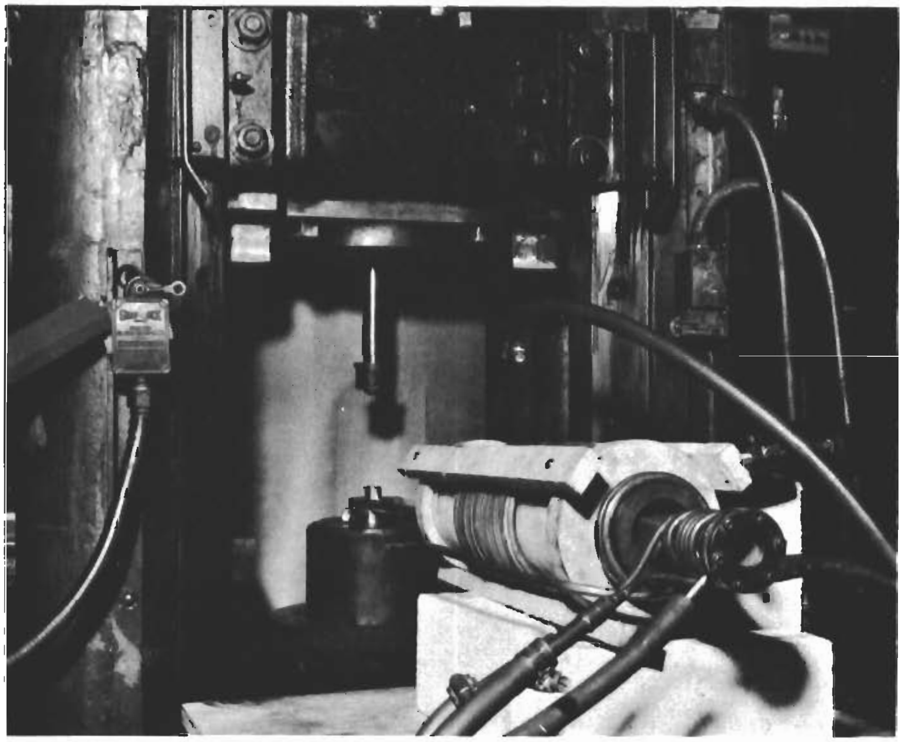

Figure 5. Photo of Indenter Test Assembly.

Billets are heated in the horizontal graphite tube furnace and compressed in the vertical hydraulic press.

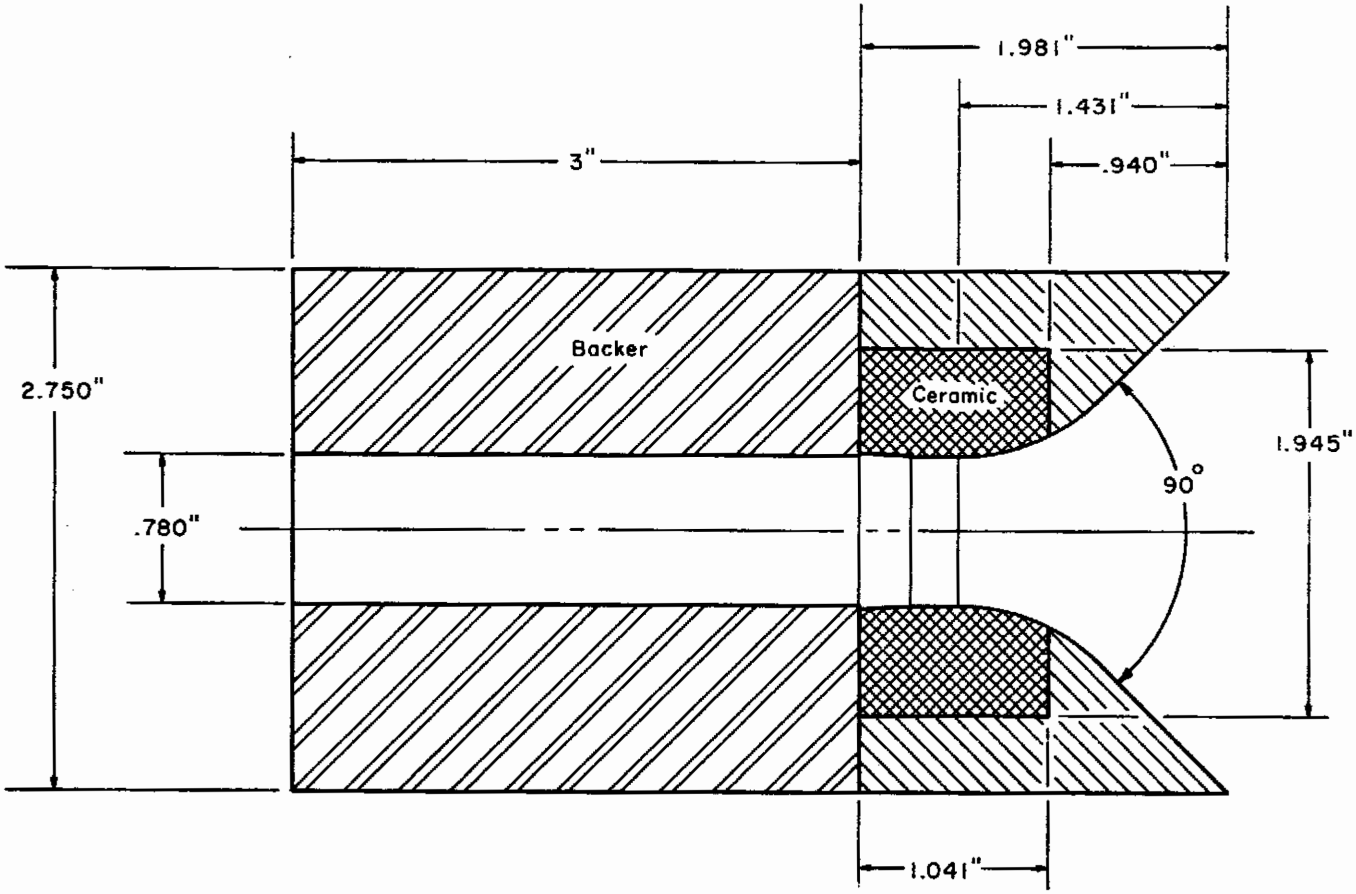

Figure 6. Support by Thermal Shrink of Hardened Steel.

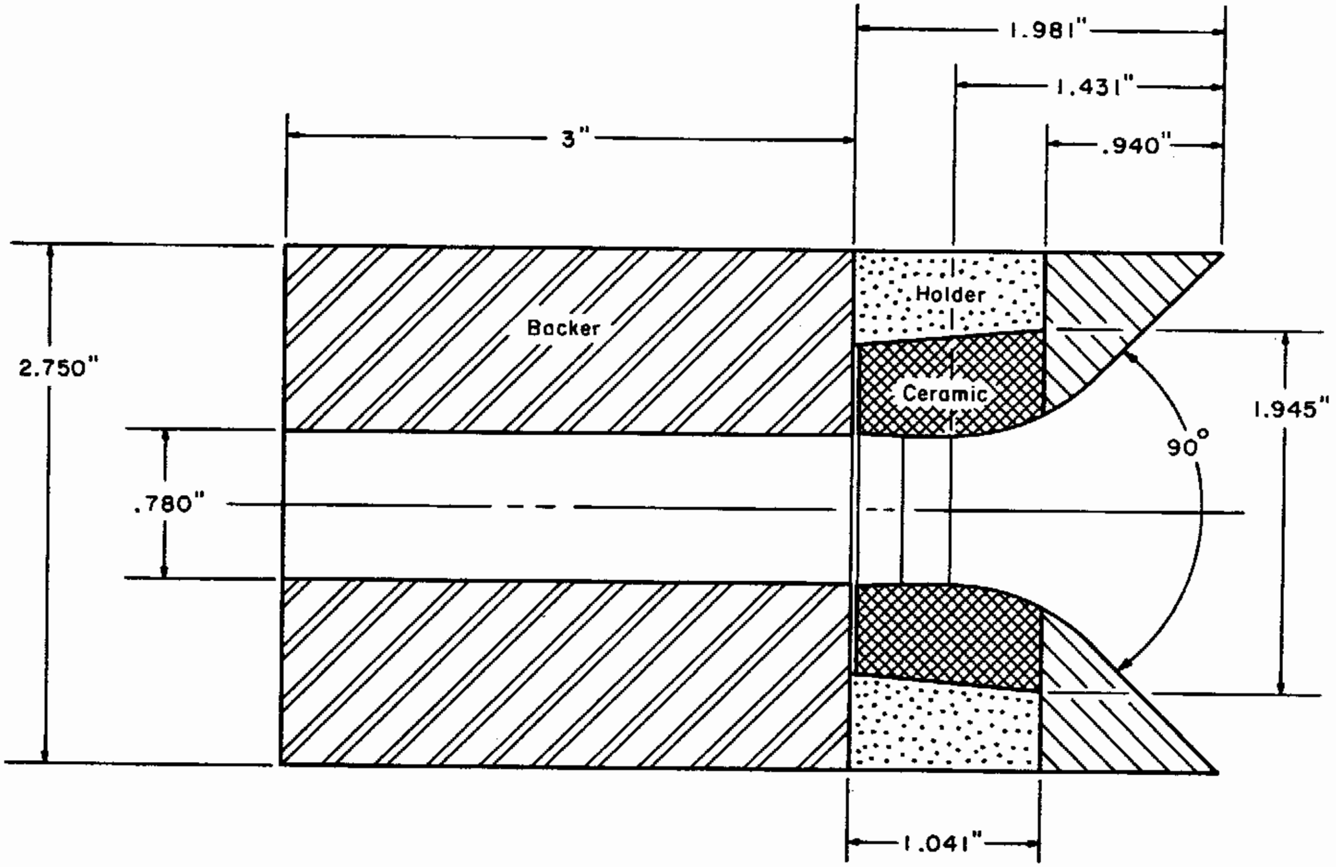

Figure 7. Support of Die Insert by Tapered Hardened Ring.

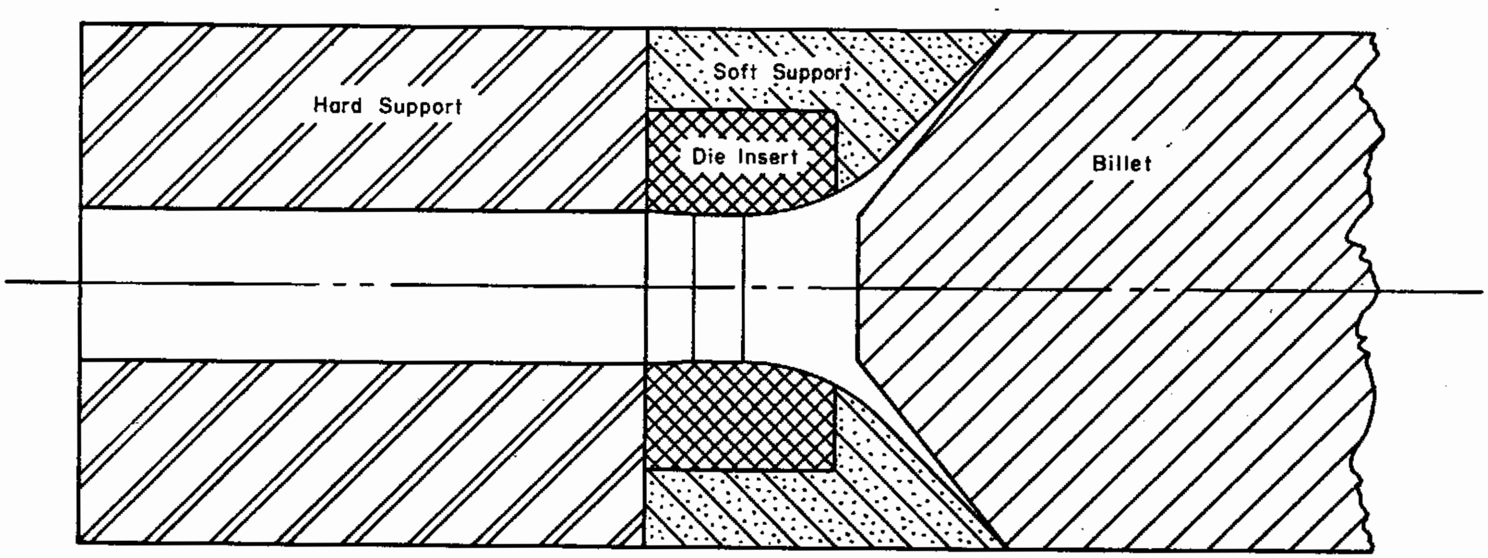

Figure 8. Support by Compression of Soft Support.

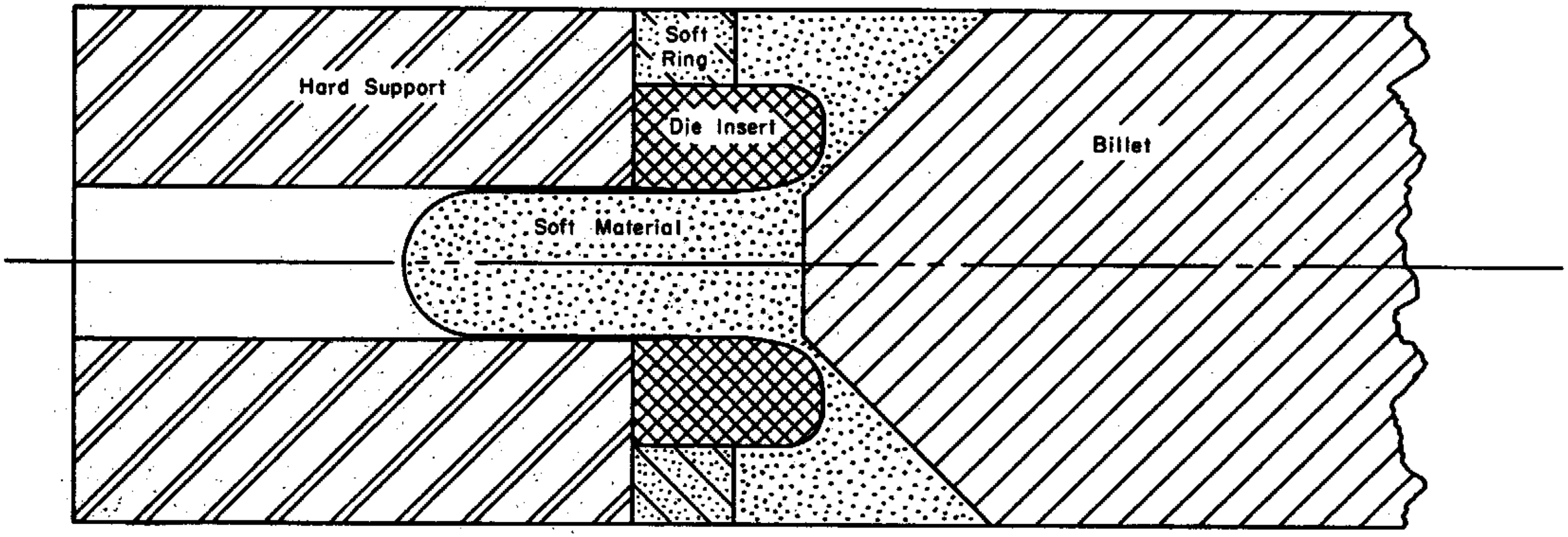

Figure 9. Support by Near-Isostatic Compression.

The force is exerted by a soft material, which has flowed both outside and inside the die, preceding the billet. Entry of the billet will trap the soft material outside the die.

B. Preliminary Experiments

The feasibility of the preliminary plans was determined in a series of experiments. These included (1) selection of specific and representative trial materials, (2) trials of fabricating specific materials, (3) trials of testing specific materials and, (4) trials of supporting specific materials. The results of these experiments led to experiments for the development of specific internal supports, and development of testing procedures.

1. Selection of Specific Trial Materials

To determine the degree of promise of various die materials systems, specific, representative materials were selected for trials. Among the metal reinforced ceramic systems, Al_2O_3 - 20 $^v/o$ W and ThO_2 - 20 $^v/o$ W were selected, because these oxides were available and no process for tungsten fibers had been developed as yet. Although oxide cermet inserts were made with tungsten powder, and not fibers, it was believed that useful information would be gained relative to the hot pressing procedure and to the performance of such cermets. If these showed promise, ultimate substitution of tungsten fibers for powder might effect an improvement. Among the intermetallic systems $MoSi_2$ was selected because of its high temperature strength.

2. Trials of Fabricating Specific Materials

The materials selected above were weighed out in duplicate and dry blended for two hours. Each composition, or two dies, was hot pressed simultaneously in graphite molds designed to produce round dies with 0.5-inch orifice in the furnaces described in Section A-3, and shown in Figures 1, 2, and 3. Pressures of 2000 psi, times of ten minutes were used for all three compositions, pressing temperatures of 3100°F were used for Al_2O_3-W and for ThO_2-W dies; 2900°F was used for the $MoSi_2$ dies. After removal from the furnace and subsequent grinding, the dies appeared to have good properties and, consequently, were prepared for testing.

3. Trial of Testing Specific Materials

The $MoSi_2$ dies were retained for experiments described in Section 4, below. The duplicate hot-pressed die inserts of ThO_2 - 20 $^v/o$ W and Al_2O_3 - 20 $^v/o$ W were shrunk with a 5-mil interference into hardened steel casings, as shown in the diagram of Figure 10. The assembled dies were subsequently shrunk into the extrusion liner. The two Al_2O_3-W dies before use are shown in Figure 11. For comparison in tests, H21 dies were made with a geometry similar to that of the assembled composite dies. The tests were made by extrusion of type 4340 steel billets at 2200°F from a 2.800-inch liner with a ram speed of 2.3 inches per second using a 600 ton extrusion press shown in Figure 12. Other experimental conditions and the results are listed in the upper half of Table 2.

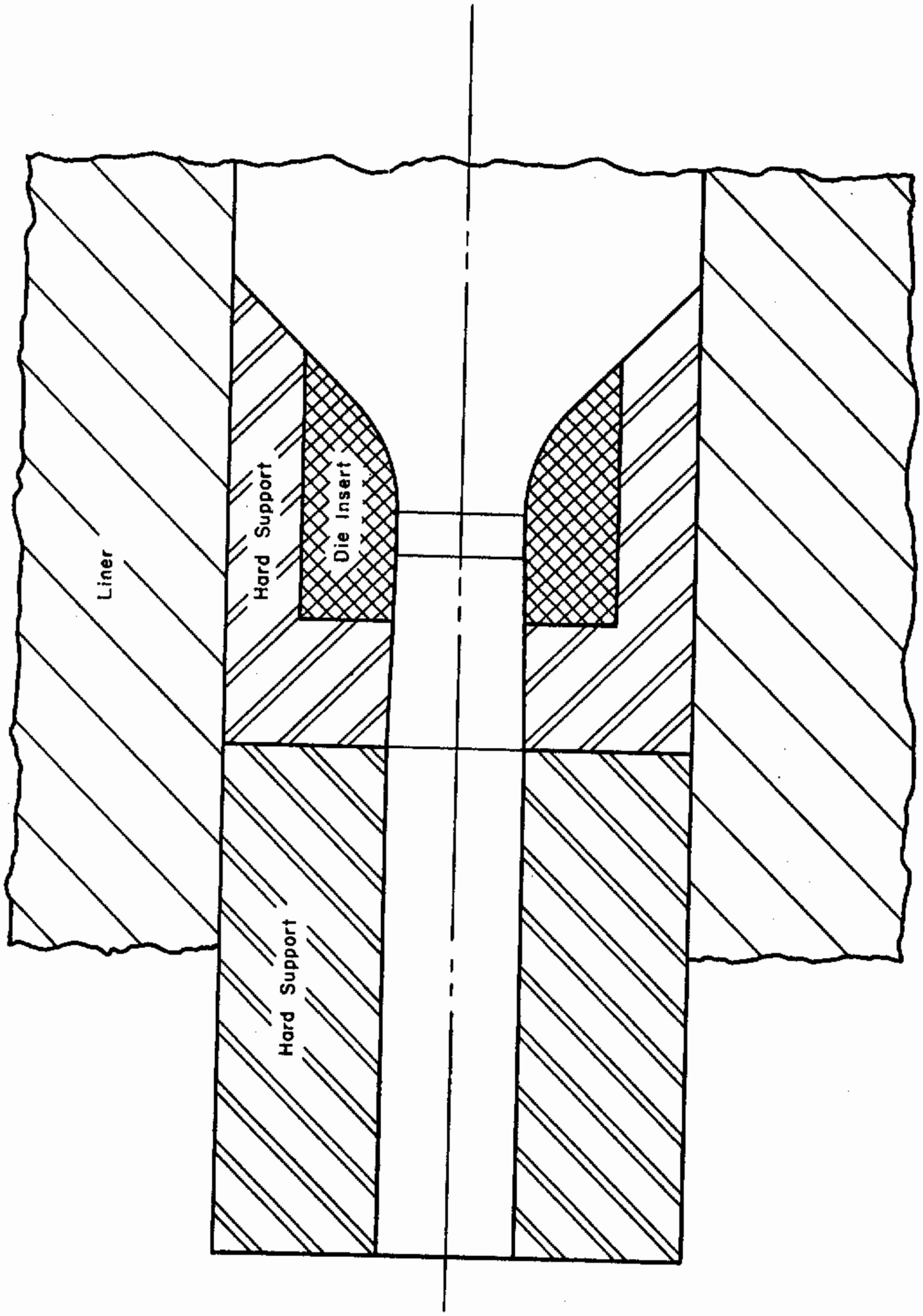

Figure 10. Die Assembly for Evaluating Trial Materials.

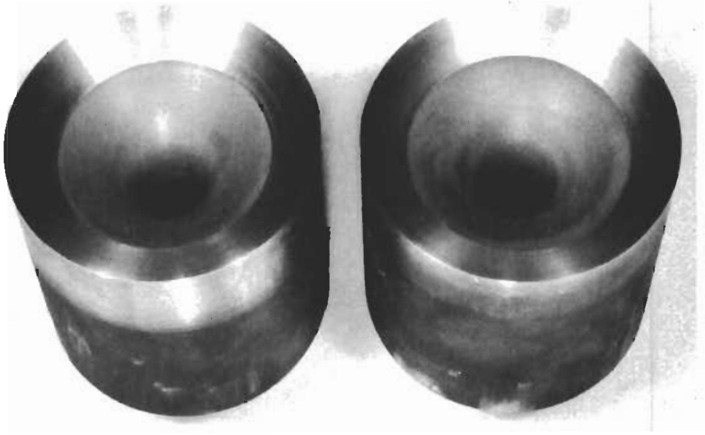

Figure 11. Trial Alumina-Tungsten Cermet before Use.

Inserts made by hot pressing Al_2O_3 - 20 $^v/o$ W powders and shrinkage into hard steel casings.

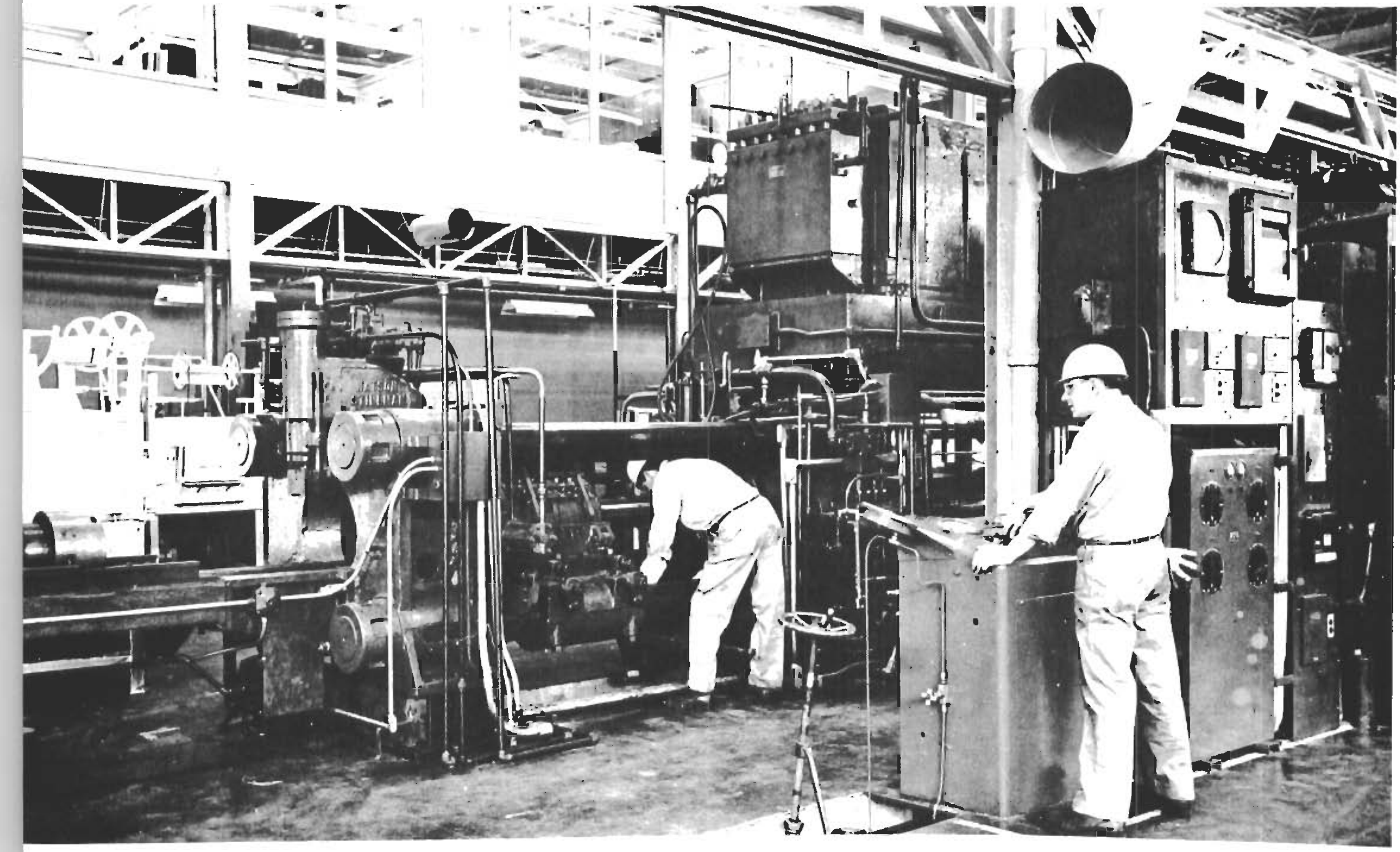

Figure 12. 1000-Ton Extrusion Press.

This press was used for the preliminary experiments described in Section B, at a ram speed of 2.3 in./sec.

TABLE 2
TRIAL MATERIALS AND SUPPORTS TESTS WITH STEEL BILLETS

Conditions				Results				
				Die		Rod		
Trial	Die Composition(1)	Temp.(2) (°F)	Support Type	Orifice Change(5) (in.)	Appearance	Diameter Variation (in.)	Length (in.)	Appearance
Materials Tests	ThO_2-W	900	Hard	-.004	Cracked	.002	132	Smooth
	ThO_2-W	900	Hard	-.006	Cracked	.002	132	Smooth
	Al_2O_3-W	900	Hard	-.005	Cracked	.001	132	Smooth
	Al_2O_3-W	900	Hard	-.001	Cracked	.004	132	Smooth
Supports Tests	$MoSi_2$	900	Hard	-.010	Cracked	.002	132	Smooth
	$MoSi_2$	900	Soft	+.117	Cracked	.004	138	Striated
	$MoSi_2$	900	Isostatic	--	Broke	.111	108	Grooved
	SiC	900	Soft	+.005	Cracked	.001	95	Smooth
	SiC	1600	Soft	+.005	Cracked	.005	95	Smooth
	Mo	1600	Soft	+.503	Closed	.489	48	Smooth
	W	1000	Soft	+.467	Closed	.074	56	Smooth
	W	900	Soft	+.235	Closed	.015	58	Smooth
Controls	Steel(4)	900	--	+.025	Washed	.006	132	Striated
	Steel(4)	900	--	-.021	Washed	.023	132	Striated

<u>Other Conditions</u>

Billet

Material - Steel
Temp. - 2200°F
Lubrication - Glass
Diameter - 2.750 in.

Die

Size - .500 in. nominal(3)
Lube - None

Extrusion

Liner Size - 2.80 in.
Ram Speed - 2.3 in/sec
Liner Lube - Necrolene

(1) Oxide composites contained 20 $^v/_o$ W

(2) Die and support

(3) Except W and SiC which were .885 and .680 in. respectively

(4) H21

(5) Minus (-) means enlargement
Plus (+) means constriction

Both steel dies exhibited some "washout", as shown by the photographs in Figure 13, but the effect was to increase the size of the opening of one die and to decrease it in the other.

The hot-pressed inserts cracked and were depressed below the surface of their casings because they either densified or were not properly seated, or because the casings changed in dimensions. There was no evidence of "washout", either from the appearance of the inserts or from the dimensions of the extrusions which were smooth and with diameter uniformity of ± .002 in. from front to rear. Dies after extrusion are shown in Figures 14 and 15. It is apparent from this series of tests that uncoated H21 steel is, as expected, an unsatisfactory die material even for extrusion of steel at the temperature and pressure conditions of the experiment. On the other hand, hot-pressed mixtures of ThO_2 or Al_2O_3 powder with tungsten powder show good resistance to erosion and might be satisfactory even under more severe conditions.

4. Trials of Supporting Specific Materials

Three methods of supporting experimental die inserts were under consideration as described in Section A-5. These are (1) hard (or conventional) supports, (2) soft supports, and (3) isostatic supports. To determine the degree of promise of these systems, the three methods were compared and evaluated in experiments described below.

Available materials were used for die inserts to obviate long waits for delivery of alternate materials. Inserts were prepared from pressings of $MoSi_2$ (Section B-1 and 2 above) which had been prepared in the laboratory, and from samples of sintered molybdenum, sintered tungsten and KT silicon carbide.* These were readily available from other programs or commercial sources. Although some of these materials were in the form of hollow cylinders, thereby pre-determining the minimum die opening, it was not believed that the size of the die was important in this series of tests. Experimental conditions and results are shown in the lower half of Table 2.

a. Hard Supports

For the evaluation of hard supports, the molybdenum disilicide ($MoSi_2$) nib was shrunk into a hardened steel casing. An attempt was made to shrink the assembled die into the extrusion liner, but the liner was worn to such a degree that the amount of support on the die was probably non-uniform and indeterminable. After extrusion of a mild steel billet the insert was cracked but did not show any evidence of "washout". The die opening was smaller by 10 mils after extrusion than before, apparently because of an upsetting action or shrink fitting or both. Figure 16 shows the die after use.

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*Product of Carborundum Company

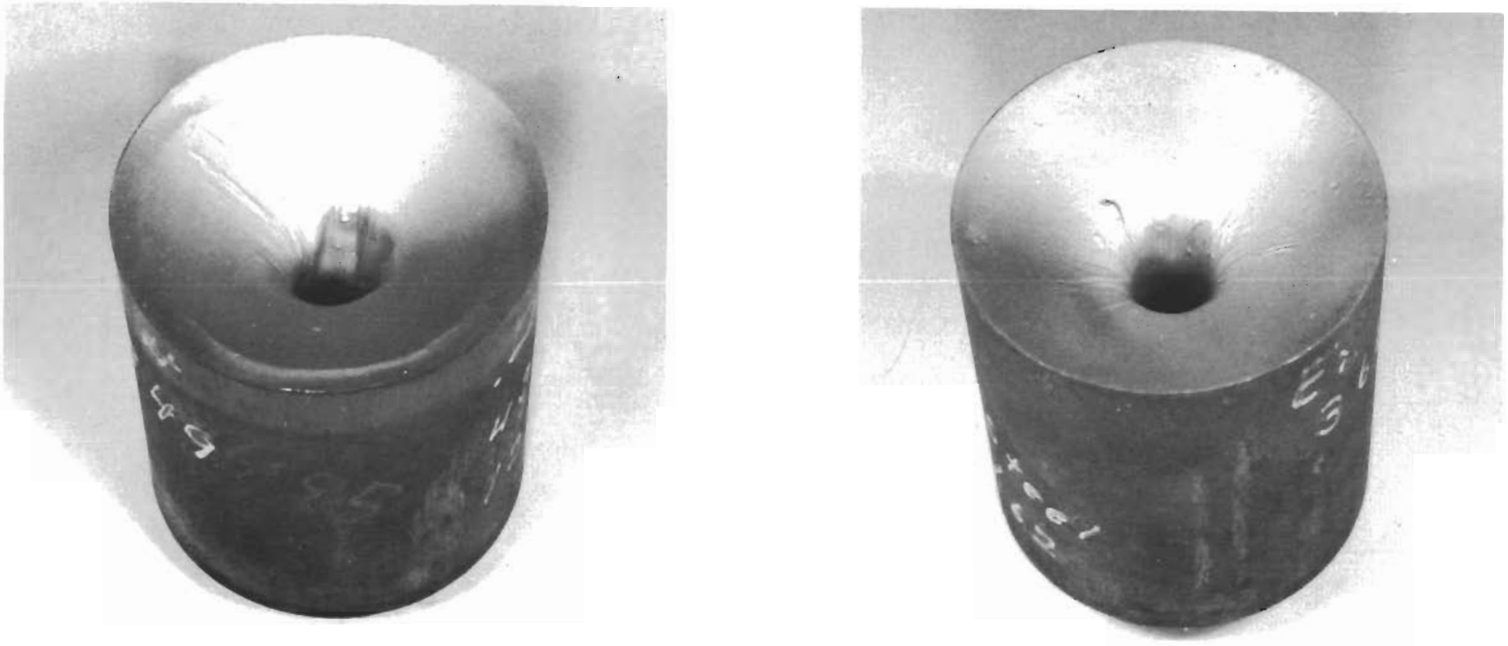

Figure 13. Steel Control Dies after Use.

These dies of H-21 steel were hardened to 52-53 R_c.

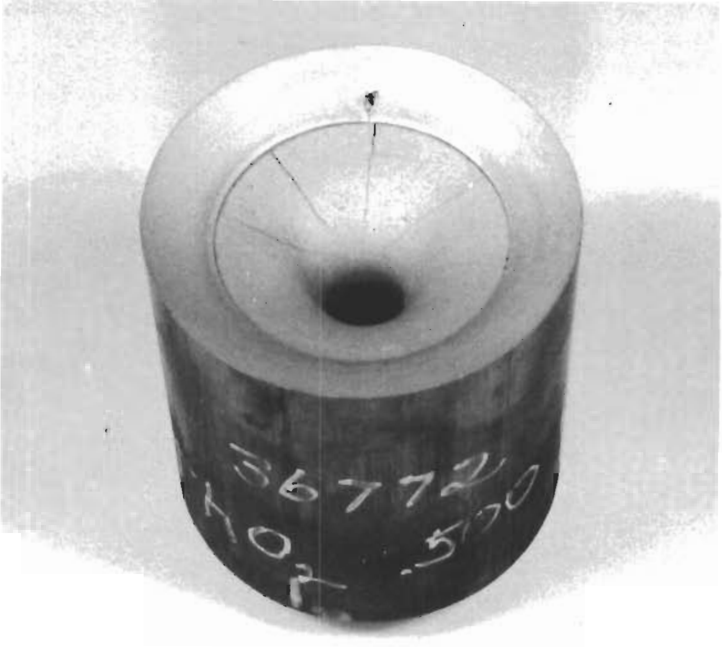

Figure 14. Trial Thoria Cermet after Use.

Insert made by hot pression ThO_2 - 20 $^{v}/o$ W powders. After one extrusion. Cleaned after one use by vapor blasting.

Figure 15. Trial Alumina Cermet after Use.

Insert made by hot pressing Al_2O_3 - 20 $^v/_o$ W powders. After one extrusion. Cleaned after use by vapor blasting.

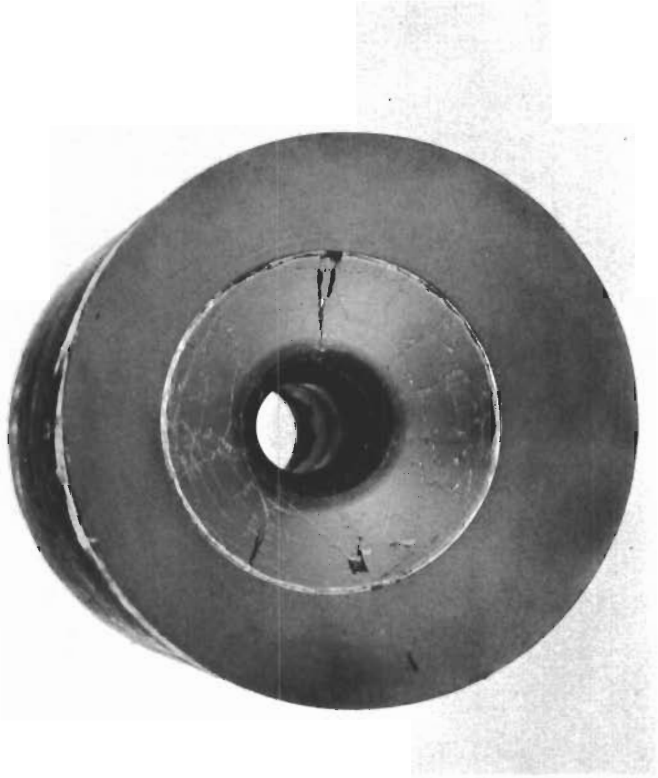

Figure 16. Trial Intermetallic after Use.

Hot pressed $MoSi_2$ supported in hard steel casing, after one extrusion. Cleaned after use by vapor blasting.

b. Soft Support

For investigation of "soft" supports, die casings of cold-rolled steel were used. By this method of support, pressure is exerted on the casings by the billet noses, whose angles are greater than those of the die approach angles; the supports, in turn, exert a hoop stress on the inserts. The die inserts and supports were assembled with a 10-mil clearance between diameters. By use of a steel "cushion" welded to the bottom of the support, the necessity for grinding the bottom of the inserts and supports was reduced or eliminated. Trials of soft supports were made with the die assemblies at 900°F and 1600°F. Stainless steel billets at 2200°F were extruded.

It appears that the inserts were placed under considerable compression since, from the data of Table 2, it is apparent that the openings were smaller after extrusion than before. The molybdenum insert in a die assembly at 1600°F extruded and blocked the opening. The openings in the tungsten inserts, one of which is shown in Figure 17, were reduced in size at both temperatures. The silicon carbide and molybdenum disilicide underwent only slight deformation before they cracked. A silicon carbide die after use is shown in Figure 18.

c. Isostatic Support

The molybdenum disilicide duplicate insert prepared in Section B-2 above, was used for the evaluation of the isostatic support technique. This technique is discussed in Section A-5 above. The isostatic medium was a cylinder of Pyrex* glass at 1400°F. Other conditions of the test are listed in Table 2. In the extrusion test, the rod was badly striated and the die collapsed when the extrusion was almost completed. It is believed that failure occurred progressively by cracking of the brittle insert until it failed catastrophically. The failure is believed to have been caused by improper alignment. Since the face of the die which bears against the die backer is not subject to isostatic pressure, attainment of a truly isostatic situation would be difficult, particularly with dies of complex shape.

5. Development of Internal Supports

From the above experiments it appears that neither soft supports nor isostatic supports are practical, since hoop stresses exerted either during extrusion or when the extrusion has been ejected are apt to destroy the die. This would be particularly important with dies of complex shapes.

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*Product of the Corning Glass Works

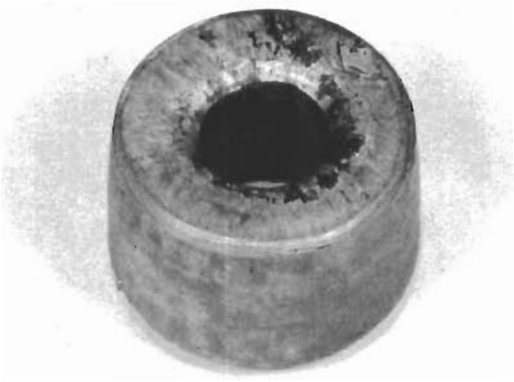

Figure 17. Tungsten Nib from Soft Support Trial.

Sintered tungsten insert after removal of soft steel casing. Shown after one extrusion. Cleaned after use by vapor blasting.

Figure 18. Silicon Carbide Nib from Soft Support Trial.

KT silicon carbide insert after removal of soft steel casing. Shown after one extrusion. Cleaned by vapor blasting.

The data also indicate that unalloyed molybdenum is too soft for a die material. Tungsten, on the other hand, flowed much less and behaved as a ductile material. This suggests that it might make a satisfactory die if strengthened by alloying or some other means to prevent flow.

The support trials of Section 5 above lead to the conclusion that a modified version of the hard support holds the greater degree of promise compared to the other two. However, a means for ensuring compression of the hard support was needed. The use of massive external supports would be expensive unless many experimental dies could be tested in a given support. This situation might prevail if it were not necessary to remove the die intact in the "as tested" condition, so that it could be accurately evaluated. Therefore, to achieve economy in tooling costs, experiments were carried out to develop a successful internal support for round dies. Results from this work generated data concerning support of external Tee dies. Magnesia-stabilized zirconia (ZrO_2-MgO), a representative brittle material, was used as the prototype nib in these die support studies.

Three types of supports were tried, as shown in Figure 19. A distinguishing feature of two of these supports (Figures 19B and 19C) is a copper ring around the casing. Since the copper ring is slightly longer than the casing, the copper is compressed by the steel cone and isostatically supports the hardened steel casing; this design is a combination of the "soft" and "hard" supports described in Section 4 above. The first (Figure 19A) has no isostatic support (copper sleeve between casing and liner) and no nib cushion (copper ring between the casing and nib), the second, (Figure 19B) has an isostatic support and no cushion, and the third (Figure 19C) has both support and cushion. Each ZrO_2 nib was shrunk into a steel casing with a 5-mil interference fit. A steel billet was extruded at a ram speed of 2.3 inches per second through each die at 2200°F and 80 tsi. The nib without isostatic support cracked severely; the others did not. In fact, their performance indicates that massive ZrO_2 is a very promising die material. Although the value of the nib cushion (copper ring adjacent to nib) is not demonstrated by these experiments, it is anticipated that a similar cushion made of aluminum can be dissolved in sodium hydroxide solution to free a used casing so that it can be re-used with a new nib. This would allow examination of the nib in the "as tested" condition.

6. Development of Testing Procedures

a. Indentor Test

The indentor test procedure described in Section A-4-a above was intended to facilitate the testing of a large number of materials in a way more economical than actual extrusion tests of dies. The equipment was designed and fabricated, and specimens were prepared and tested. Con-

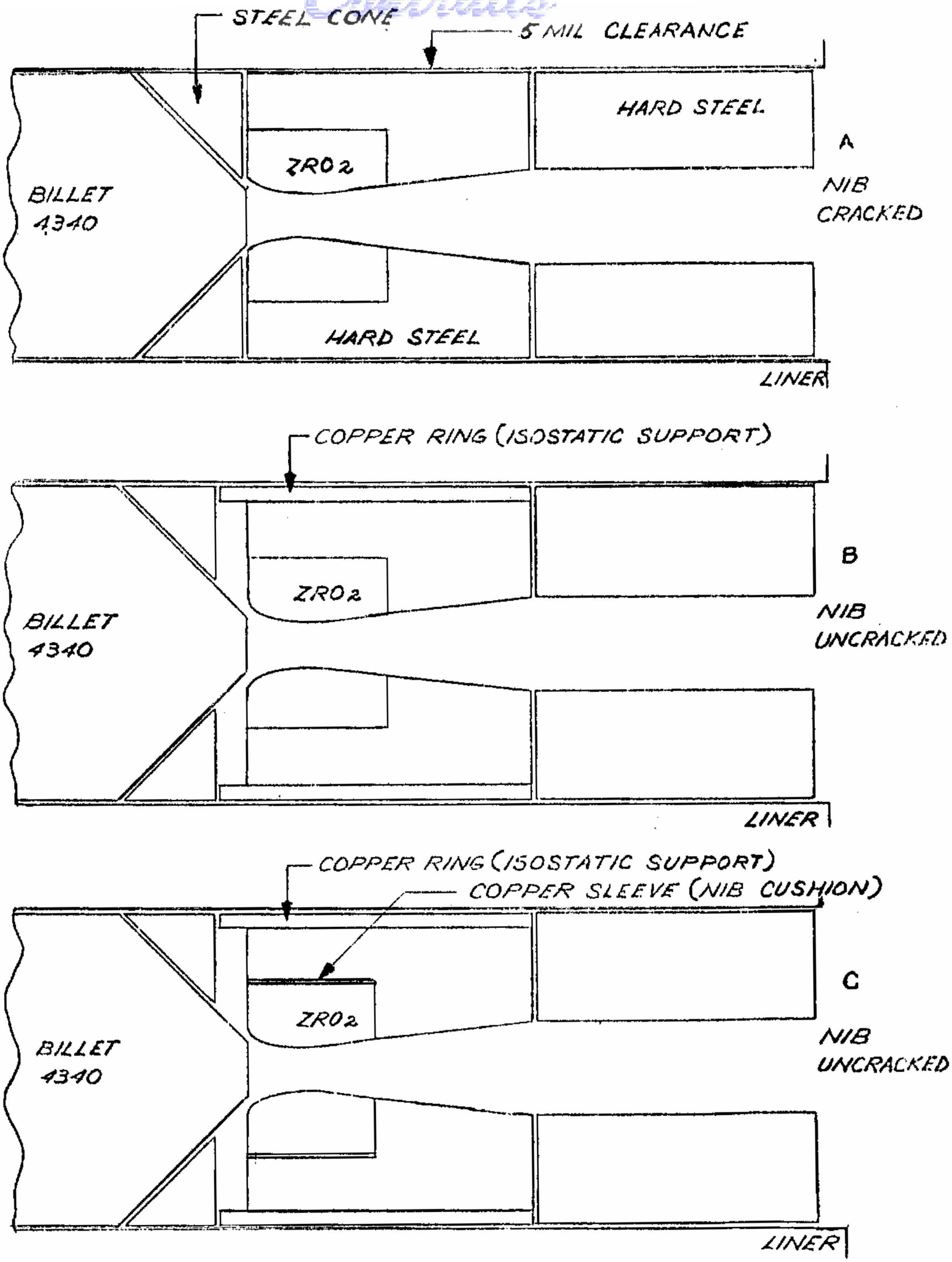

Figure 19. Designs of Three Internal Supports.

(All ribs shrunk into casings with 5-mil interference fit).

A. Supported by liner.
B. Supported by copper ring (isostatic support).
C. Supported by copper ring with copper cushion.

currently, coated dies were prepared (Section D-1, below) with the same substrate and coating as the indentors. Tests were performed to determine whether a correlation in performance existed between specimens with the geometry of an indentor and with the geometry of a die. The results showed that alignment of the indentor and billet are difficult to achieve and reproduce, a fact which prevents any useful comparison between the indentor results and the die results. The indentor test appeared to contain greater complications than the extrusion die test without giving consistent or meaningful results. Consequently, further work and use of the indentor test facility was abandoned in favor of the extrusion test only.

b. Extrusion Test

Another standardized procedure was established for evaluating experimental die materials in view of the unsatisfactory results from the indentor test. A candidate material was to be tested as an internal round die for the extrusion of 4340 steel at 2200°F at a reduction ratio of 80:1 with 8-inch long billets. The die geometry to be used is shown in Figure 20. If the material performed promisingly with steel, a TZM billet was tried with a new die with the billet heated to about 3400°F and at a reduction ratio of 22:1. Die materials successful under these conditions were to be fabricated into externally supported Tees.

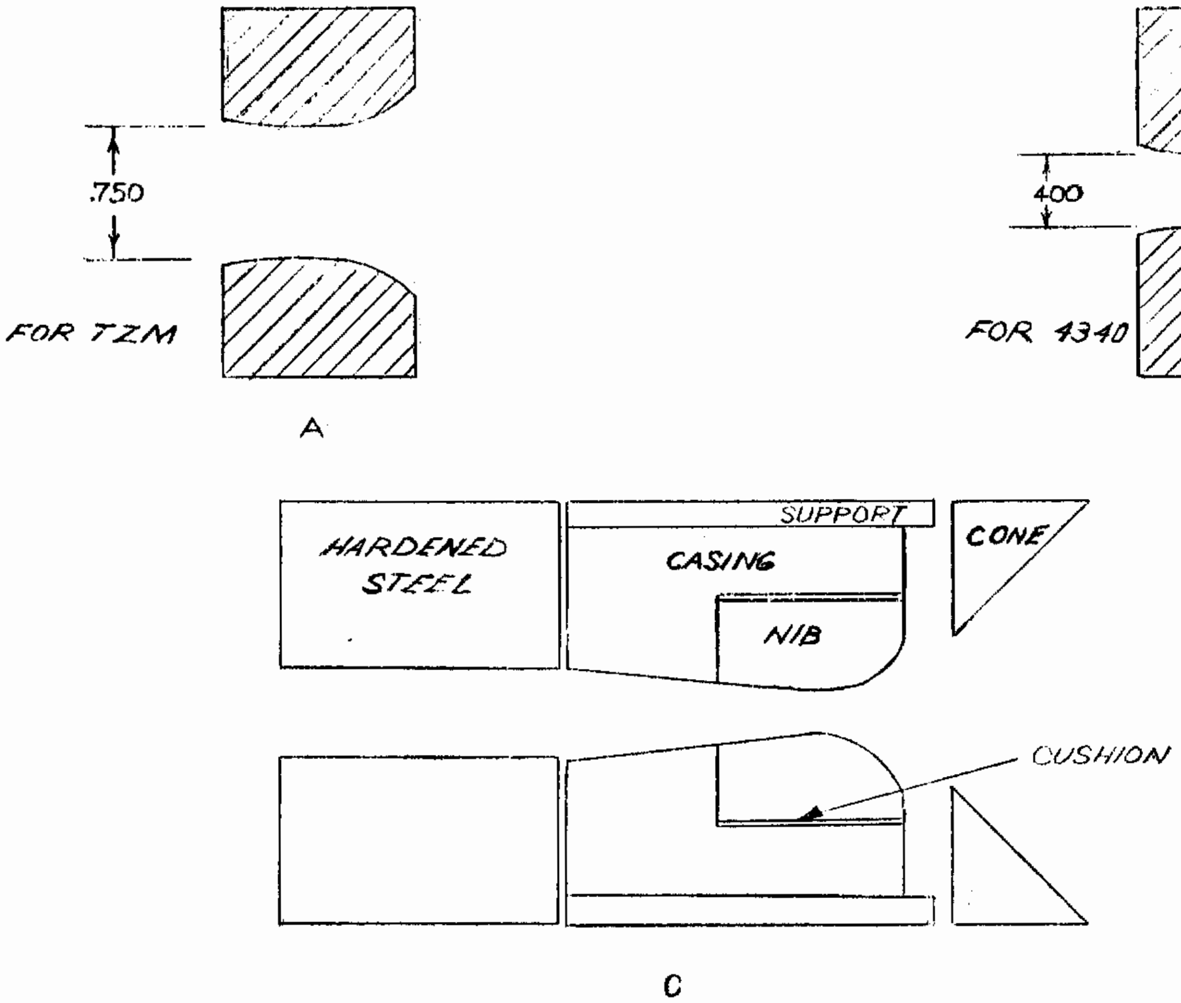

Figure 20. Design for Experimental Round Dies.

A. Geometry for TZM billets.
B. Geometry for 4340 steel billets.
C. Support design.

C. Fabrication of Die Materials

Application of established hot-pressing technology and development of new techniques were necessary for the fabrication of experimental dies made from materials systems selection in Section A-2. These techniques may be divided for discussion into (1) preparation of powdered materials, (2) preparation of tungsten fibers, (3) hot pressing of round dies, (4) hot pressing of Tee dies, and (5) preparation of metallic alloy dies.

1. Preparation of Powdered Materials

Prior to hot pressing, all constituents of experimental dies were originally obtained in the form of powder with the exception of the tungsten fibers, discussed in Section 2 below. Generally, materials were purchased as -325 mesh, and no attempt was made to commutate them further. Mixing was achieved by tumbling the weighed out powders in individual glass jars without balls. A double bladed stainless steel paddle, welded to the metal cap, divided and re-divided the material with each revolution on rolling-mill rollers. Four hour moving times were generally used.

2. Preparation of Tungsten Fibers

In order to fabricate tungsten fiber reinforced ceramics, experiments were carried out to develop a process for tungsten fiber manufacture. The selection of desirable lengths and fiber diameters was based on experiments described by Tinklepaugh,(9) who reported that the best results were obtained with .125 to .500-inch lengths in diameters less than 0.006 inches. Experiments at Nuclear Metals were performed to produce material in this size range. Tungsten wool, a form of loosely matted wire scrap from commercial wire drawing manufacturing, was used as the starting material because it is more economical and convenient than other forms of tungsten. The wool contains wire of various diameters from 1 to 10 mils. Prior to cutting, consolidation of tungsten wool by isostatic pressing, cold rolling in a steel can and cold swaging in a steel tube were attempted.

a. Isostatic Pressing in Rubber

Isostatic pressing in rubber under a unit pressure of about 90 tsi yielded a "bar" about one-inch diameter by approximately 1.5 inches long. Even with this pressure poor bonding was obtained, and the fibers did not hold together during the cutting operation subsequently attempted. Isostatic pressing was therefore abandoned.

b. Cold Swaging in Steel

The second method - cold swaging of steel-jacketed bundles of fibers - gave the straightest and most uniform product. Tungsten wool was tamped into a steel tube one-inch diameter by about 90 mil wall. The tube was cold swaged in a series of passes to about one-half inch diameter, at which point the steel casing ruptured at one spot, and swaging was discontinued. The individual tungsten wires were not reduced in cross-section, but they were packed densely by the swaging operation. The rod was then sectioned transversely into 0.125-inch lengths and the steel casing was consumed in nitric acid solution. The compacted tungsten fibers were freed by tumbling on a screen with a few steel balls. Approximately three quarters of the fibers had a length of 0.125-inch, the cut length; some were shorter, and a small amount were longer (caused by doubling over). About two pounds of fibers fabricated by the swaging method were processed completely, and an additional eight pounds have been tamped into steel tubes and were ready for swaging if the need arose. The yield of usable fibers is roughly estimated to be 50 percent - in a three-foot long rod; approximately six inches at each end are not compacted sufficiently, and the remaining losses occur in the cutting operation. Higher yields can probably be obtained by swaging longer rods. A photograph of typical swaged fibers is given in Figure 21.

c. Cold Rolling in Steel

To consolidate the tungsten wool by canned rolling, the "picture frame" method was used. An open cover "box" was fabricated by welding approximately one-inch high strips of steel around the periphery of a steel sheet, 4 inches by 8 inches by 0.125-inch thick. The tungsten wool was arranged carefully by hand in the box so that the orientation of the fibers favored the rolling direction. A cover plate was welded to the assembly, with a small hole left unwelded to permit air to escape during rolling. Unidirectional rolling was carried out at room temperature until the can ruptured. The total thickness of the composite at this point was approximately 0.30-inch, of which about 0.20-inch was tungsten. The ruptured steel covers were removed mechanically, and the resulting compacted tungsten wool was found to consist of densely matted lengths of kinked wire, which were friable and readily separable by screening. Approximately three quarters of the fibers were less than one-half inch long, and some were as short as 0.005-inch. Approximately five pounds of usable fibers were obtained by this method.

An alternative rolling method was developed by which the amount of fabricated fibers per "box" is substantially increased. This revised process, in which the yield is increased by a factor of ten, involved precompacting "pellets" about 1.5-inch in diameter and 1-inch high using pressures of about 50 tsi. The "pellets" were loaded loosely in a square steel can with 0.062-inch wall, about 6 inches square and about 2 inches high. Rolling was carried out until the can has been reduced to a thickness of about 0.125-inch. The can, which had usually split open, was removed, and the fibers are found to be kinky and in random lengths between about 0.02 and 0.40-inch long. Apparently the shearing in rolling tears the compacts apart and in so doing cuts the wire on itself. After slight screening the wire is readily mixed with -325 mesh powders.

Figure 21. Tungsten Fibers Prepared by Swaging.

The tungsten wool was loaded into a steel tube and swaged at room temperature until no further densification was observed. The rod was sectioned into short lengths and the steel shroud was removed in dilute nitric acid solution.

3. Hot Pressing Round Dies

Prior to their fabrication as Tee dies, a series of selected materials was fabricated as round dies, in the hot press equipment described in Section A-3, page 14. In general, two sizes were fabricated in the hot press simultaneously using a mold design similar to that of Figure 3. The first was with a 0.400-inch orifice for testing with steel billets and the second was with a 0.750-inch orifice for testing with TZM molybdenum alloy billets. The design of the nibs is shown in Figure 22.

a. Dies from Powders without Fibers

Preparation of powders before pressing is described in Section C-1 above. The conditions of pressing are shown in Table 3. Generally, pressures of 2,000 psi, and pressing times of ten minutes in graphite molds at various holding temperatures were used. Compositions were primarily oxide bonded borides, carbides, and nitrides and tungsten bonded nitrides and silicates. In a total of fifty-one hot-pressed nibs, twenty-three underwent subsequent grinding and finishing for testing.

b. Dies from Powder with Fibers

Three trial routes were selected for the fabrication of metal fiber reinforced ceramic composites which could be densified by hot-pressing: (1) dry blending tungsten fibers and ceramic powder (tungsten fiber development is described in Section C-2 above), (2) infiltrating matted tungsten wire felt by ceramic slip, (3) electrophoretically depositing a ceramic coating on a randomly tangled, continuous length of tungsten wire. The first route was attempted by Nuclear Metals; the other two were performed commercially by other laboratories. Two dies were hot pressed from each type of composite by Nuclear Metals. After pressing, they were sectioned and inspected.

The die or nib made by blending tungsten fibers and zircon powder (as described in Section A-1) and hot-pressing at 2900°F was successful. A photograph of the nib is shown in Figure 23. The tungsten appears to be well distributed and randomly oriented. Metallographic examination revealed no microcracking of the ceramic around the tungsten wire. A minute amount of contamination of the tungsten is visible, primarily at the exterior of the specimen. This may represent reaction of the tungsten with the atmosphere in the graphite hot-pressing furnace which, at low temperature, may contain some free oxygen.

The die made by infiltration was less successful since the tungsten fiber was not uniformly distributed, and more porosity was present. The die made by electrophoresis was less successful because the ceramic material (Si_3N_4) was not present in the interior of the die or consolidated tungsten wire. As a result of these experiments, all metal fiber reinforced composites were made by the first route: dry blending short lengths of fibers with ceramic powder and then hot-pressing.

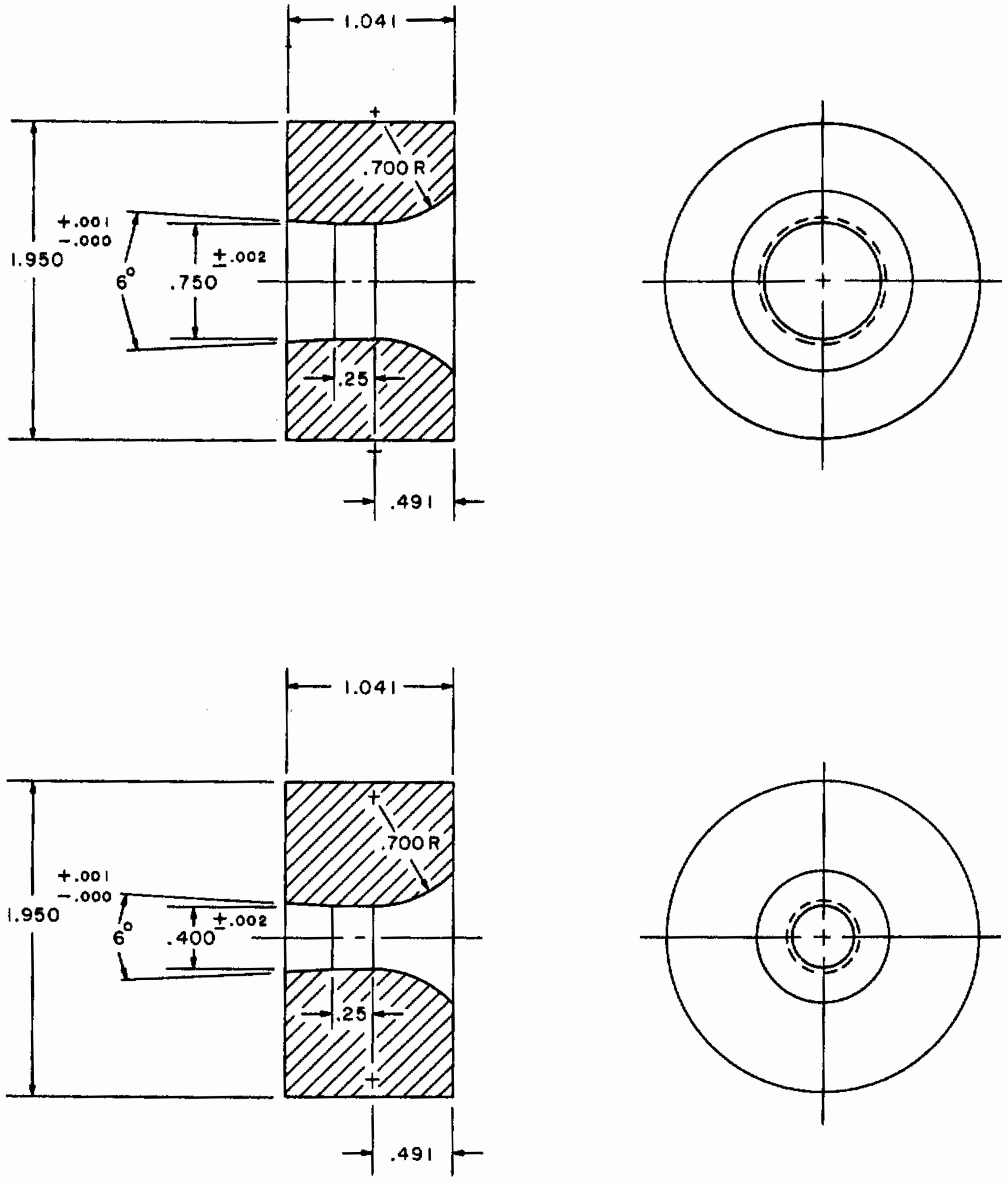

Figure 22. Design of Round Dies.

Pressings with .400 in. orifice were for tests with steel billets; those with .750 in. orifice were for TZM billets.

TABLE 3

FABRICATION OF HOT PRESSED ROUND DIES (1)

Base	Addition ($^v/o$) (2)	Temp. (^oF)	TD (%)	Result (3)
Al_2O_3	$20W_p$	3100	92	S D T
	-	2900	-	P
	$20W_p$	3000	95	S D T
ThO_2	$20W_p$	3100	-	U D T
	$20W_p$	3100	93	U D T
	$67W_p$	2750	-	S D
	$67W_p$	2750	96	S D
B_4C	-	3700	-	M
	$13W-12Al_2O_3$	2250	-	M
WC	20Al2O3	3400	-	S P
	$20W_p$	3650	84	S P
SiC	$20W_p$	3300	-	M
$MoSi_2$	-	2900	99	S D T
	-	2900	98	S D T
	20Al2O3	3200	98	S D T
	$20ZrO_2$	3200	94	C D T
	$20ThO_2$	3200	93	S D T
	$20W_p$	3450	-	M
TiC	$20W_p$	3450	-	S P
	13W-12Al2O3	3300	75	S P
	$20Al_2O_3$	3400	90	S P
	$20ZrO_2$	3500	90	S P
	$20ThO_2$	3550	91	S P
TiB_2	$20Al_2O_3$	3550	99	S D T
	$20ZrO_2$	3550	97	S D T
	$20ThO_2$	3600	99	S D T

Base	Addition ($^v/o$)	Temp. (^oF)	TD (%)	Result (3)
ZrC	$20W_p$	3600	-	S P
	$20Al_2O_3$	3450	86	S P
	ZrO2	3500	77	S P
	ThO_2	3500	85	S P
	$10MoSi_2$	3630	91	S P
	$2Al_2O_3$	3900	92	S D
	2TiO	3000	-	S P
ZrB_2	-	3300	-	S P
	$20W_p$	3550	-	S P
	20Al2O3	3550	99	S D T
	$20ZrO_2$	3540	-	C D
	$20ThO_2$	3630	-	C D
Si_3N_4	$20W_p$	2820	-	S P
	$20W_f(4)$	3000	-	U P
	$20W_f(4)$	3200	-	U P
	$20W_p$	3000	-	S D
	5MgO	3360	98	S D T
	$5MgO-20W_p$	3000	95	S D T
$ZrSiO_4$	-	2750	-	S P
	$20W_p$	2750	93	S D T
	$20W_f(5)$	2750	-	U P
	$20W_f(6)$	2900	94	S D T
	$20W_f(5)$	2900	-	U P
	$20W_f(5)$	2900		
AlB12	-	3700	-	C D

(1) Two dies of each composition 2.0 in. in dia. were pressed at the indicated temperature for ten minutes at 2000 psi in graphite molds. Lengths of pressings varied between 1.00 and 1.50 in, and generally the orifices were .40 and .75 in.

(2) W_f = W fibers W_p = W powder

(3) S = Sound P = Porous C = Cracked U = Unhomogeneous
D = Dense M = Melt T = Tested

(4) Ceramic electrophoretically deposited on wire

(5) Slip infiltrated

(6) Powder blended

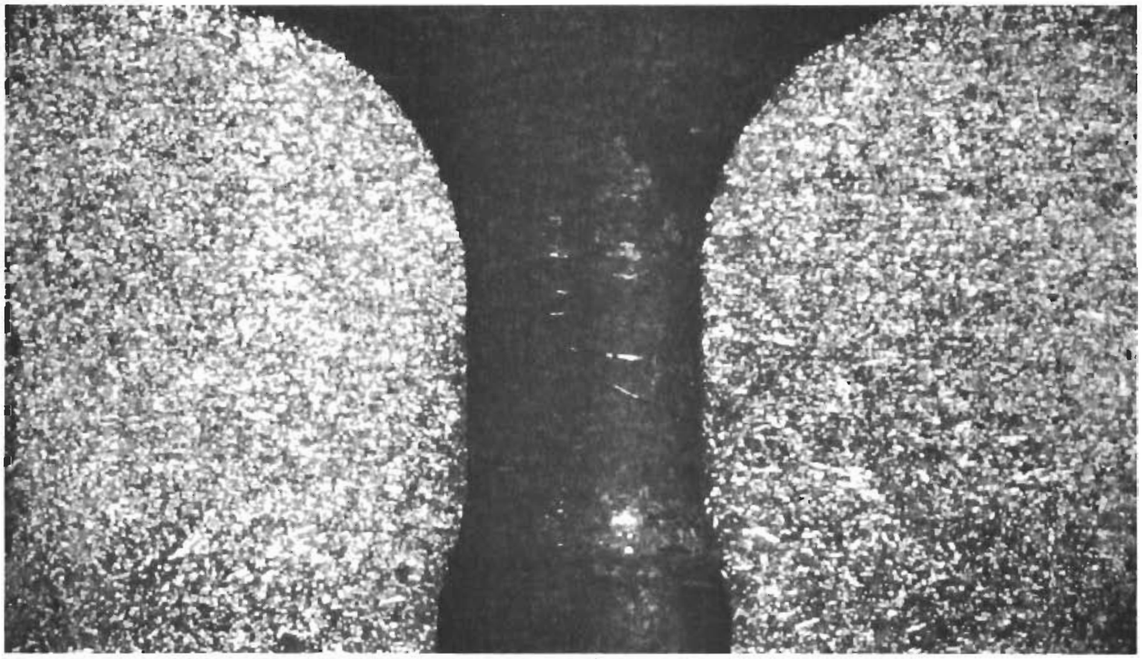

Figure 23. Sectioned W Fiber - Reinforced $ZrSiO_4$ Nib. Magnification: about 3 times.

Tungsten fibers and zircon powder were dry blended and hot-pressed in a graphite mold at 2900°F. The graphite mandrel of the mold was machined out. (This produced the score marks on the internal surfaces of the die). The die was then cut in half with an SiC wheel, and shown above in cross section.

4. Hot-Pressing Tee Dies

In order to fabricate dies with the specified 0.250 inch internal Tee orifice, various mold designs were investigated. The finished 0.250 inch Tee die was to conform to the design shown in Figure 24.

In the first trial of mold design, an ATJ grade graphite* hot-pressing mandrel was made by replicating forms built up from steel and "Plastic Steel"**. Two such master forms are shown in Figure 25. Then a preliminary pressing of a Tee die, made of ThO_2-66 ^{v}/o W, was fabricated by hot-pressing at a temperature of 3200°F and a pressure of approximately one tsi in a graphite mold. A Tee with complex entry shape and good surface in the as-pressed condition was obtained, as shown in Figure 26. No relief of the die land was attempted in this first pressing. A steel casing with 5-mil interference fit was successfully shrunk around the nib without causing cracking of the nib. Based on this trial it was concluded that the hot-pressing technique could be used to fabricate the ceramic based Tee dies desired. Techniques for making tungsten fiber reinforced ceramic composites are discussed in Section C-3-b.

In the second trials the mold design was modified so that the mandrels would produce the desired entry radius, land, and relief angle in both the 0.250 and 0.063 inch thick dies. The mandrel design for the 0.063 inch die is shown in Figure 27.

In general, with this mold design, 0.250 inch Tees were successfully fabricated of the low thermal expansion composites - the Si_3N_4 and $ZrSiO_4$ based compositions. However, an attempt to hot-press the nitride composition with the 0.063 inch Tee was unsuccessful because the mandrel broke during pressing because of poor alignment or uneven powder fill, and an improper internal configuration was formed as shown in Figure 28. Successful pressings of zircon and silicon nitride-tungsten powder composites with and without tungsten fiber reinforcement are shown in Figure 29 and Figure 30, respectively. The silicate-based materials, in general, were pressed to reasonable density without difficulty. However, the nitride-based materials, without additions of tungsten powder but with tungsten fibers, were found to possess zones of low as well as high density. Composites of Si_3N_4-5^{v}/o MgO tungsten powder appeared to be uniformly dense. (MgO was added as a sintering aid for Si_3N_4.) Apparently the tungsten powder also aided in the densification of Si_3N_4. Consequently, 20 volume percent tungsten powder was added to the Si_3N_4-MgO-W fiber composites. This mixture produced a hard, dense material, as shown in Figure 30A. This result led to the improvement of the $SiZrO_4$ - 20^{v}/o W fiber composite by the addition of 20 volume percent tungsten powder shown in Figure 30B.

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*Made by National Carbon

**Made by Devcon

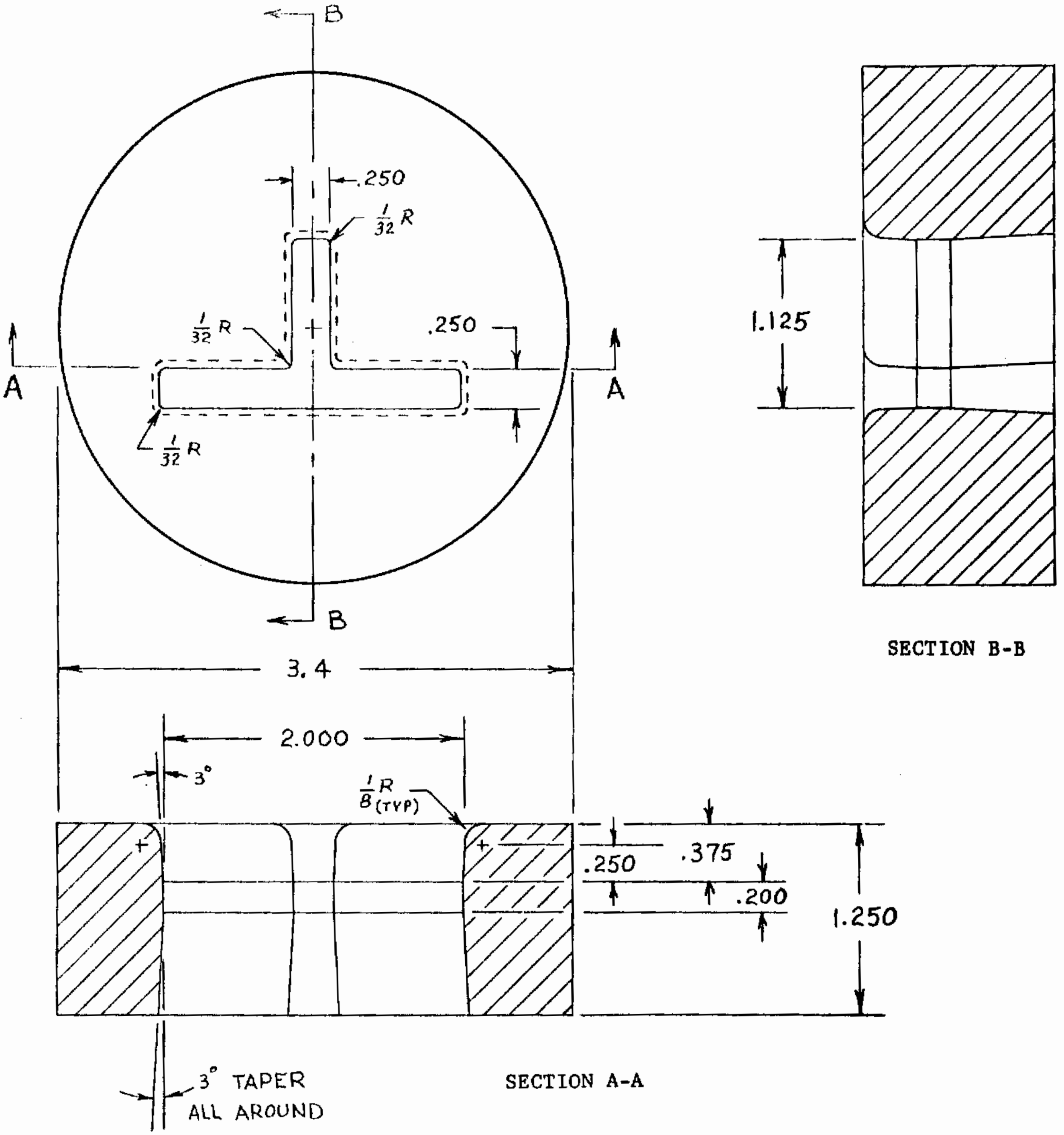
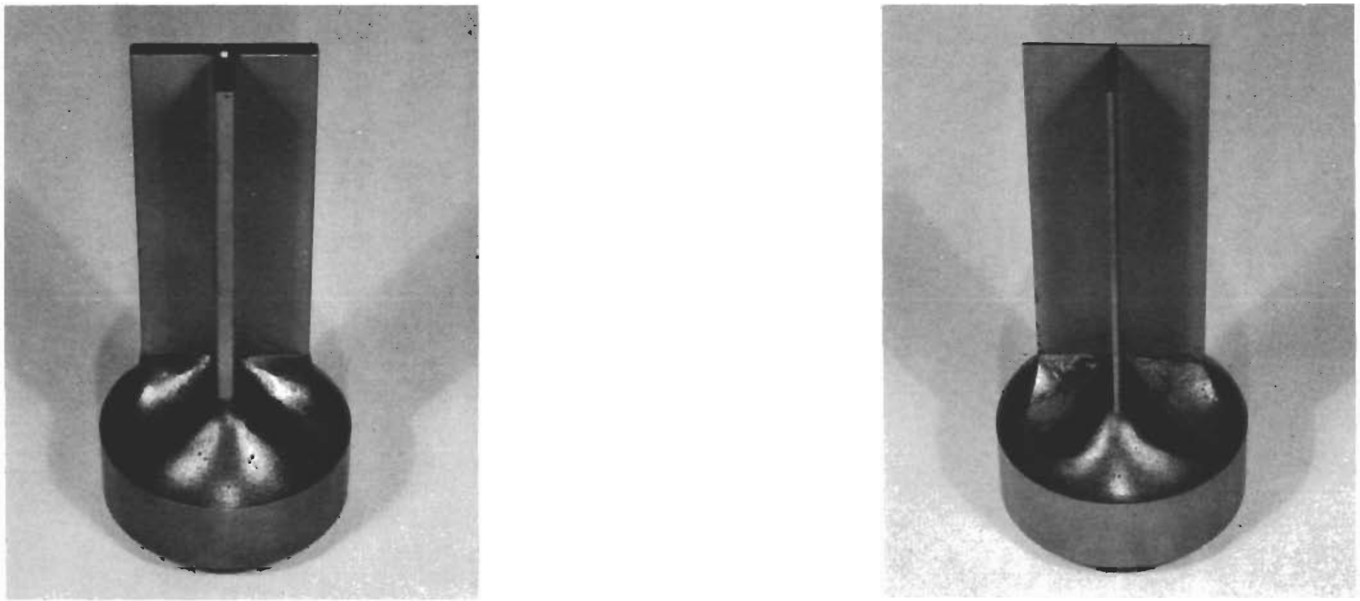


Figure 24. Design of Tee Nibs.

The relief in the nib in some cases was prepared manually and may not conform exactly to this design. The nibs made by NMI have a 3.0-inch rather than a 3.4-inch diameter.



For .250 in. Thick Tee

For .062 in. Tee

Figure 25. Forms for replicating graphite mandrels.

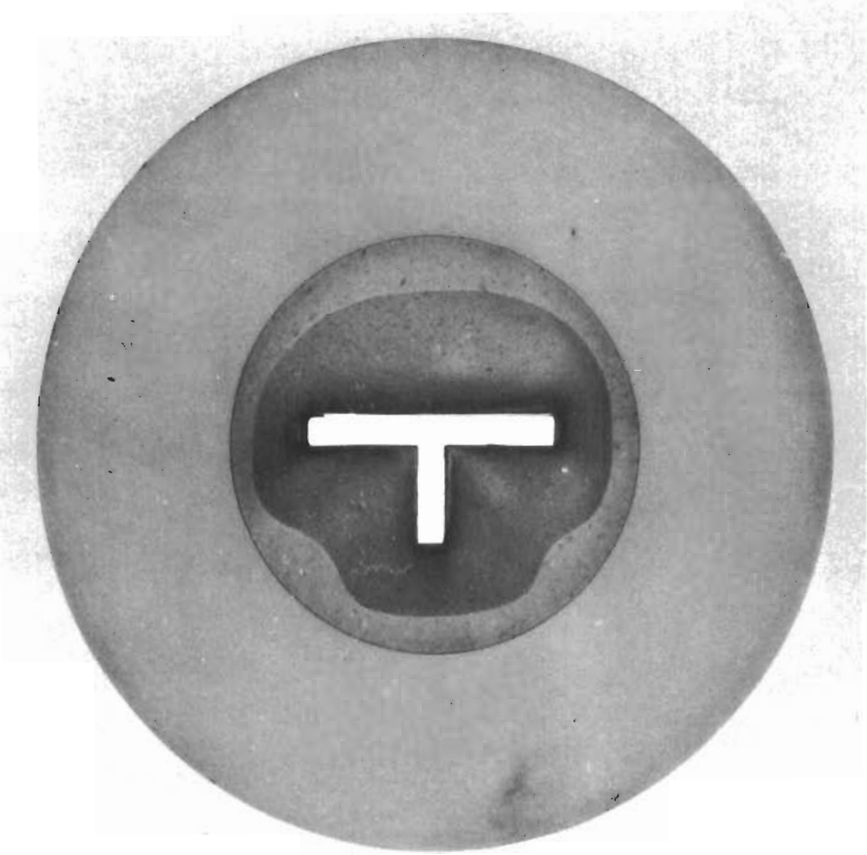
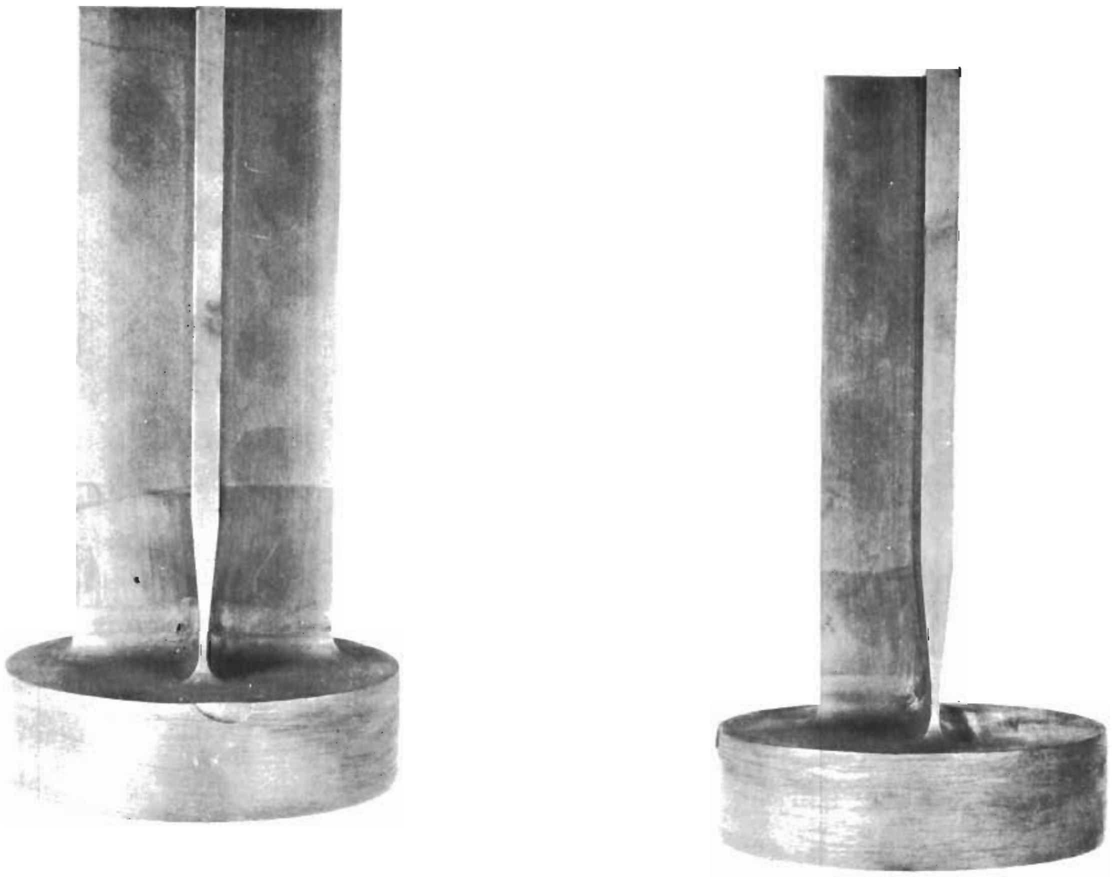


Figure 26. Hot Pressed ThO_2 - 66 $^v/o$ W Tee Nib.

ThO_2 - W was dry blended, and then hot pressed at 3200°F and one tsi for ten minutes in a preshaped ATJ grade graphite mold. After grinding the nib was shrunk without failure into a steel casing. (The spots on the nib are due to poor mixing which occurred because of agglomeration of the ThO_2).

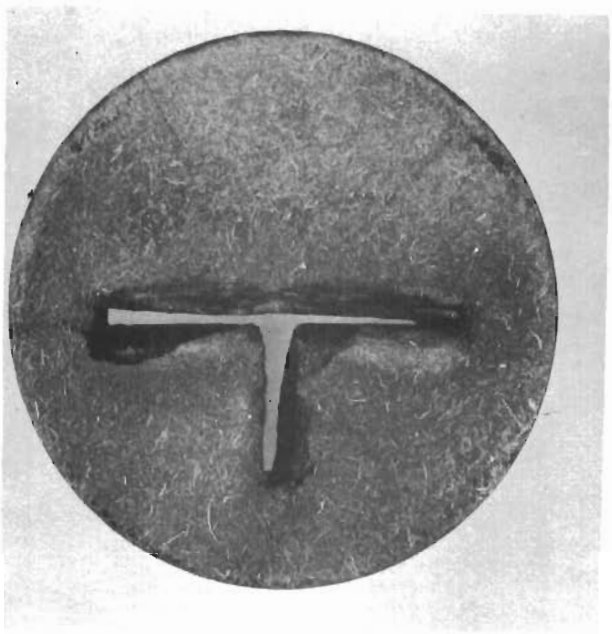


(a) View parallel with stem

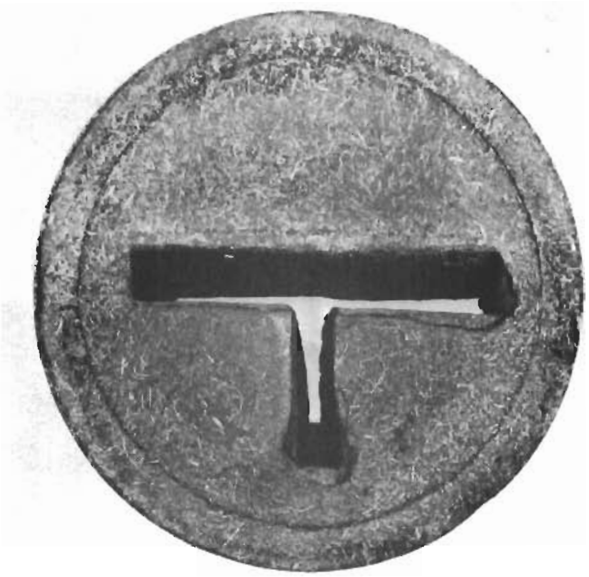
(b) View parallel with flange

Figure 27. Mandrel for Pressing .062-inch Tee Dies. (About 2/3 actual size).

The graphite mandrel was machined so that its internal shape conformed to that desired for the finished die. This mandrel fits into the assembly shown in Figure 31.



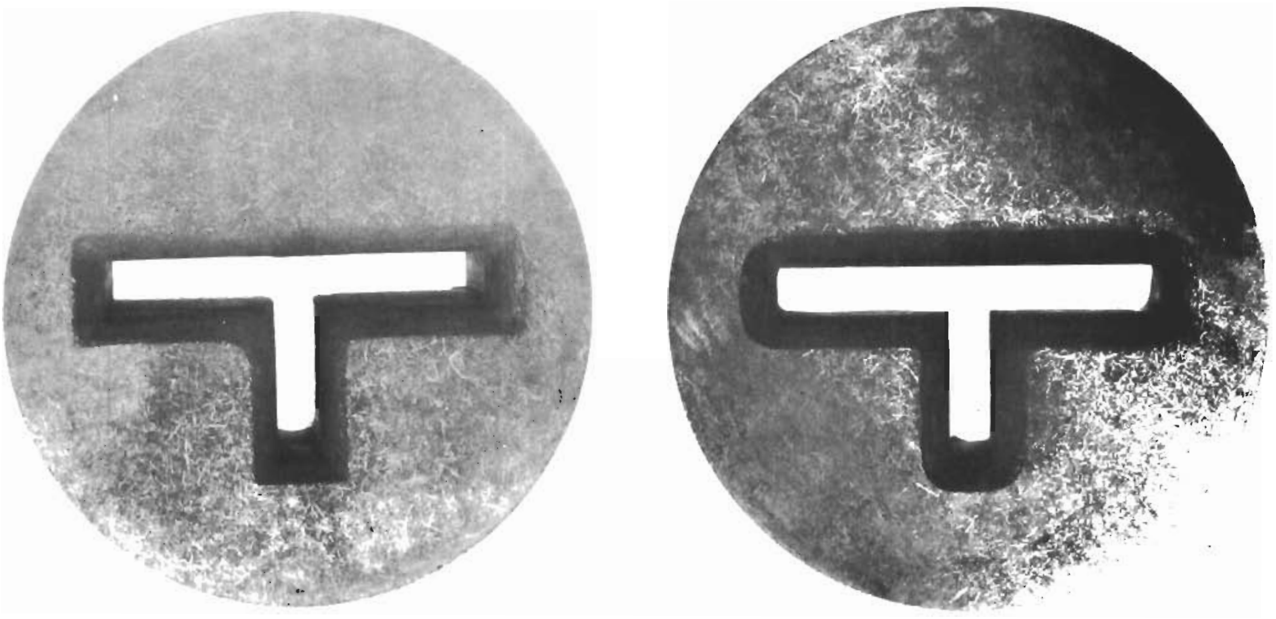
(a) Entry face



(b) Exit face

Figure 28. Unsuccessful Pressing of .062-inch Tee Die. (About actual size).

The thin section of the mandrel broke during pressing so that a die with undesired internal shape was produced. (The material is Si_3N_4 - 20 $^v/o$ W powder - 20 $^v/o$ W fibers. The conditions of hot pressing were 2000 psi, 3000°F for 20 minutes).



(a)

About actual size

Si_3N_4 - 50 $^{v}/o$ MgO - 20 $^{v}/o$ W powder - 20 $^{v}/o$ W fibers

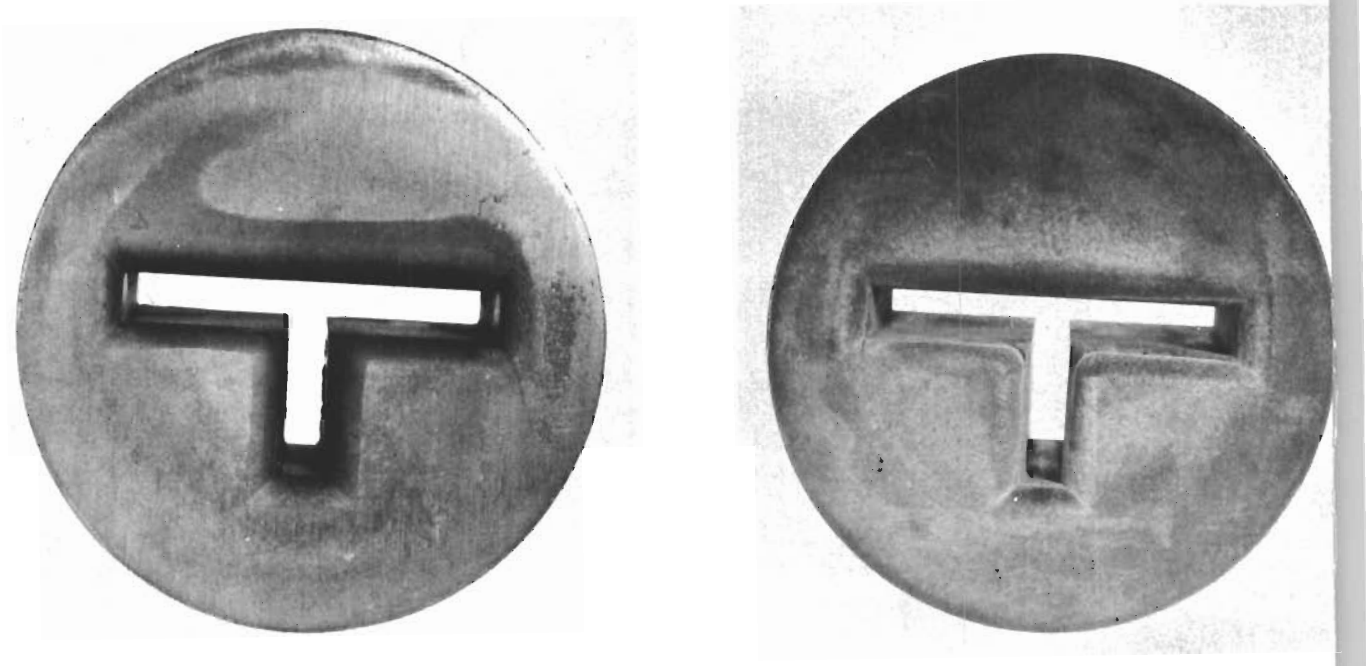
(b)

About actual size

$ZrSiO_4$ - 20 $^{v}/o$ W powder - 20 $^{v}/o$ W fibers

Figure 29. Hot Pressed W Fiber - Composite Tee Dies.

These dies were pressed using solid mandrels. The addition of tungsten powder to the ceramic-tungsten fiber composition was found to increase the theoretical density about 3 percent.



(a)

(b)

Figure 30. Hot-Pressed W Powder - Composite Tee Dies. Magnification: about actual size.

A. Si_3N_4 - 20 $^v/o$ W - 5 $^v/o$ MgO

B. $ZrSiO_4$ - 20 $^v/o$ W

The powders were dry-blended and then hot-pressed at one tsi for 10 minutes in a preshaped graphite mold. The nitride was pressed at 3100°F and the silicate at 2900°F.

The design described above, which incorporated a solid graphite tee shaped mandrel, was found to be unsuccessful in pressing those materials which had a higher expansion coefficient than graphite. These included all the materials of interest except the silicon nitride and zirconium silicate composites. Failure of the mold design occurred because of splitting and cracking of the die during cooling around the graphite mandrel. In an attempt to alleviate this problem, the mold was redesigned to include a small Tee slot into which some powdered die material was placed. This design is diagrammed in Figure 31. Presumably, this change would reduce the stresses which occurred on cooling. The revision was not successful, however, because the mandrel collapsed and the pressing still cracked. A typical failure by cracking on a slitted mandrel is seen in Figure 32. Use of higher expansion mandrels such as boron nitride or other selected materials may be successful.

These alternatives were not investigated. The desired compositions were fabricated instead as discs and then finished by electrospark discharge machining. A summary of Tee die fabrication, conditions, and results is listed in Table 4. Some of the commercially prepared Tee dies are shown in Figure 33.

5. Preparation of Metallic Alloys

Because of the extreme hardness and refractoriness reported for experimental alloys W - 5.5 $^{w}/_{o}$ Ru and Ta - 6 $^{w}/_{o}$ Ru (investigated in other programs under sponsorship of the Air Force and the Atomic Energy Commission) these compositions were fabricated into dies. Elemental powders were blended, pressed into pellets, arc melted, canned in molybdenum, and upset forged in an "insulated" extrusion container at 3600°F and 100 tsi. Each "forging" was cut in half transversely, machined, and shrunk into a steel casing.

Since unalloyed tungsten nibs were found to suffer little wear with steel billets (Section D-1 below) these tungsten and tantalum alloys were not tested with steel, but, for economy, were prepared for testing with TZM billets. Thus, the diameter of the die land was machined to 0.750 inch rather than 0.400 inch. Because of the great hardness of these materials, electrospark discharge machining was used. Using powder-metallurgy techniques, a commercial source prepared a round W - 25 $^{w}/_{o}$ Ta die, also with 0.750-inch diameter orifice.

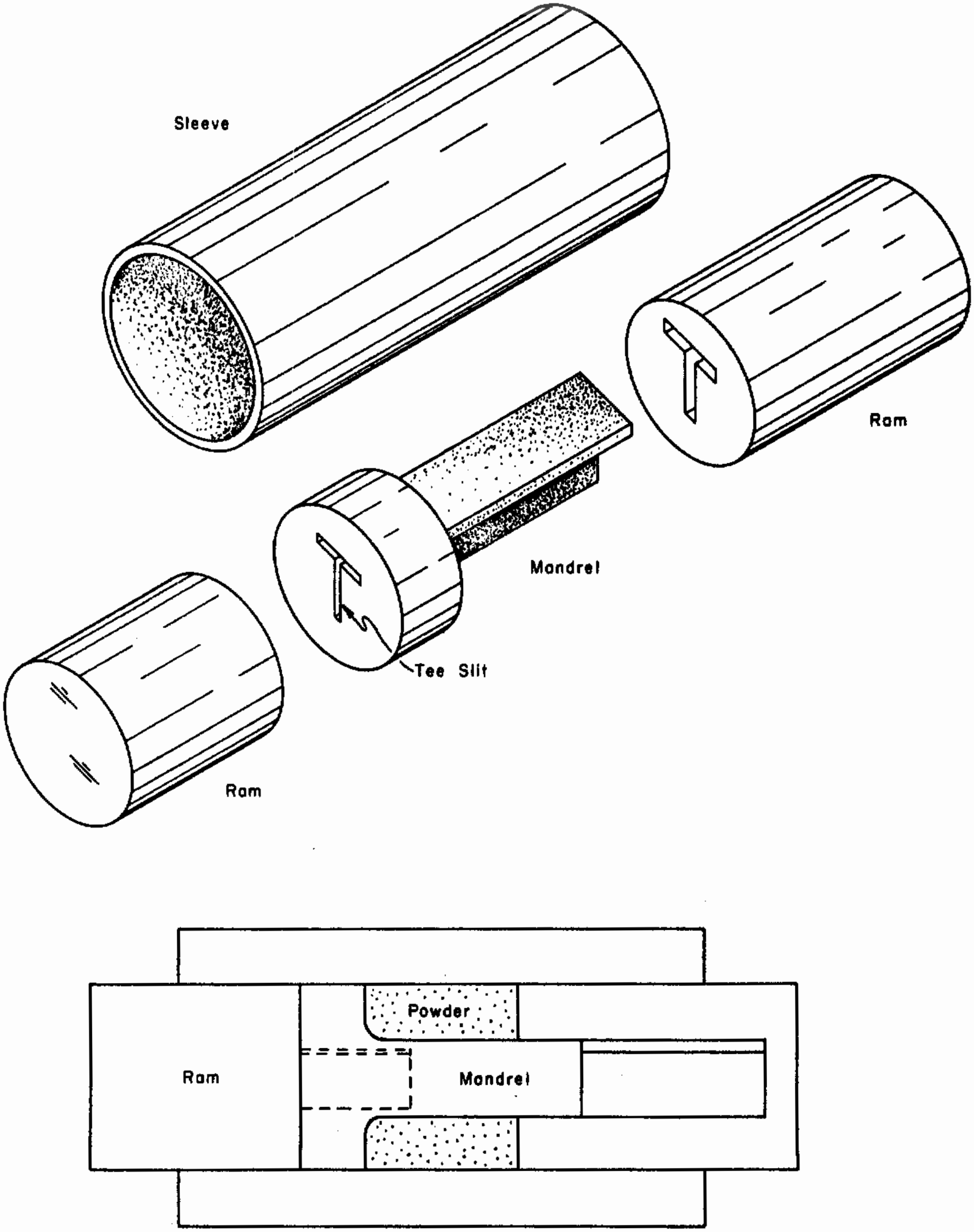
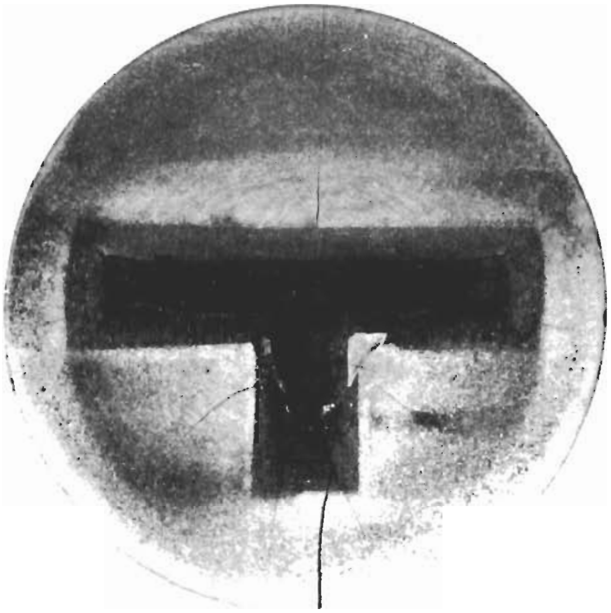


Figure 31. Mold for Higher Expansion Tee Dies.

The Tee slit within the mandrel (indicated by the dotted line in the lower view) was incorporated in an unsuccessful attempt to prevent cracking nibs with "high" expansion coefficients. For those materials with "low" coefficient a solid mandrel was used successfully.



ZrB_2 - 20 $^{v}/o$ Al_2O_3 - graphite mandrel

Figure 32. Cracked Hot Pressing of Tee Die.
Magnification: about actual size.

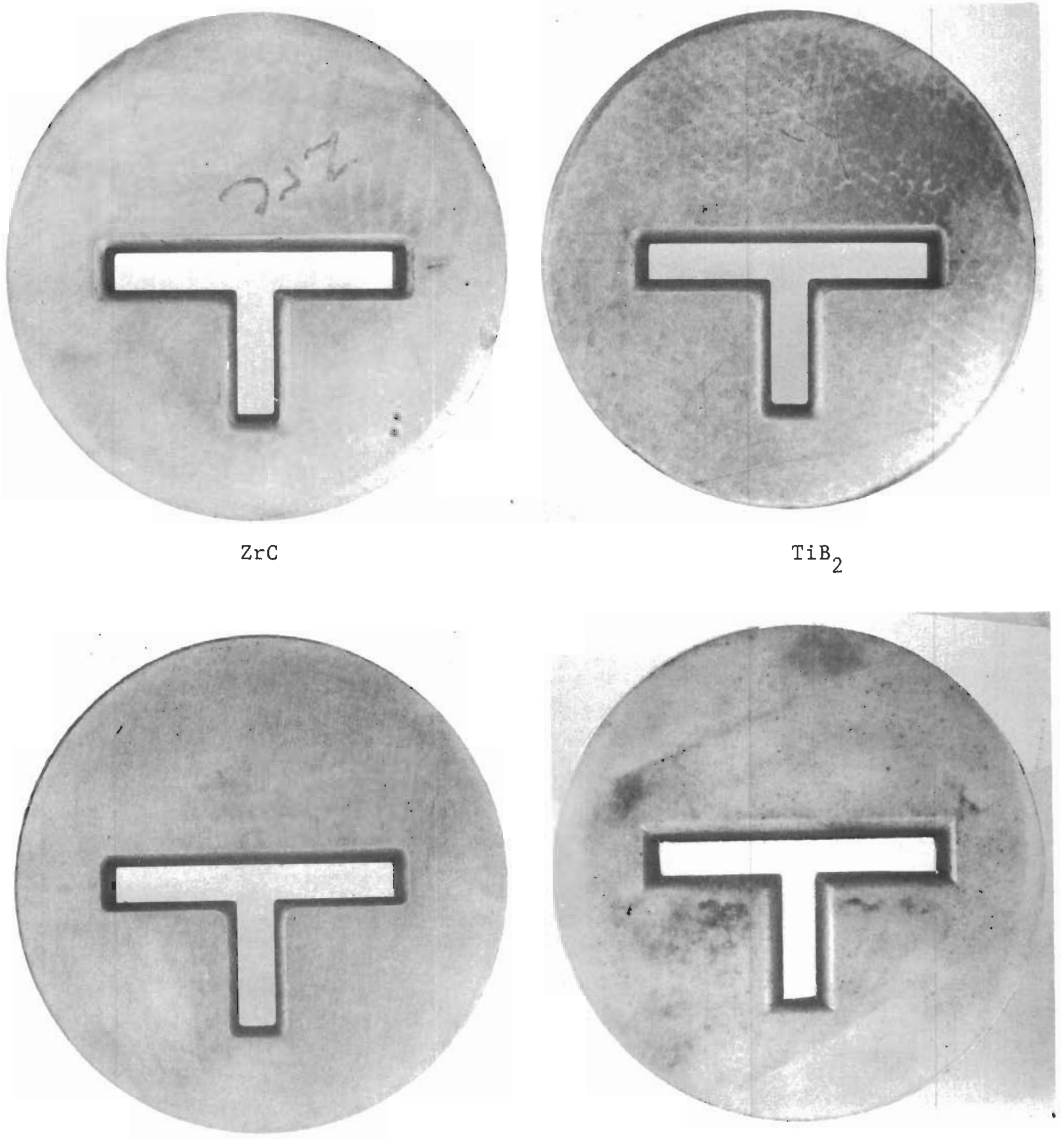
The higher expansion coefficient of the ZrB_2 - Al_2O_3 compared to the graphite mandrel caused the nib to crack on cooling. Slitting the mandrel to alleviate this problem caused the mandrel to collapse during pressing, so that the die still cracked.

TABLE 4

FABRICATION OF HOT PRESSED TEE DIES

Conditions (1)							Results	
Composition		Temp. (°F)	Estimated Expansion Coefficient (2) x 10^{-6} (in./in./°F)	Time at Temp. (min.)	Shape	Mandrel	Visual Appearance	Theoretical Density (%)
Base	Addition (v/o)							
ThO_2	66W	3200	1.8	10	Tee	Solid	Uncracked, dense	-
$MoSi_2$	$20Al_2O_3$	3200	4.5	20	Tee	Solid	Cracked, dense	-
		3200		20	Tee	Slit	Cracked, dense	-
		3250		20	Disc	None	Uncracked, dense (6)	-
		3150		10 (3)	Disc	None	Uncracked, dense	92
TiB_2	$20Al_2O_3$	3550	4.5	20	Tee	Solid	Melted	-
		3950		20	Disc	None	Cracked, dense	-
		3300		10 (3)	Disc	None	Uncracked, dense	93
ZrB_2	$20Al_2O_3$	3450	3.7	20	Tee	Solid	Cracked, dense	-
	20Al203	3300		10 (3)	Disc	None	Uncracked, dense	92
$ZrSiO_4$	20Wf (4)	3000	1.9	20	Tee	Solid	Uncracked, soft	-
	20Wp (5)	2900	1.9	10	Tee	Solid	Uncracked, dense	93
	20Wp - 20Wf	3000	1.7	10	Tee	Solid	Uncracked, dense	93
Si_3N_4 (7)	20Wf	3200	1.6	20	Tee	Solid	Uncracked, soft	73
	20Wp	3100	1.6	10	Tee	Solid	Uncracked, dense	93
	20Wp - 20Wf	3100	1.7	10	Tee (8)	Solid	Distorted	-
	20Wp - 20Wf	3100	1.7	10	Tee	Solid	Uncracked, dense	93
	25Wp - 25Wf	3100	1.7	10	Tee	Solid	Uncracked, dense	93

(1) Pressed in ATJ graphite at about 2000 psi.
(2) Thermal expansion coefficient of graphite about 1.5 x 10^{-6} in/in/°F.
(3) Cooled 400°F under 2000 psi from soak temperature, then pressure released.
(4) Wf = W fibers.
(5) Wp = W powder.
(6) Broke in grinding.
(7) 5 v/o MgO added as sintering aid.
(8) .062 in section.



ZrC

TiB_2

TiC

ZrO_2

Figure 33. Ceramic .250-inch Tee Dies Made Commercially. Magnification: about actual size.

The ZrO_2 die was made by cold pressing, sintering and grinding, the others by hot pressing, Eloxing, and grinding.

D. Testing of Round Dies with Steel Billets

Before tests with TZM billets either as rounds or Tees were performed, a series of experiments involving steel billets was undertaken. These included: (1) bare refractory substrates, (2) ceramic coated dies, and (3) ceramic based dies.

1. Bare Refractory Substrates

a. Purpose

To investigate substrates possibly superior to tool steel in utility and compatibility with prospective coatings, various materials were compared with 18-4-1 alloy steel hardened to 52 R_c and H21 steel hardened to 52 R_c, the latter coated with ZrO_2 by flame spraying. The experimental substrate materials were wrought tungsten, and stress relieved TZM and TZC molybdenum alloys.

b. Conditions

The experimental conditions were complicated by not having all die openings the same size (the materials were available from other work). The tungsten and the 18-4-1 alloy steel were tried in two die sizes, nominally 0.500 and 0.750 inch diameter, and a performance correlation was possible. The die supports were like those described in Section B-5 and shown in Figure 19B. Steel billets heated to 2200°F in conventional resistance furnaces were extruded at a ram speed of 2.3 inches per second with glass lubrication No. 2. Necrolene* was used in the liner and a glass pad was used in the die. Up to five 4340 steel billets, each six inches long, were extruded through each die. The dies that did not fail in the extrusion of steel were used to extrude pure molybdenum at 3400°F with glass lubricant No. 3. The diameter of the die land was measured before and after each extrusion.

c. Results

The results of these measurements and comments on the condition of the dies are given in Table 5. From these data it can be concluded that under the conditions used, bare tungsten, TZM, and TZC are better die materials than bare tool steel, and that ZrO_2 coated tool steel is superior to the bare refractory metals tried. There was evidence, however, that after the sixth extrusion the ZrO_2 coating on the tool steel was about to spall because of severe cracking. The results also implied that refractory metals might make substrates for coatings superior to tool steel.

After the sixth extrusion the zirconia coated steel die was sectioned to reveal the steel-ZrO_2 interface. The hardness of the steel

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* Product of the Crawford Emulsions Company

TABLE 5

ROUND DIE SUBSTRATE TESTS WITH STEEL AND Mo BILLETS(1)

Billet Material =	Steel (4340)						Mo
Billet Temp. (°F) =	2200						3400
Liner Diam (in) =	2.8				3.5		2.8
Sequential Ext. No. =	0	1	2	3	4	5	6
Die Material	Die diameter (in)						
Wrought W	.501	.494	.494	.494	.495	.495	WC
ZrO_2 coated H21	.500	.500	.500	.501	.502	.505	SO
18-4-1	.507	.506	.505	.513	WO	-	-
Wrought TZC	.747	.747	.747	.731	.754	.729	WC
Wrought TZM	.707	.709	.709	.704	.731	.677	WC
Wrought W	.750	.751	.750	.747	.743	.742	WC
18-4-1	.753	.753	.750	.743	WO	-	-

Other Conditions

Billet

Ext. Speed = 2.3 in/sec
Lube = Glass 1(2)
Cutoff = Graphite
Length = 9 in.

Die

Lube = Glass 4
Type = Internal
Temp.= 300°F (estimated)

Liner

Lube = Necrolene
Temp. = 900°F
Clearance = .075 in.

(1) WO = Die washed and opened
WC = Die washed and closed
SO = Die spalled and opened

(2) Except sequential extrusion No. 6 which used Glass 3

50 mils below the ZrO_2 was found to be 50 R_c. The thickness of the ZrO_2 was about 300 mils. The outstanding performance of this die may be attributable to the excessive thickness of the coating. If the coating had been about 30 mils, a standard thickness for coated dies, failure may have occurred earlier. Since some cracking was evident in the coated die after considerable use, a die made entirely of ZrO_2 may perform better than the ZrO_2 coated die because of the absence of the metal-oxide bonding problem and expansion coefficient differences, and lower density in the sprayed ZrO_2 coating. The possible use of massive ZrO_2 as a die appears promising in view of its satisfactory performance in the die support study (Section B-5). Moreover, heavy ZrO_2 coatings may not be practical with Tee dies because of spalling that may be associated with a complicated shape.

2. Ceramic Coated Dies

a. Purpose

Experimental coated dies were fabricated to determine the effect of various coating parameters on coating wear and overall die performance. These variables included the use of refractory and non-refractory substrates, graded and ungraded coatings, and the presence of a hard facing layer.

b. Conditions

The dies were prepared commercially, following the design seen in Figure 34, by arc-spraying powders on substrates that had previously been grit-blasted. Gradations were achieved by spraying one-to-one mixtures of the respective constituents between each constituent layer. The substrates were H 21 steel, hardened to 48 Rockwell C, and stress-relieved TZM molybdenum alloy. The coatings were zirconia based, and approximately 30 mils in thickness; the hard facing was TiB_2. Four dies with steel substrates were coated with either ungraded ZrO_2* or ungraded ZrO_2-TiB_2, graded Fe-ZrO_2, or graded Fe-ZrO_2-TiB_2. Four dies with TZM substrates were similarly coated, except molybdenum was substituted for iron in the graded coatings. The coated dies were polished and measured.

Billets of 4340 alloy steel were machined to 2.90-inch diameter by 8 inch length, with a 90° including angle conical nose and 0.5-inch flat. They were cleaned with trichloroethylene and then coated with lubricating glass (No. 2) which was suspended in cellulose nitrate. Lubrication of

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*ZrO_2 + 5 to $10^{w}/o$ CaO (stabilized)

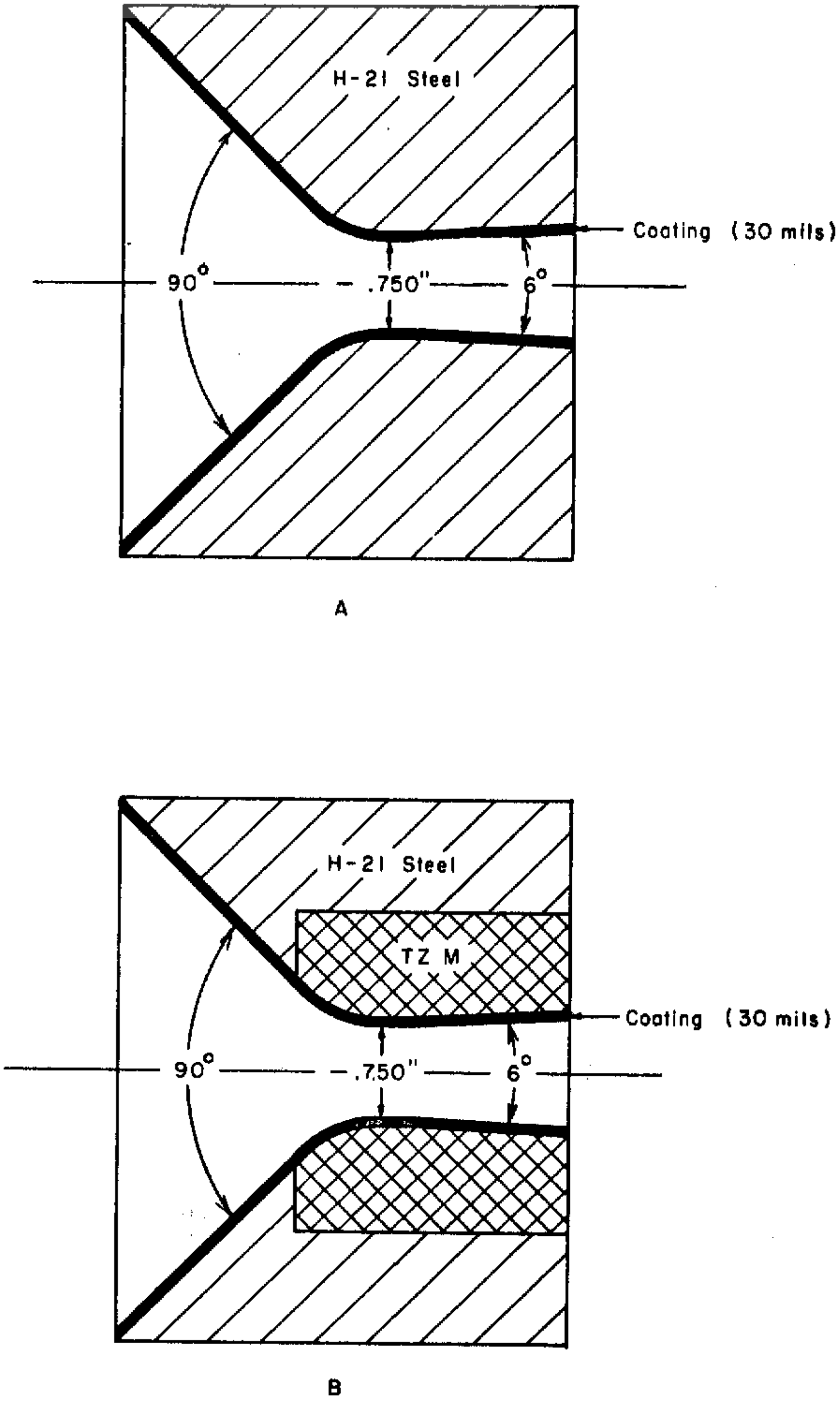


Figure 34. Design of Coated Dies.

A. Coating on H-21 steel.

B. Coating of H-21 steel and TZM nib.

Dies were prepared by grit-blasing, arc-spraying, and polishing.

the liner and die was obtained by swabbing with Necrolene after cleaning the liner with a wire brush. The dies were used unheated. The liner was maintained at 900°F. The 1400 ton extrusion press shown in Figure 35 was used.

Heating for extrusion was carried out in a furnace whose design is similar to that used by several contractors working on related programs. The glass coated billets were successively heated inductively by a coil energized by a 3000 cycle generator under flowing argon as they rested on a steel support. The temperature was measured by an optical pyrometer. After the desired temperature of 2200°F was reached (12 minutes) and maintained (one minute), the billets were lowered on a hydraulic pedestal, automatically tipped horizontally, clasped with tongs manually, and placed on the press loader. The billets were automatically lifted to a position concentric with the liner cavity and ahead of the three inch long graphite cutoff and two inch long steel backer. The stem pushed these materials into the liner and then pushed the billet and part of the graphite through the die at about sixteen times reduction and 11 in./sec. The loading operation took twenty seconds, and the extrusion less than one second. Upset pressures of 49 ± 3 tsi and running pressures of 31 ± 3 tsi were required, corresponding to extrusion constants of 17.5 and 11.1 tsi, respectively. The die, billet, extrusion conditions, and results are tabulated in Table 6.

c. Results

After use, half the die entry was masked off and the opposite side was "vapor-blasted" with a mixture of approximately one-to-one 200 mesh silica and 320 mesh alumina for about 30 seconds to remove glass and graphite adhering to the die surface. The die land and entry of each die was examined for wear or other degradation. The appearance of three typical coated dies after use is illustrated in Figure 36. Since almost all the coatings failed severely, no meaningful measurements of the die land was possible. However, the dies were evaluated in terms of the estimated percent area of the coating still intact. The surfaces of the rods were examined and measured in two planes in four places from front to rear. The rods were evaluated in terms of the maximum variation in diameter and surface condition. The evaluation of dies and the rods produced by them are listed with the die composition and type (graded versus ungraded) in Table 6.

As can be seen in the table, there is little or no correlation among good coating performance, rod uniformity, die composition and die type. The rod diameters were found to vary as much as .008 in. and were galled and striated. This is probably attributable to the fact that all the dies tested in this experiment evidently had non-adherent coatings, and extrusion conditions were not sufficiently severe to degrade the dies with either substrate. Although testing of replicates of these dies with TZM molybdenum alloy was planned, dies with more promising properties were sought instead.

TABLE 6

COATED DIE TESTS WITH STEEL BILLETS

Conditions			Results			
Die			Substrate Condition	Coating % Area Intact	Rod	
Substrate	Coating	Type			Surface	Size Variation (mils)
Steel*	ZrO	Ungraded	Intact	25	Galled	3
	Fe-ZrO_2	Graded		75	Galled	5
	Fe-ZrO_2-TiB_2	Ungraded		0	Scored	6
	Fe-ZrO_2-TiB_2	Graded		75	Scored	3
TZM	ZrO	Ungraded	Intact	90	Scored	8
	Mo-ZrO_2	Graded		25	Scored	7
	Mo-ZrO_2-TiB_2	Ungraded		50	Smooth	3
	Mo-ZrO_2-TiB_2	Graded		75	Scored	2

Other Conditions

Billet

Diameter = 2.90 in.
Length = 8.0 in. + 90° Nose
Cutoff = Graphite
Reduction = 16.5:1
Material = 4340 Steel
Temp. = 2200°F
Heating Time = 12 min.
Soak Time = 1 min.

Extrusion

Upset Force = 340 ± 20 tons
Running Force = 230 ± 20 tons
Upset Pressure = 49 ± 3 tsi
Running Pressure = 31 ± 3 tsi
Upset Pressure = 17.5 tsi
Running Pressure = 11.1 tsi
Speed = 11 in/sec.

Liner

Diameter = 3.050 in.
Identity = 31B
Load Time = 20 sec.
Condition = Fair

Die

Type = Internal
Temperature = 150°F (estimated)
Diameter = 0.750 in.
Lubrication = Aquadag + Necrolene

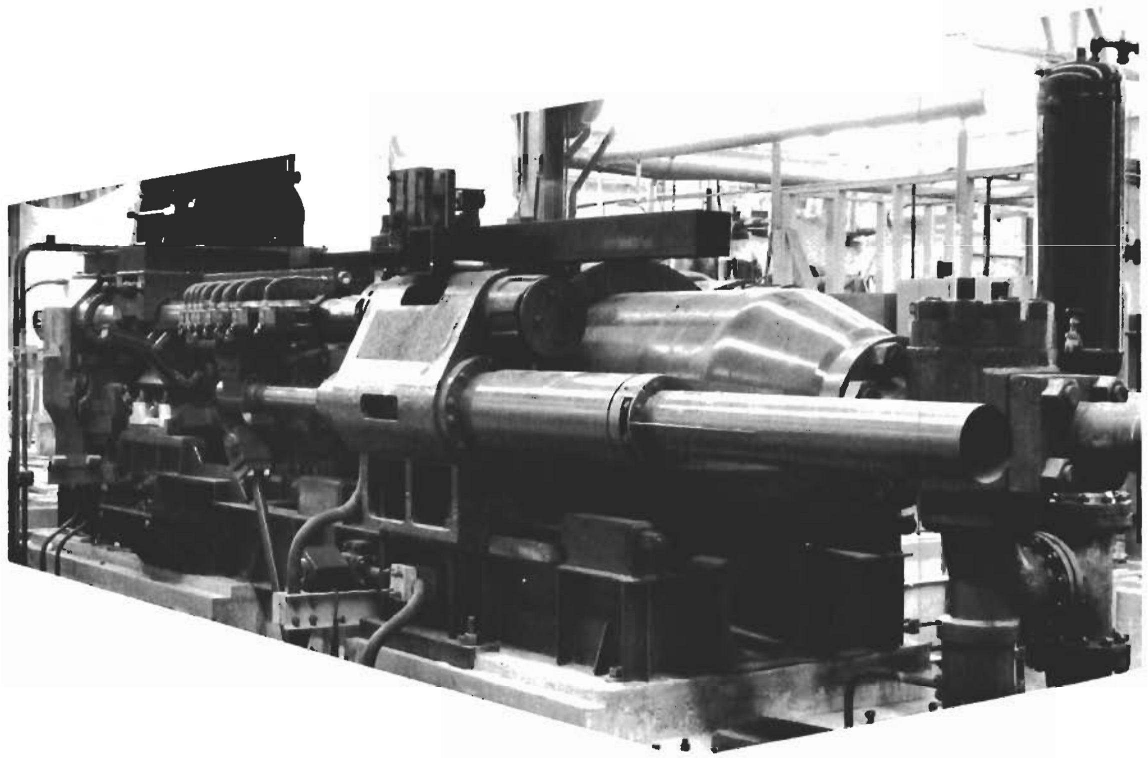
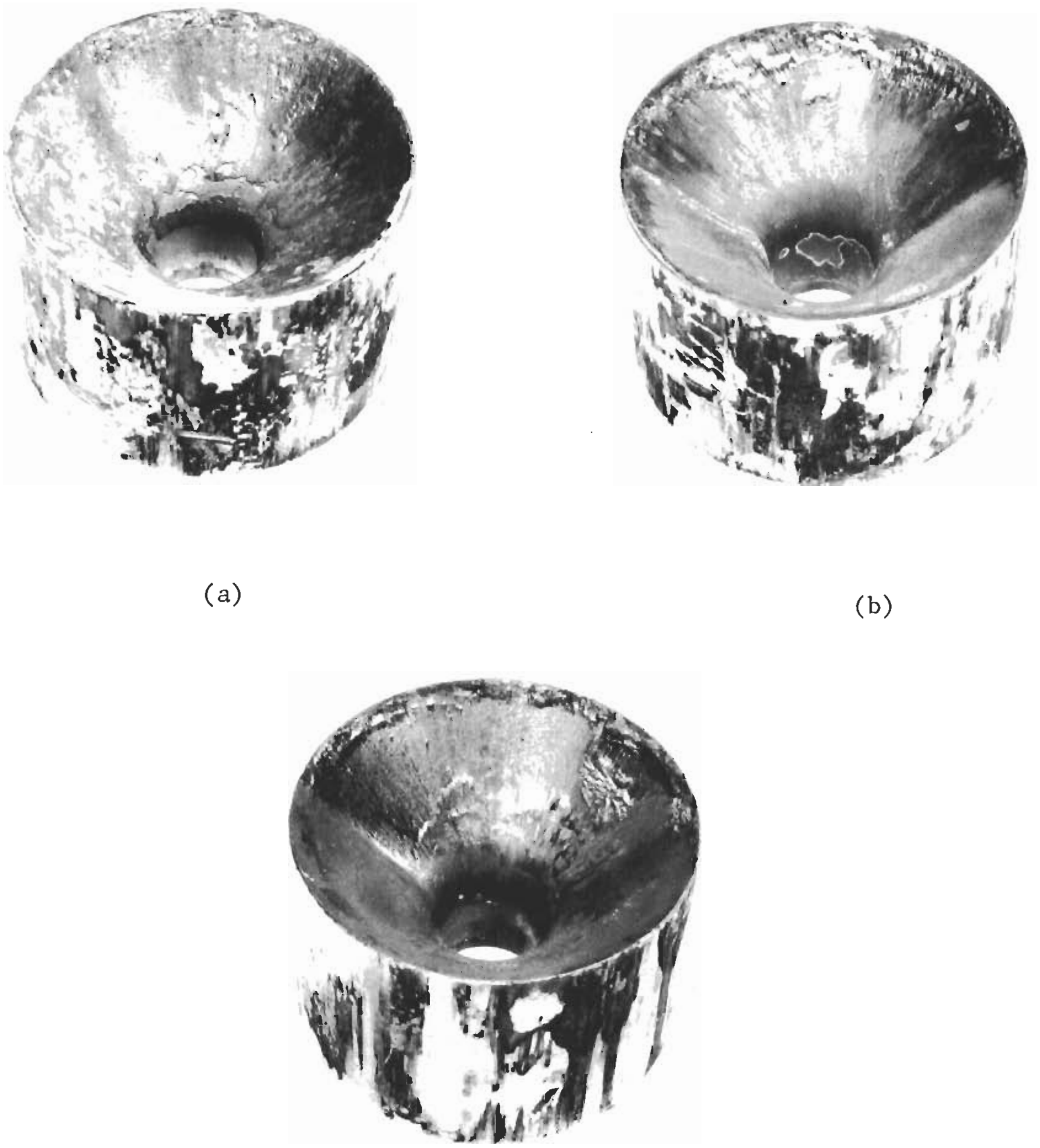


Figure 35. 1400-Ton Extrusion Press.

All experimental extrusions, except the preliminary experiments described in Section B, were performed on this press at speeds up to 25 in./sec.



(a)

(b)

(c)

Figure 36. Typical Appearance of Used Coated Dies. Magnification: about 0.7 actual size.

A. Ungraded ZrO_2 on steel substrate

B. Graded Fe - ZrO_2 on steel substrate

C. Graded Fe - ZrO_2 - TiB_2 on steel substrate

3. Ceramic Based Dies

a. Purpose

In Section C above, a variety of dies was prepared which represented several categories of ceramic based dies. These include self-bonded, metal-bonded (by powder as well as fibers), and oxide-bonded dies. To distinguish the merits of these types and to determine their qualifications for further testing with TZM molybdenum alloy and ultimately Tee dies, the ceramic-based dies were tested with 4340 alloy steel billets.

b. Conditions

The nibs were prepared by hot-pressing and grinding both at Nuclear Metals and commercial sources. The steel support or casing was prepared by the following steps. An aluminum sleeve with oversized wall was pressed into the nib cavity of the casing. The aluminum was then machined to a thickness of about 50 mils, with the inside diameter 2 mils smaller than the outside diameter of the nib. The casing was heated for three hours at 900°F and withdrawn from the furnace. The nibs, at room temperature, were pressed into the casings and cooled under a weight. In this way a layer of aluminum existed between the casing and the nib. Finally, a copper sleeve 0.10 inch longer than the casing and preheated to 900°F was shrunk over the outside of the casing to complete the assembly, as shown in Figure 37. In this design, described in Section B-5, the force exerted by the billet results in a compressive force on the nib to prevent severe cracking which would otherwise occur.

Billets of 4340 alloy steel were prepared like those used with coated dies except that the diameters were 3.400 inch and their rear surface had a centered, threaded hole. These billets were heated in a furnace of special design, originally intended for TZM molybdenum alloy only, but was found useful for steel as well. A sketch of the furnace and loading setup is seen in Figure 38. After being coated with lubricating glass (using the same technique for coated die tests), the billets were successively placed in the furnace, which was evacuated to about 500 microns and then backfilled with argon. Each billet was heated by induction in flowing argon atmosphere as it sat with its nose on a tantalum foil wafer. The latter rested on a graphite pedestal threaded into a carbon support. The temperature was measured by an optical pyrometer, which was used to sight into a drilled and tapped recess at the rear of the billet. (This recess was also used to load the billets into the furnace with a threaded rod.) During billet heating, the liner, maintained at 900°F, was cleaned with a wire brush and then lubricated with Necrolene. The dies were loaded without heating. Then a dummy block two inches long was fastened to the ram by means of a tubular pin. After the extrusion temperature was reached (seven minutes) and maintained (two minutes), a latch, which supports the billet, was released. The carbon

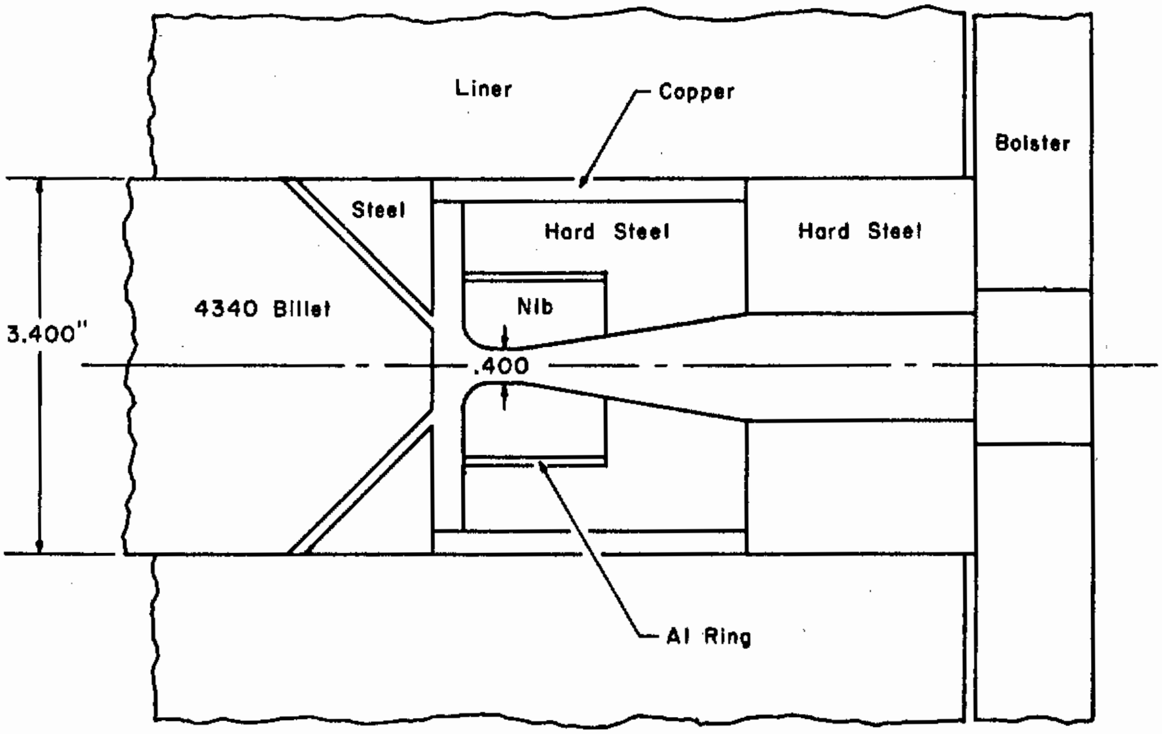


Figure 37. Tooling for Testing Round Dies with Steel Billets.

Force exerted by the billet results in a compressive force on the die nib to prevent severe cracking which would otherwise occur.

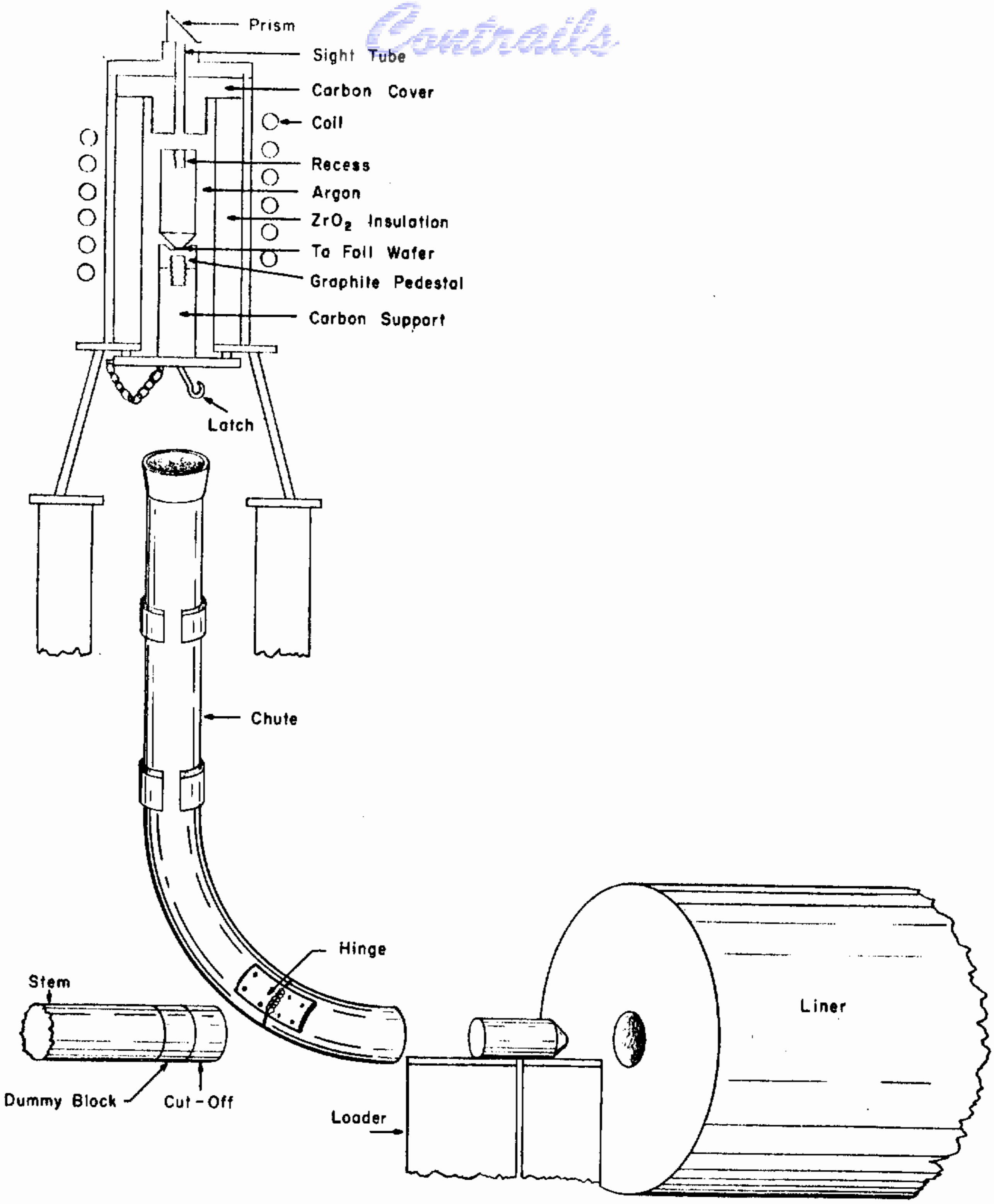


Figure 38. Design of Furnace and Loader.

Release of the latch causes the billet to free-fall behind the liner. Then the stem pushes the billet and part of the cutoff through the die.

support was pushed down and sideways by the weight of the billet, which in turn fell freely down a curved chute onto a platform behind the 3.545-inch diameter extrusion liner. A three-inch long copper, 0.5-inch long graphite cutoff assembly was placed by hand behind the billet. The stem pushed this column of materials into the liner and then the billet and the graphite through the die. The loading operation took two to three seconds and extrusion proceeded with a speed of 12 in./sec. The size of the nibs (nominally 0.400 inch) required a reduction ratio of about 80 times. (Thus, less than a second was required to extrude the billet which yielded a rod about sixty feet long.) Upset pressures of 55 ± 5 tsi and running pressures of 45 ± 5 tsi were required, corresponding to extrusion constants of 12.5 and 10.3 tsi, respectively.

c. Results

Before evaluation, each nib was removed from its support by dissolving the aluminum ring in hot NaOH. Some of the rods did not completely clear the dies as intended. In these cases, the remaining steel was dissolved in nitric acid without disturbing the nib mechanically. To remove adherent glass and graphite, each nib was vapor-blasted with the same grit that was used for the coated dies (Section D-2). The land diameter was measured for comparison with the original size. Rods were measured in four places from front to rear in two planes and examined for surface quality. A comparison of performance for various massive ceramic-based dies with the control dies is shown in Table 7. The control dies were (1) H-21 without coating, (2) H-21 steel coated with 0.030 inch of ZrO_2 and (3) H-21 steel coated with 0.300 inch of ZrO_2.

As is shown in the table, there is good correlation between die performance and rod quality (which is in contrast to the results obtained in the coated die experiment in Section D-2). Dies which eroded produced scored, nonuniform rods; dies which withstood wear produced uniform, smooth rods. The self-bonded types performed well with the exception of alumina which cracked severely, and B_4C which eroded badly. Of the metal-bonded dies, the relatively porous materials (Al_2O_3-W, ThO_2-W), and those bonded with nonrefractory materials (TiC-Ni-Mo, TiC-Fe, TiC-SS)* were badly degraded by erosion. The performance of the oxide-bonded materials was good except for those which were known to have low relative density. There were eleven dies which gave particularly good results: $ZrSiO_4$-W powder, $ZrSiO_4$-W fibers, TaC-W, TaC-Ni, Si_3N_4-MgO-W, TiB_2, ZrB_2, ZrC, ZrO_2, $MoSi_2$-Al_2O_3, TiB_2-Al_2O_3, ZrB_2-Al_2O_3 and Al_2O_3-SiO_2-Mo. These nibs, based on their appearances, probably could have withstood additional extrusions. Of the controls, the die with the thicker ZrO_2 coating appeared to show less cracking and spalling than the die with the thinner coating, while the bare H-21 was severely eroded. A visual comparison of all the dies tested in this experiment is seen in Figures 39, 40, 41, 42, and 43 which show the self-, oxide-, and metal-bonded types and control dies respectively. Three types of cracks, most of which are visible in the figures,

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* SS=Stainless Steel

TABLE 7

CERAMIC-BASED DIE TESTS WITH STEEL BILLETS

Type	Die(1)					Rod	
	Composition(2)	Relative Density	Wear (mils)	Cracks	Performance Rating*	Size Variation (mils)	Surface Condition
Metal Bonded	$ZrSiO_4$-W_p(3)	0.93	3	Very Few	Good	3	Smooth
	$ZrSiO_4$-W_f(4)	0.94	0	"None"	Good	4	Smooth
	TaC-W**	0.99+	1	Very Few	Good	3	Smooth
	Al_2O_3-W	0.92	8	Few	Poor	6	Scored
	ThO_2-W	0.93	12	Few	Poor	8	Scored
	Si_3N_4-MgO-W	0.95	2	None	Good	3	Smooth
	TiC-Ni-Mo**	0.99+	85	Several	Poor	35	Scored
	TiC-Fe(5)**	0.99+	203	Several	Poor	142	Scored
	TiC-SS(6)**	0.99+	-32(7)	None	Poor	(8)	Scored
	WC-CO	0.99+	72	None	Poor	69	Scored
	W-Ni-Cu	0.99+	28	None	Poor	22	Scored
	TaC-Ni	0.99+	3	Few	Good	3	Smooth
Self-Bonded	TiB_2**	0.99	1	Few	Good	5	Smooth
	ZrB_2**	0.96	0	Few	Good	4	Smooth
	ZrC**	0.94	0	Very Few	Good	4	Smooth
	B_4C**	0.99	23	Very Few	Poor	18	Scored
	Al_2O_3**	0.99	1	Many	Fair	3	Smooth
	ZrO_2**	0.95	0	Several	Good	2	Smooth
Oxide Bonded	$MoSi_2$-Al_2O_3	0.98	1	Few	Good	3	Smooth
	$MoSi_2$-ZrO_2	0.94	3	Several	Fair	6	Smooth
	$MoSi_2$-ThO_2	0.93	7	Many	Fair	8	Rough
	TiB_2-Al_2O_3	0.99	1	Few	Good	2	Smooth
	TiB_2-ZrO_2	0.97	2	Few	Good	6	Smooth
	TiB_2-ThO_2	0.99	5	Many	Good	5	Smooth
	ZrB_2-Al_2O_3	0.99	3	Several	Good	4	Smooth
	Si_3N_4-MgO	0.98	2	Several	Good	3	Smooth
	Al_2O_3-SiO_2-Mo(9)	0.99+	3	One	Good	4	Smooth
Controls	ZrO_2H21(.03 in)**	-	1	Spalling	Good	8	Smooth
	ZrO_2H21(.30 in)**	-	1	Several	Good	6	Smooth
	Bare H21 Steel	-	84	None	Poor	61	Scored

* Based on wear (dies and rods change less than ± 3 mils).

** Obtained commercially

(Table continued on next page).

TABLE 7 (continued)

Billet

Temperature = 2200°F
Diameter = 3.400 in.
Length = 9.0 in.
Reduction = 80:1
Cutoff = Copper-Graphite
Lubrication = Glass 2

Die

Size = .400 in.
Temperature = 150°F (estimated)
Lubrication = Aquadag

Liner

Diameter = 3.545 in.
Identity = 32-A
Lubrication = Necrolene

Extrusion

Speed = 12 in/sec.
Load time = 3 secs.
Upset press = 55±5 tsi
Running press = 45±5 tsi
Upset k = 12.5 tsi
Running k = 10.3 tsi

Notes:

(1) Nominal size 0.400-inch except ZrO_2, ZrB_2-Al_2O_3 and the two ZrO_2-coated dies which were 0.500-inch.

(2) Nuclear Metals binary mixtures consist of 80 volume percent of first constituent shown plus 20 volume percent of second constituent; the Si_3N_4 mixture has a 75-5-20 volume percent ratio. Commercial sources indicated by **

(3) W_p = W powder

(4) W_f = W fibers

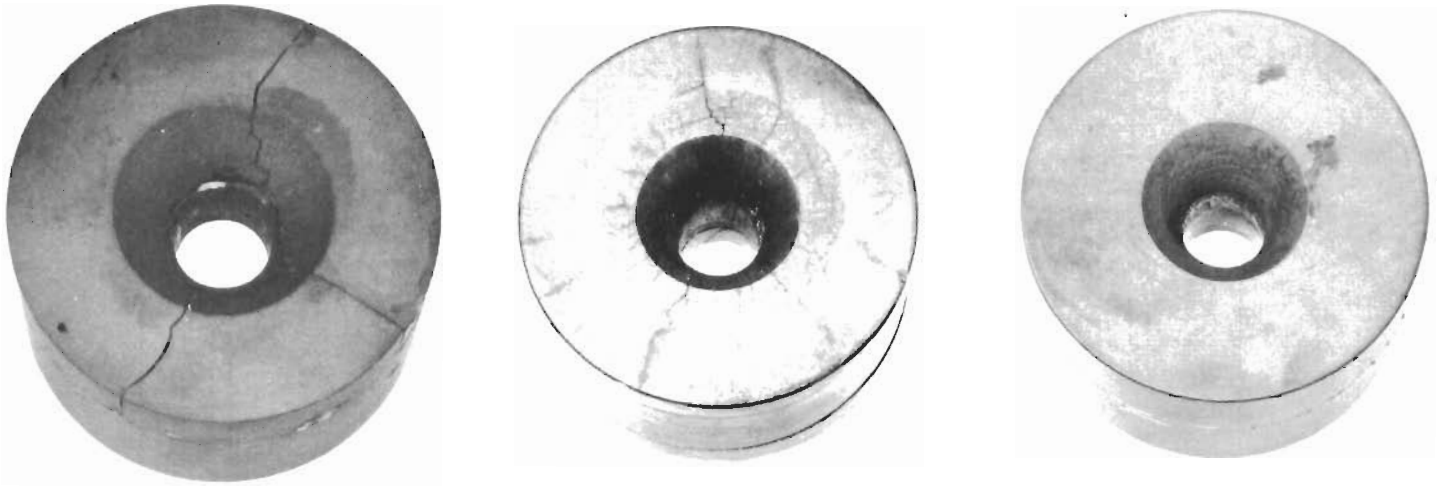
(5) Fe = hardened steel

(6) SS = stainless steel

(7) Die closed in

(8) Billet 85% unextruded

(9) Actually Al_2O_3-$SiO_2 \cdot 3Al_2O_3$-Mo



TiB_2 ZrB_2 ZrC

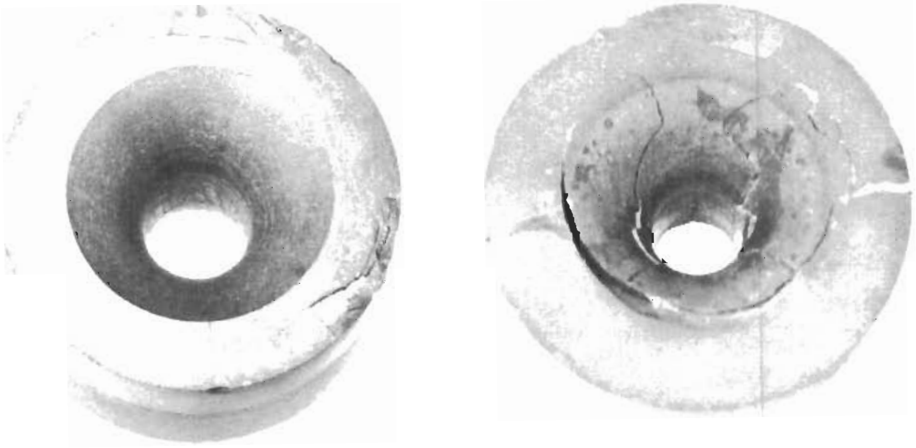
ZrO_2 Al_2O_3 B_4C

Figure 39. Self-Bonded Ceramic Dies after Steel Tests. Magnification: about actual size.

All dies performed well except Al_2O_3 which cracked severely and B_4C which eroded badly.

$MoSi_2$-Al_2O_3 $MoSi_2$-ZrO_2 $MoSi_2$-ThO_2

TiB_2-Al_2O_3 TiB_2-ZrO_2 TiB_2-ThO_2



ZrB_2-Al_2O_3 Si_3N_4-MgO

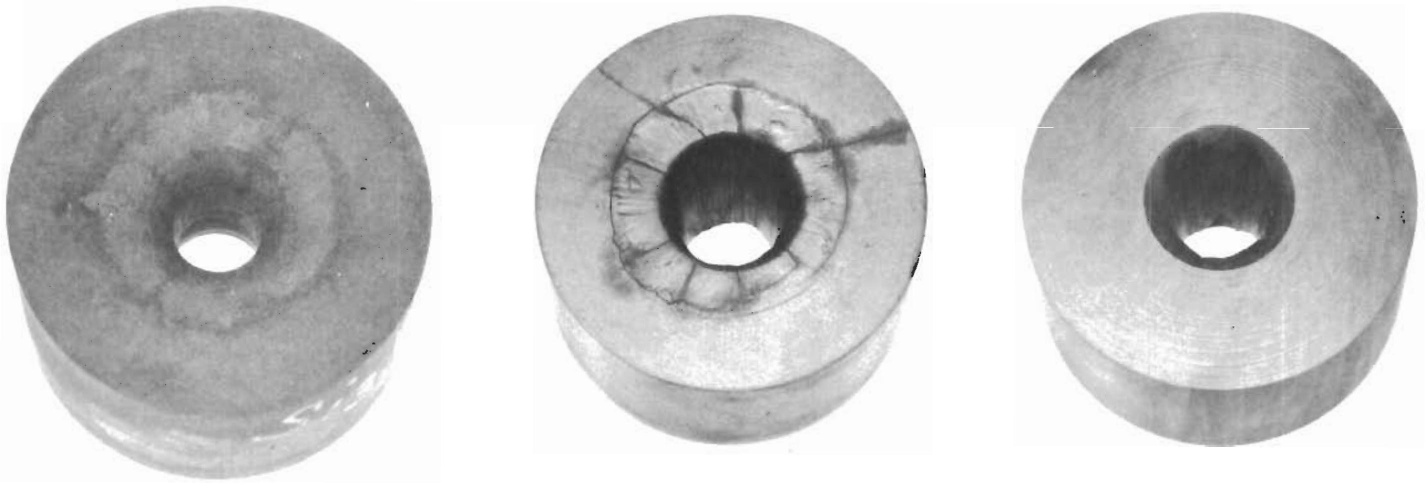
Figure 40. Oxide-Bonded Ceramic Dies after Steel Tests. Magnification: about actual size.

Performance of dies with high relative density was good. Those with considerable porosity were fair; these include $MoSi_2$-ThO_2 and TiB_2-ZrO_2.

$ZrSiO_4$-W powder

$ZrSiO_4$-W powder

TaC-W



Si_3N_4-MgO-W

TiC-Fe

TiC-SS

TiC-Ni-Mo

ThO_2-W

Al_2O_3-W

Figure 41. Metal-Bonded Ceramic Dies after Steel Tests. Magnification: about actual size.

The presence of low melting binders (Fe, Cr, Ni) appears to cause severe erosion. Low density includes buckling and distortion (ThO_2-W, Al_2O_3-W).

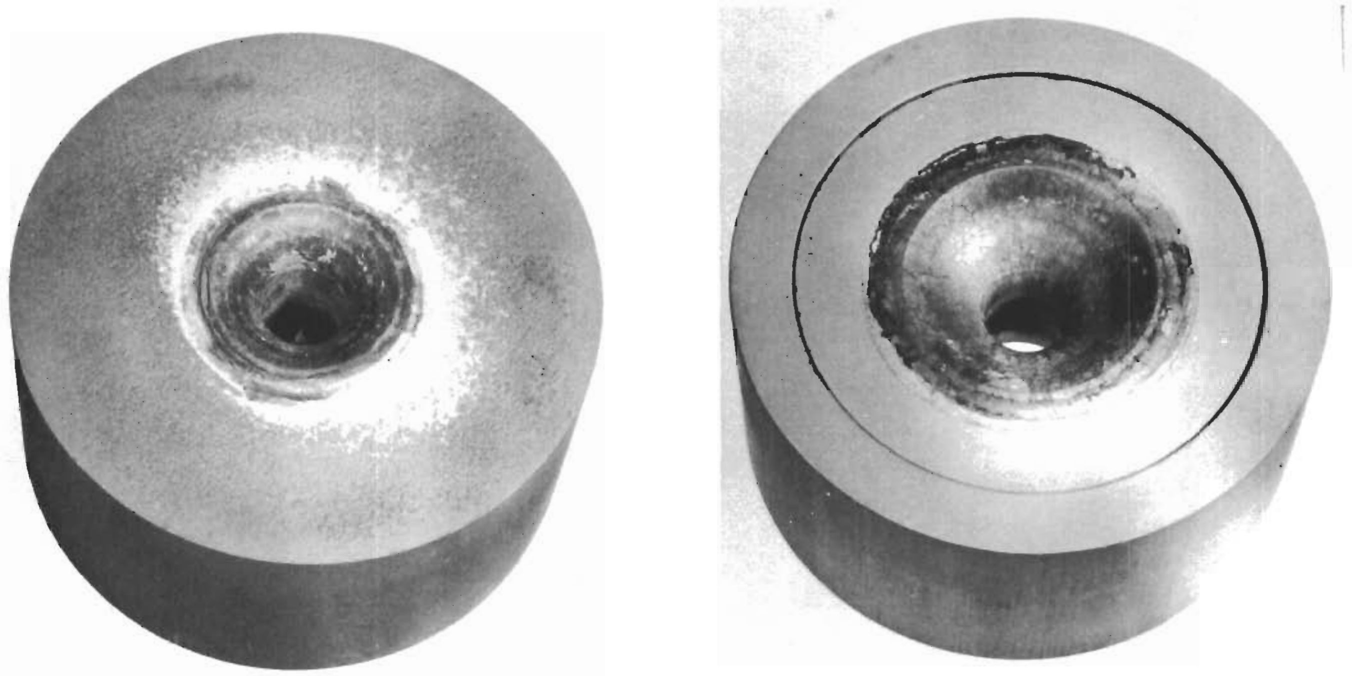
TaC-Ni

W-Cu-Ni

Al_2O_3-SiO_2-Mo

Figure 42. Metal-Bonded Ceramic Dies after Steel Tests.

The TaC-Ni and Al_2O_3-SiO_2-Mo dies resisted erosion whereas the W-Cu-Ni "washed". The cracks on the outside bottom surface of the TaC-Ni nib were found in an abortive shrinking and press fitting (nib assembly) operation.



0.030 in. ZrO_2
coating in steel

0.300 in. ZrO_2
coating in steel

Figure 43. ZrO_2-Coated Control Dies after Steel Tests. Magnification: about actual size.

The thinner ZrO_2 coating (left) showed some spalling, whereas the thicker coating did not. Neither die was worn significantly.

were found in most of the nibs. The commonest was a crack around the nib's circumference, about in the middle, in a plane parallel with the bottom. Many nibs had radial cracks around the orifice which sometimes propagated to the outside of the piece. Others had cracks which were more or less in a plane around the orifice. Several of the nibs had all three types. These cracks are probably attributable to the support design.

E. Testing of Round Dies with TZM Billets

Prior to their testing as Tee shaped dies some experimental dies were tested with TZM molybdenum alloy billets. These were (1) promising materials which were not tested with steel because of the expense of preparing dies for both steel and TZM, and, (2) materials which had given promising performance in steel extrusion. Verification of lubrication procedures, however, preceded the evaluation of these two groups of materials.

1. Tests of Lubrication Procedure

a. Purpose

Prior to testing round, massive, ceramic or ceramic based nibs with TZM molybdenum alloy, an experiment was performed to determine standard lubrication procedures to TZM billets. The ZrO_2 nibs previously used for support study experiments described in Section B-5 were prepared by enlarging the 0.500 inch diameter land to 0.750 inch. These nibs were chosen primarily because a number of them were immediately available, and they had performed well in the earlier steel extrusions. They were not removed from their casings, but were reworked in place. A steel sleeve, whose outside diameter was five mils under the liner size (3.545 inches) was shrunk around the 2.5 inch casing. This latter part was originally used in the 2.8 inch liner with a 150 mil copper sleeve, as described in Section B-5. Molybdenum instead of steel cones were used for the tests. The assembly is illustrated in Figure 44.

b. Conditions

Billets of TZM molybdenum alloy were prepared which had 8 inch length plus a nose with 90° included angle. The nose had a 1.5 inch flat and a rear with a centered, drilled and tapped hole 0.625 inch diameter by 0.500 inch depth. The billets were heated and loaded in the same manner in the high temperature furnace described in Section D-3. However, the billet temperature was increased to 3400°F for six billets, and 3200°F for one. The heating time and holding time were twelve and two minutes, respectively. The liner temperature was maintained at 500°F. Seven billets with various lubrications were tried, involving six coded glass variations which are listed in Table 8. Ram speeds employed were 17 in./sec. and extrusion reductions were 22:1. Upset pressures were about 80 ± 10 tsi, and running pressures about 70 ±10 tsi, corresponding to extrusion constants of 26 and 22.5 tsi, respectively. To minimize the escape of heat into the graphite-copper cutoff used for the steel extrusions in Section D-3, the cutoff design was modified. Attached to the 0.500-inch thick graphite disc were successively two layers of carbon cloth and a disc of 5 mil molybdenum sheet which faced the billet.

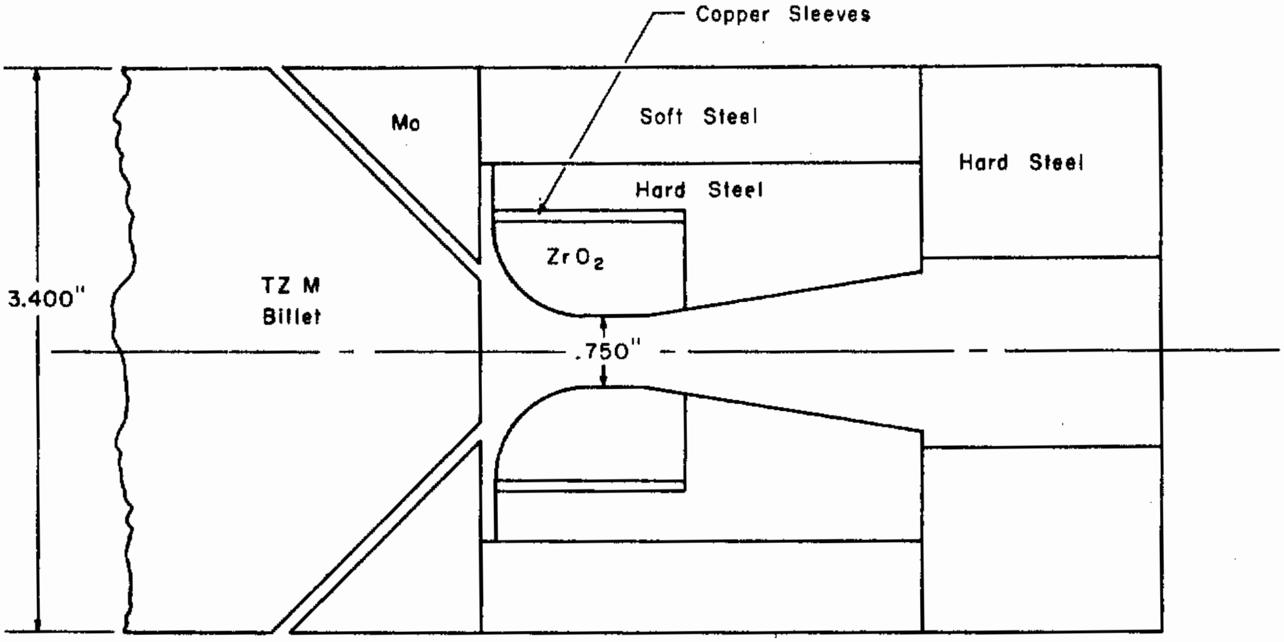


Figure 44. Support Design for ZrO_2 Nibs for TZM Extrusion.

The support system used in the testing of massive ceramic nibs with steel Figure 19 was modified for use with TZM by replacement of the steel cone with molybdenum, and the copper support with steel. The glass pad used for these extrusion trials is not shown.

TABLE 8

ROUND DIE LUBRICATION WITH TZM BILLETS

Conditions						Results			
ZrO_2 Nib	Billet Temp.	Billet Lubrication		Die Pad(1) Length (in)	Linear Lubrication	Extrusion Pressure (tsi)		% Smooth Rod Surface	Die Wear (mils)
	(°F)	On Heating	After Heating			Upset	Running		
A	3400	Glass-3	None	4	Necrolene	85	85	20	0
B	3400	None	None	4	Glass-2(3)	90	75	10	1
C	3400	None	None	4	Glass-3(3)	80	70	75	1
D	3200	None	Glass-2(3)	4	Glass-2(3)	80	Stall	--	-
E	3400	None	Glass-2(2)	12	Mo Coat G	70	60	80	2
C	3400	None	Glass-2(2)	12	Mo Coat G	95	85	20	2
E	3400	None	Glass-2(2)	12	Mo Coat G	90	Stall 1/2	20	1

Other Conditions

Liner

Load Time - 2 sec.
Diameter - 3.545 in.
Condition - Fair

Die

Material - ZrO_2
Orifice - .750 in.
Type - Internal

Billet

Diameter - 3.400 in.
Cutoffs - Cu-Gr
Reduction - 22:1
Ram Speed - 17 in/sec

(1) Glass-4

(2) Billet was placed on one pad Glass-4 which was heavily coated with Glass-2, and then covered with another pad similarly coated.

(3) Dusted on sticky surface within liner, formed by mixture of Glass-2 and molasses which was previously swabbed in liner.

c. Results

Five of the seven extrusions were fully extruded; the 3200°F billet stalled automatically because the stress limit of the tools was approached; one billet extruded only about 50 percent due to the premature closure of a damaged limit switch.* The surfaces of the rods varied from fair to bad, and considerable variation in surface smoothness existed on the same rod. The extrusion which had the best surface was obtained from a billet heated bare and, after falling down the chute to the loader, was placed between two pads of glass-4. These were coated with glass-2. The excess glass was scraped off as the billet was pushed into the liner. Since about 25 percent of the surface along one side was galled, the lubrication procedure is characterized as inadequate. The use of a "glass table" on which the billet can be revolved a sufficient number of times in glass was probably needed.

All the dies withstood these additional extrusions (they were used earlier for steel) with 2 mils of wear or less without serious degradation, as shown in Figure 45 and Table 8. Two of the dies withstood two extrusions. (Probably the others could have as well.) Correlation between die degradation and rod smoothness is not evident, since the smoothest die, A in Figure 45, gave the second roughest rod.

2. Tests of Experimental Rounds

a. Purpose

Prior to testing as Tee dies, the W-5.5^{w}/o Ru, Ta-6^{w}/o Ru and W-25^{w}/o Ta round dies described in Section E-5 were tested with TZM billets. (Round dies of these expensive materials were not prepared for tests with 4340 steel to minimize costs.) Three tungsten dies available from other work were used in preliminary extrusions for comparison and to determine desirable ram speeds. In addition, dies with ceramic-based composition, which had resisted degradation in steel extrusion, were extruded as round dies with TZM billets prior to their fabrication and testing as Tee dies.

b. Conditions

The nibs were supported in a steel casing with the soft copper support used previously for steel extrusions and described in Section D-3. The aluminum ring used in the support for steel extrusion

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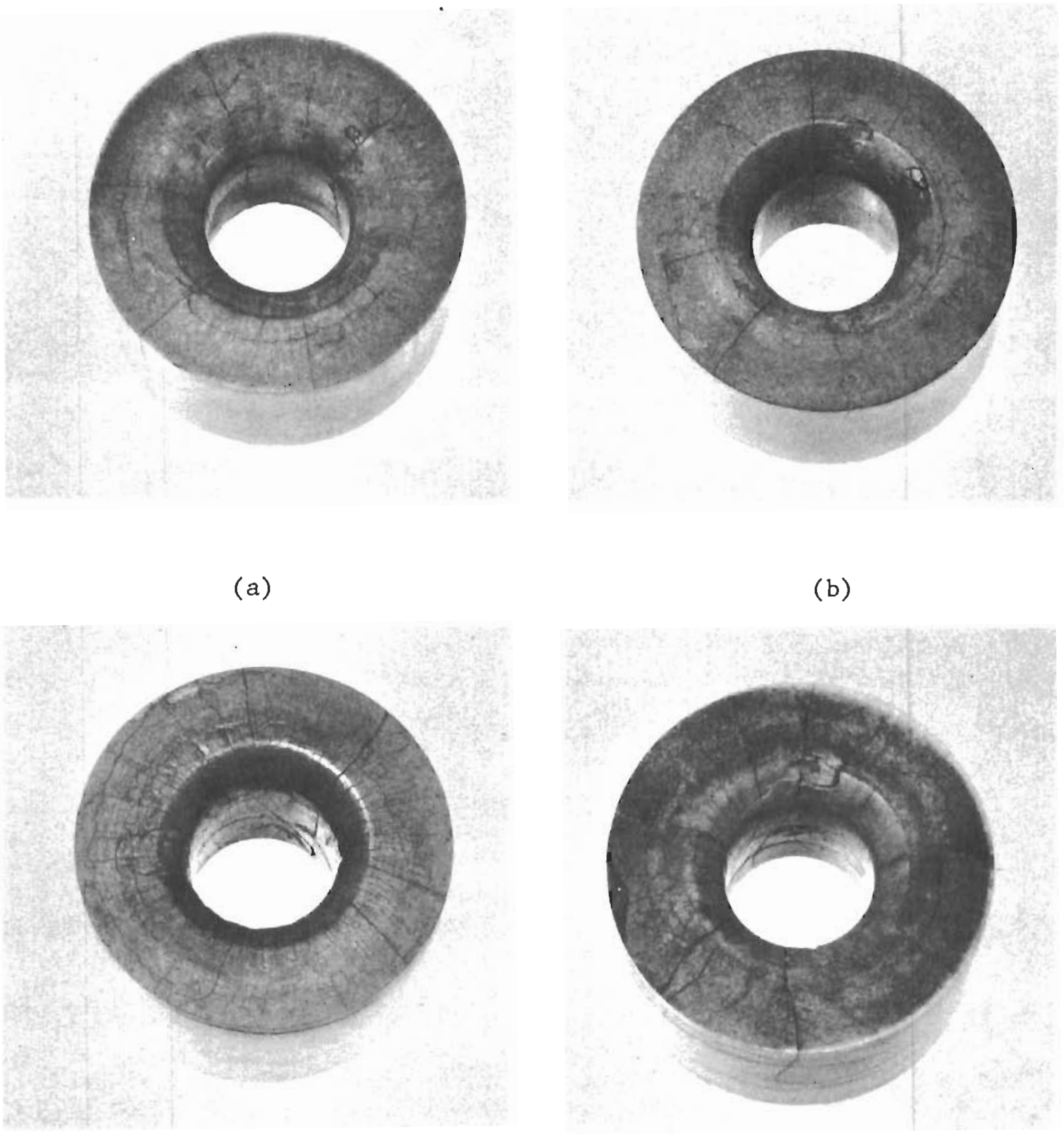
*A hot billet in an abortive attempt slipped off the loader and fell against a limit switch.



(a) (b)

(c) (d)

Figure 45. Zirconia Nibs after TZM Tests. Magnifcation: about actual size.

A. After one extrusion, 0 mil of wear
B. After one extrusion, 1 mil of wear
C. After two extrusions, 2 mils of wear (total)
D. After two extrusions, 1 mil of wear (total)

was replaced by higher melting mild steel, because of the close proximity of the sleeve to the extruding molybdenum alloy. (A sleeve of some sort was necessary to utilize the casings previously used for the steel extrusions.) The steel cones used in the steel extrusion were replaced by molybdenum cones as shown in Figure 46

Billets of TZM molybdenum alloy, with an eight inch length (plus a nose with a 90° included angle) and 3.375 inch diameter, were prepared. The nose had a 1.5 inch flat and the rear a centered, drilled and tapped hole, 0.625 inch diameter by 0.500 inch depth. The billets were heated and loaded in the high temperature furnace as described in Section D-3 and E-1 above. The heating time and holding time were about twelve and one minutes, respectively. The liner temperature was maintained at 900°F. The cutoff consisted of a three inch copper cylinder with a heat shield of 0.5 inch graphite disc, two layers of carbon cloth and a disc of five mil molybdenum sheet which faced the billet. To shorten the cutoff loading operation, which was formerly carried out by hand, the cutoff assembly was fastened by a graphite pin to a dummy block which was pinned to the stem.

Eleven billets with various lubricants were tried, involving two glass variations. Ram speeds employed were from 15 to 4 in./sec.; intentional upset delay times between "zero" and five seconds and extrusion reductions were 22:1. Upset pressures were about 82 ± 6 tsi, and running pressures about 70 ± 5 tsi, corresponding to extrusion constants of about 27 and 22 tsi, respectively. The temperature was increased from 3400°F to 3450°F in the cases where the ram speed was slower or the upset time was longer. These conditions are listed in Table 9.

c. Results

Before examination each nib required some degree of preparation. None of the rods cleared the dies as intended, and it was necessary to press a short, extruded section backwards through the die. To remove adherent glass and graphite, the dies were vapor blasted with a mixture of approximately one to one 200 mesh silica and 300 mesh alumina for about thirty seconds. The land diameter was measured for comparison with the original size. Rods were measured at four intervals from front to rear in two planes and examined for surface quality. The dies are as rated in Table 9 in terms of their change in size resulting from extrusion, and their ability to produce a rod of uniform diameter.

Even though the conditions of temperature, speed and lubrication were not identical, the performance of the dies can be readily evaluated. In general, the expectation that materials which behave well in steel extrusion do likewise for TZM extrusion appears to be borne out. A marked exception can be found in the use of tungsten. At the slower speeds, tungsten becomes ductile, possibly due to greater heating by the billet, and closes in. The TiB_2, TiB_2-Al_2O_3, and W-Ru dies are rated "good"

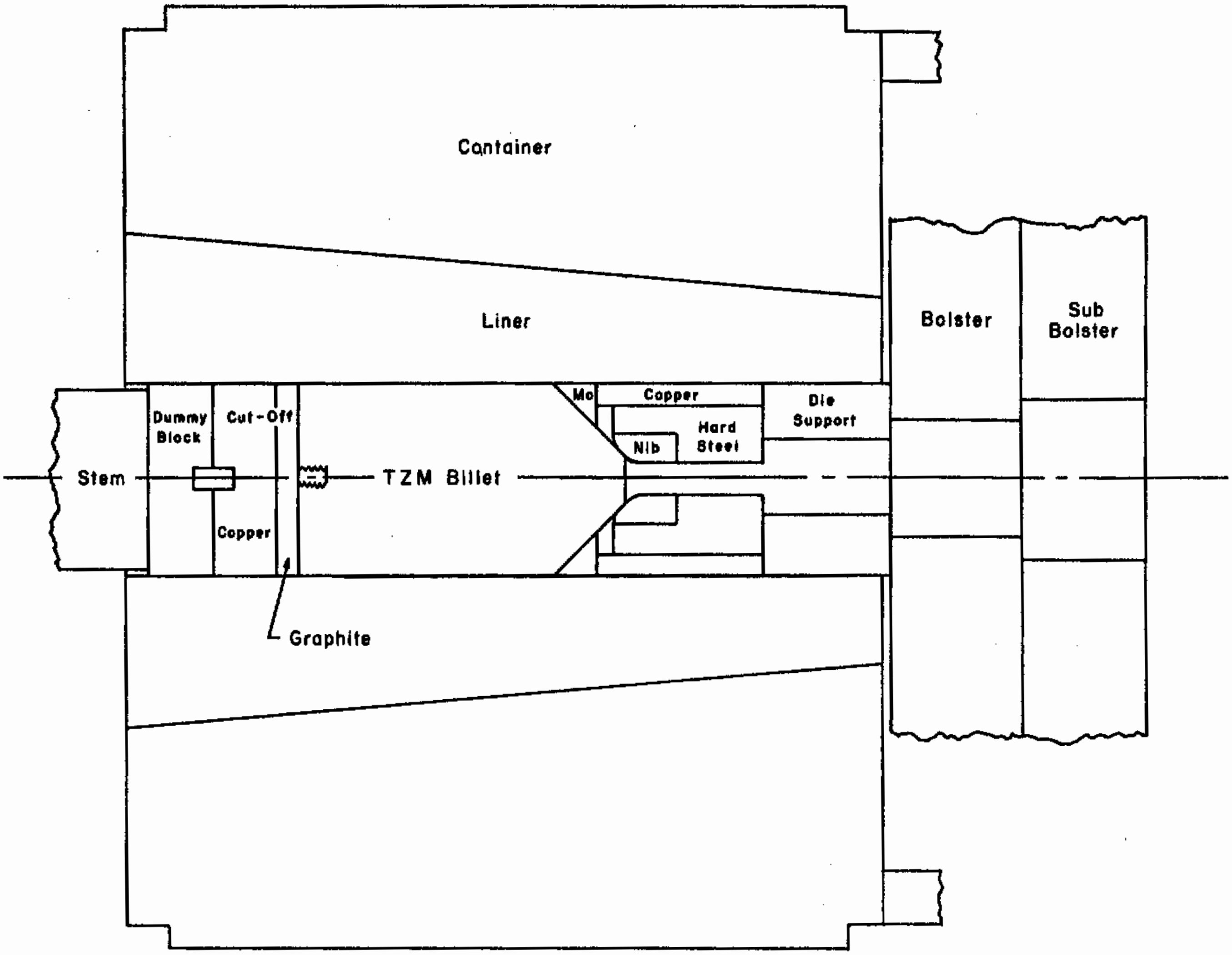


Figure 46. Tooling for Testing Round Dies with TZM Billets.

After assembly of the die support, die and Mo cone, a glass powder preshape is placed in the liner. The billet is released from the furnace and falls to the loader (Figure 39). Billet, cutoff, and dummy block are pushed into the liner for extrusion.

TABLE 9

ROUND DIE TESTS WITH TZM BILLETS

Die					Billet			Rod	
Composition	Relative Density (%)	Diameter Increase (mils)	Cracks	Performance Rating	Extr. Speed (in./sec.)	Lubr. Glass	Temp. (°F)	Maxium Variation from initial Die Size (mils)(1)	Surface Condition
W (sintered)	95	-118	None	Poor	15	1	3400	-111 + 0	Poor
W (sintered)	95	-125	None	Poor	13	1	3400	-114 + 0	Poor
W (sintered)	95	-260	None	Poor	7	1	3400	---	Stall(3)
Si_3N_4-M_gO	94	+ 11	Many	Fair	4	1	3400	- 2 + 9	Poor
$ZrSiO_4$-Wf(2)	92	+ 96	Several	Poor	6	1	3400	- 3 +88	Poor
ZrO_2	96	- 0	Many	-	5	1	3400	---	Stall(4)
TiB_2	98	+ 2	Many	Good	6	1	3450	- 1 + 3	Fair
TiB_2-Al_2O_3	95	+ 3	Many	Good	5	5	3450	- 2 + 2	Fair
W-Ru	99	- 4	Many	Good	7	5	3450	- 5 + 0	Good
Ta-Ru	99	- 8	Many	Fair	6	5	3450	- 7 + 0	Good
W-Ta	98	- 71	Several	Poor	5	5	3450	- 75 + 0	Good

Other Conditions:

Liner

Temp. = 900°F
Dia. = 3.545 in.
Lube. = Necrolene
Condition = Some scores

Die

Diameter = 0.750 in. (nominal)
Temp. = 200°F (estimated)
Lube. = Pad Glass 4 except the last three dies which had no pad.

Billet

Cutoff = 3 in. Cu 5 in. Gr
Reduction = 22:1
Diameter = 3.375 in.
Length = 8 in. + 90° Nose

Pressure

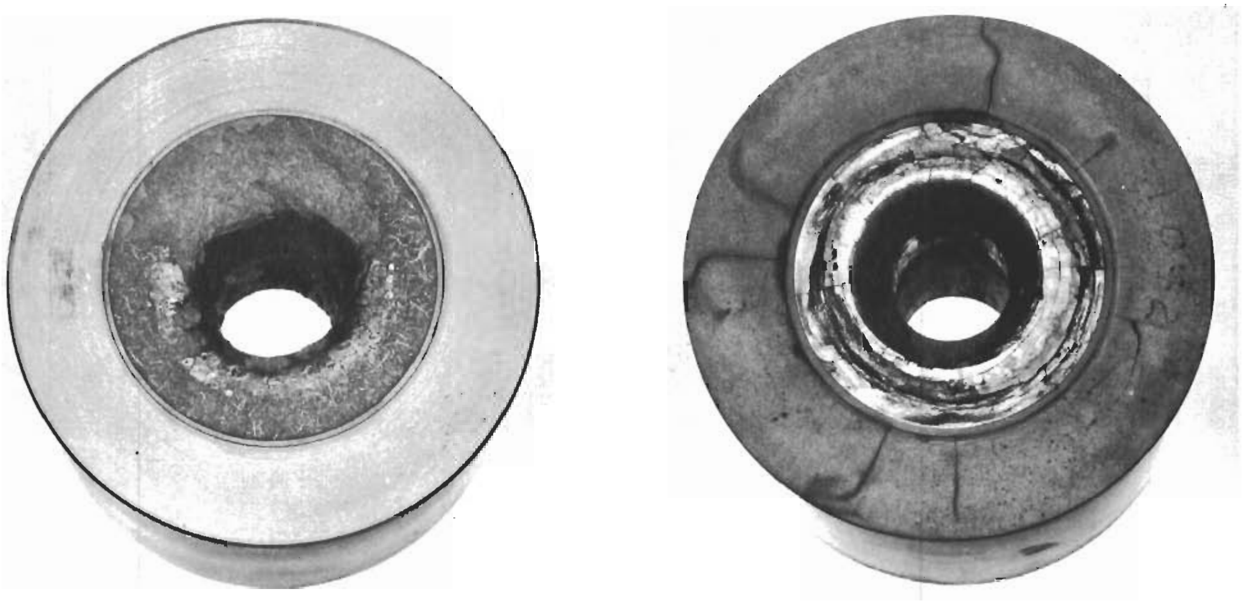
Upset = 82 ± 6 tsi
Running press. = 70 ± 15 tsi
Upset k = 27 ± 2 tsi
Running k = 25 ± 5 tsi

(1) Diameters larger than the die size are indicated (+), smaller as (-).
(2) Wf = W fibers.
(3) Slow ram speed caused die to soften and close in.
(4) Five-second delay in extrusion.

Si_3N_4-MgO and Ta-Ru are rated "fair"; $ZrSiO_4$-W (fibers) and W-Ta are rated "poor". The TiB_2 and TiB_2-Al_2O_3 nibs, which had theoretical densities of 98 and 95 percent, respectively, showed less wear than the Si_3N_4-MgO and $ZrSiO_4$-W fiber nibs, which had about 93 and 92 percent theoretical densities. (The compressive strengths of the first group compared to the second group would probably be higher even if all had the same relative density.) The 96 percent dense zirconia dies used in the lubrication described in Section E-1 above were found to resist wear even after two extrusions of TZM. These facts emphasize the importance of the die fabrication conditions of these materials. The ceramic dies appear in Figure 47 and the metal dies in Figure 48.

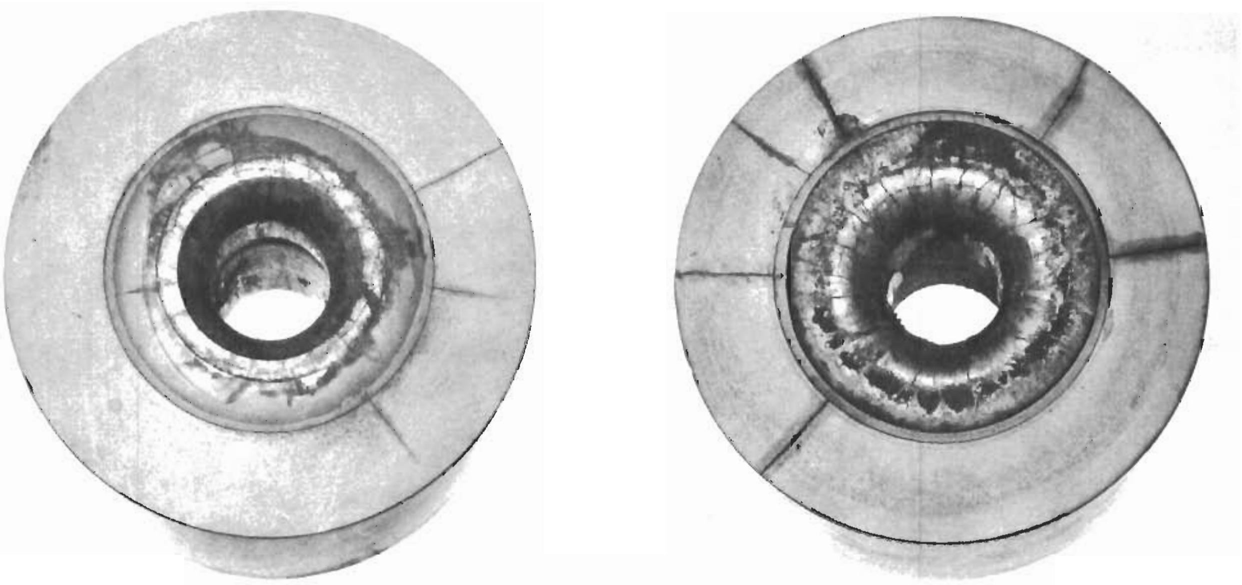
The refractory metal alloys, W-5 Ru, Ta-6 Ru and W-25 Ta, performed in similar fashion; each die was smaller after use than before. In the case of the ruthenium - bearing alloys, this amount was small, being four mils for the tungsten and seven mils for the tantalum alloy. The W-25 Ta, however, closed in 71 mils. These dies showed a few more cracks than were present before extrusion.

On the basis of these results it was apparent that W-Ru and Ta-Ru alloys had good potential for development as Tee dies. However, the cost of ruthenium and the difficulty of making ruthenium alloys reduced the applicability of such alloys for extrusion dies. Limitations of time and funds disqualified them for additional testing in this program. At least sixteen die materials were developed, which as rounds have adequately withstood steel and /or TZM extrusions and are, therefore worthy of testing as Tee dies. These include: W-6 $^{w}/o$ Ru, Ta-5.5 $^{w}/o$ Ru, TaC-Ni, TaC-W, ZrC, ZrB_2, and ZrO_2, TiB_2, $MoSi_2$-20 $^{v}/o$ Al_2O_3, ZrB_2-20 $^{v}/o$ Al_2O_3, TiB_2-20 $^{v}/o$ Al_2O_3, and Mo-Al_2O_3-SiO_2. Because of its similarity with ZrC and TiB_2, TiC was also to be tested. In addition, composites of $ZrSiO_4$ and Si_3N_4-5 $^{v}/o$ MgO, each with 20 $^{v}/o$ W powder and 20 $^{v}/o$ W fibers, were to be tested.



$ZrSiO_4$-W fibers

TiB_2-Al_2O_3

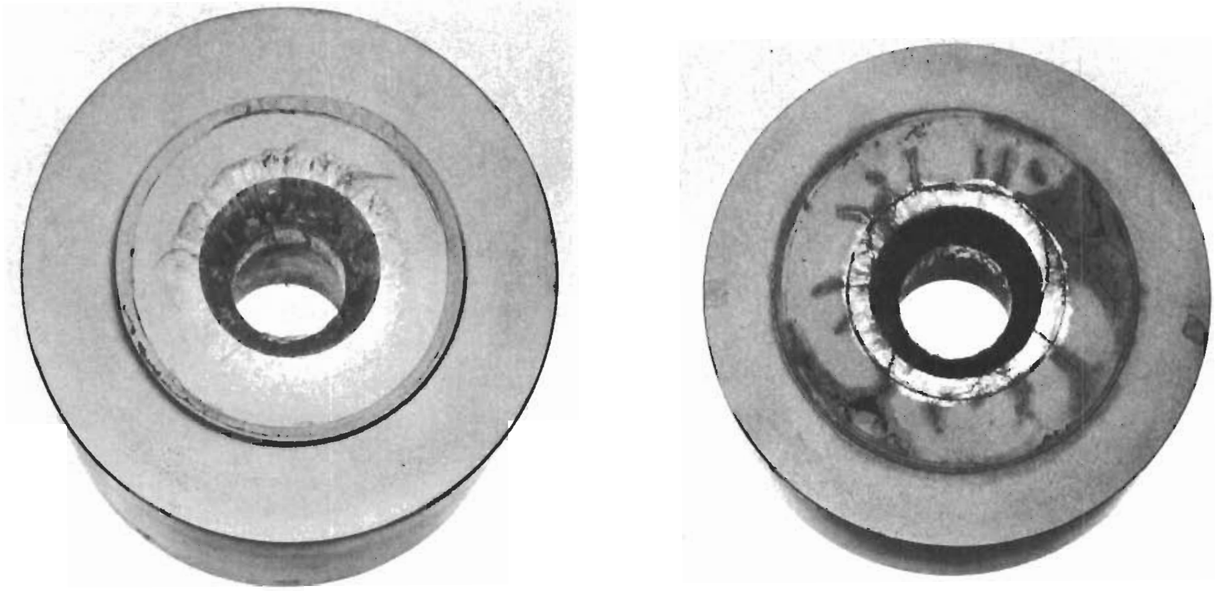


TiB_2

Si_3N_4-5 $^v/o$ MgO

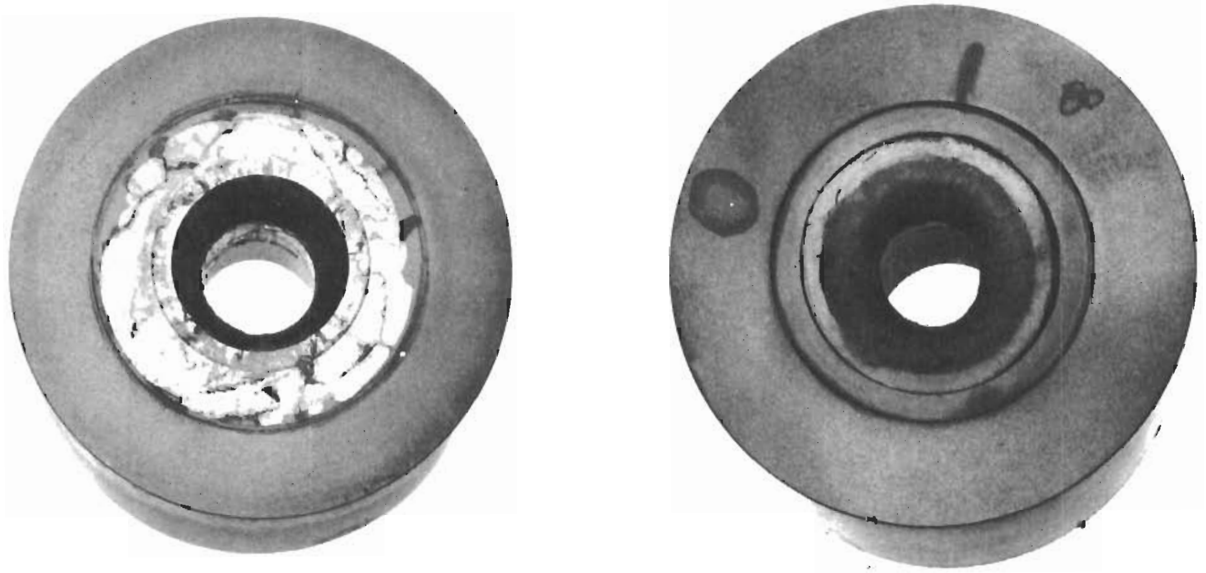
Figure 47. Round Ceramic-Based Dies after TZM Test.

Although the ram speeds and lubrication were different for these nibs, it is apparent that the denser or harder materials (TiB_2, TiB_2-Al_2O_3) performed better than the others. The tungsten dies used in this series (not shown) closed in and caused stalling of the billet at the slower ram speeds.



W-25 Ta

Ta - 6 Ru



W - 5.5 Ru

W

Figure 48. Round Refractory Metal Dies after TZM Test.

The W - 5.5 Ru and Ta - 6 Ru show considerable promise as dies for high temperature billets. The tungsten die shows considerable deformation (closing-in). Most of the cracks seen in the ruthenium-bearing dies were present before use. The irregular circular outline near the outside diameter of the W-Ru nib delineates the boundary of the Mo canning used in the forging step of the fabrication of the nib.

F. Testing of Ceramic Tee Dies with TZM Billets

1. Purpose

Extrusion of TZM molybdenum alloy billet through ceramic based shaped dies was designed to demonstrate that dies of this type, represented by specific materials, could endure the extrusion conditions and produce smooth, non-striated and uniform Tee extrusions. Prior to testing, equipment modification and die preparation was necessary.

2. Conditions

a. Modification of Equipment

Two billet heating furnaces and three lubricating facilities were used in this segment of the work. One furnace shown in Figure 49 was modified from the design of the other furnace, shown in Figure 38.

The former included a "high" vacuum induction furnace, a curved steel chute, and an enclosed argon purged box enclosing a "glassing" table. Although the billet loading times were increased to about twenty seconds compared to five seconds in the former design, the improved furnace atmosphere and enclosed glassing table were expected to yield the advantage of improved lubrication. Instead of releasing the billet with a mechanical latch, as in the old design, the billet, held in place by atmospheric (argon) pressure, was released by a gate valve. Zirconia insulation, a carbon pedestal, and tantalum wafer (to prevent billet-carbon interaction) were copied from the former design.

Although some successful heats were obtained in this furnace, difficulties were encountered. To expedite the course of the experimental work the earlier furnace (Figure 38) was also used. However, this equipment was modified so that the chute of the modified furnace, instead of delivering the billet to the liner as before (Figure 38) passed it to the top of an inclined glassing table as shown in Figure 50. The billets were rolled down over a layer of glass powder in the open air in contrast to the technique used with the vacuum furnace which used an argon purged box. Some billets were heated in the older furnace (Figure 38), covered (not rolled) in glass and extruded (Test Procedure A). Some billets were heated in the modified version of this furnace (Figure 50) and rolled in glass in air and extruded (Test Procedure B). Others were heated in the vacuum furnace and rolled in glass under argon and extruded (Test Procedure C).

b. Preparation of Dies

A series of dies was fabricated (see Section C-4) to conform to the design shown in Figure 25. The materials intended for

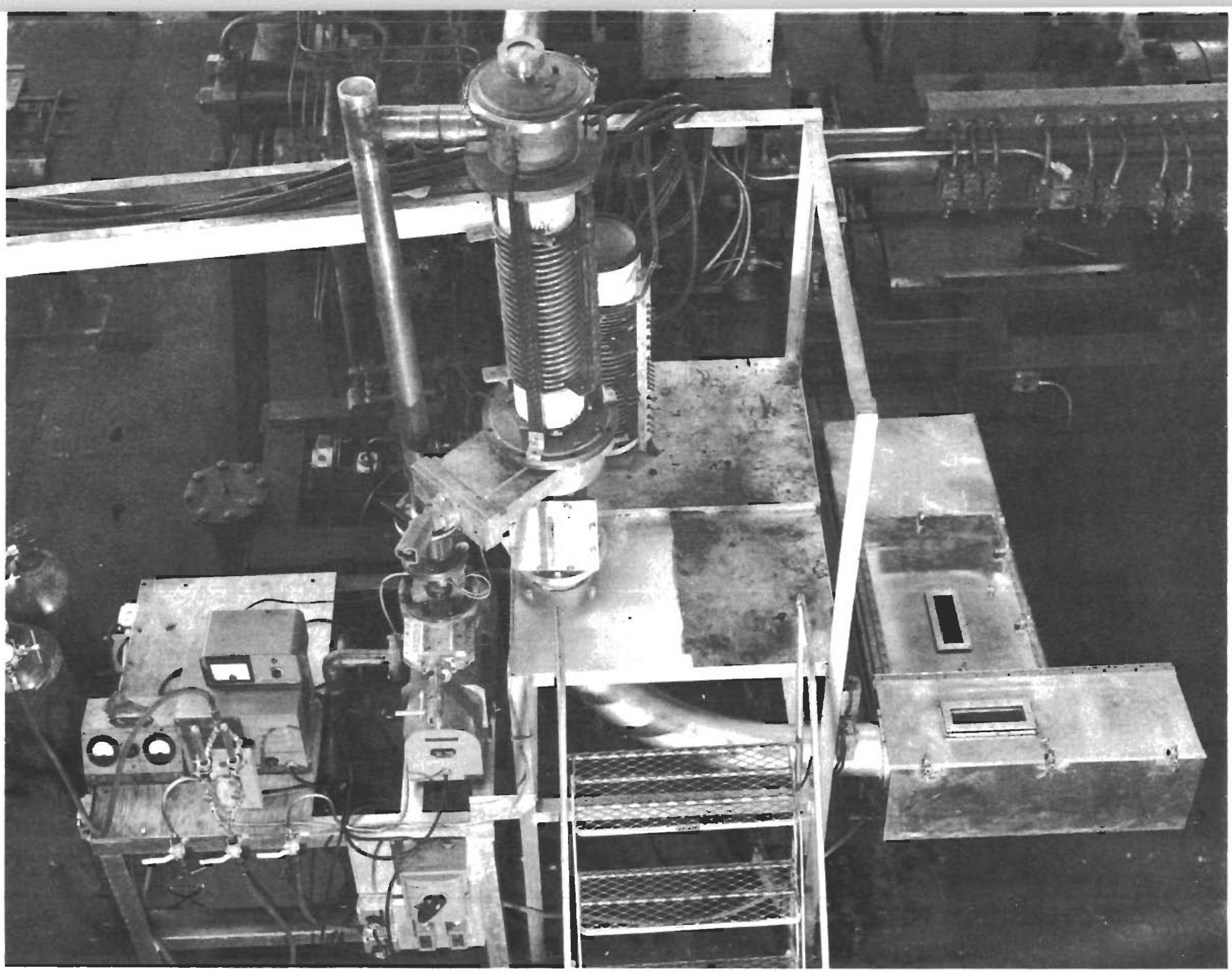


Figure 49. Vacuum Heating and Glassing Apparatus (in Argon).

Vacuum induction furnace at top is connected by means of a gate value and steel chute to argon-filled glassing table at lower right. The design is a modification of the furnace used previously (Reference 5).

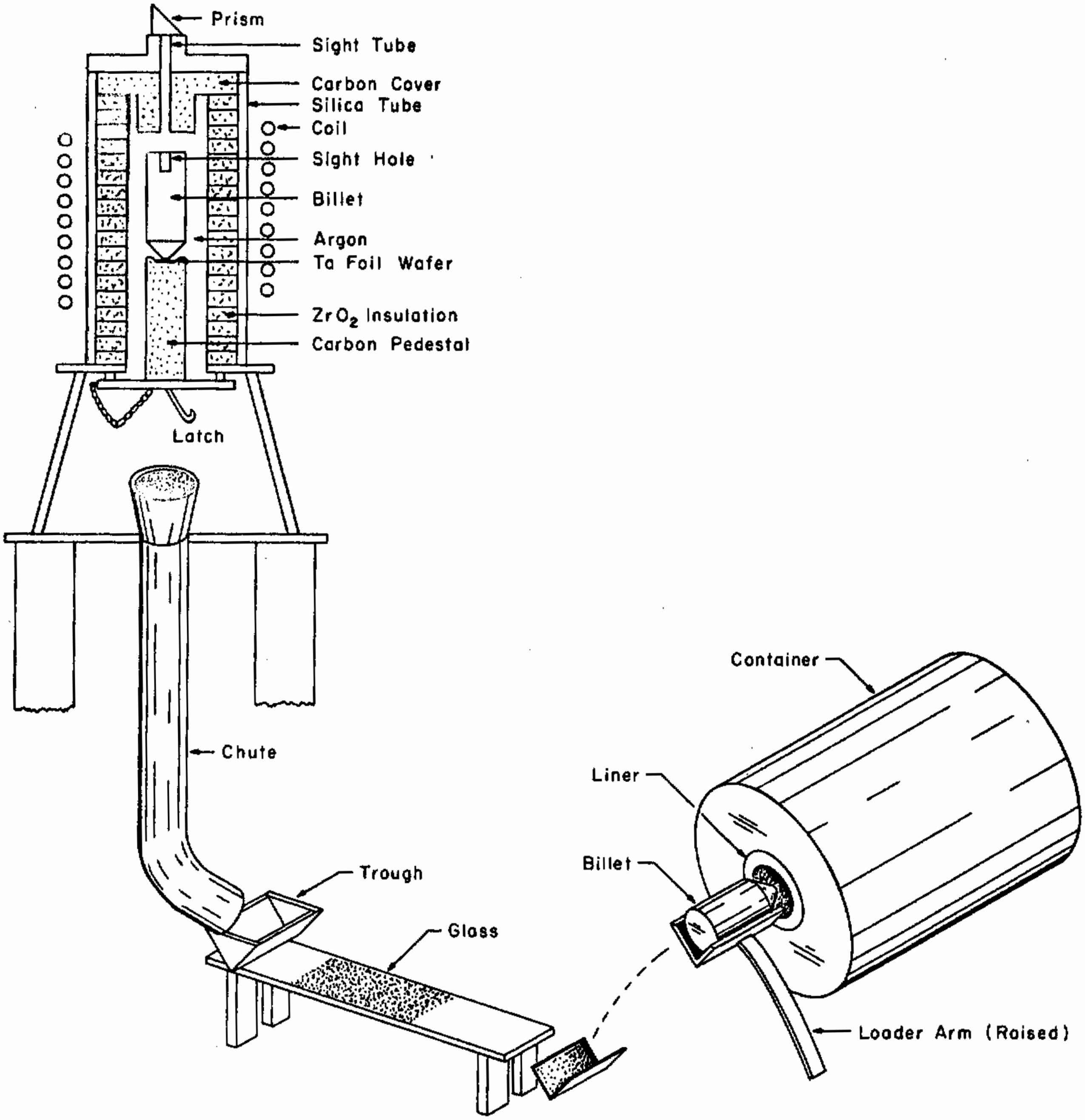


Figure 50. Argon Heating and Glassing Apparatus (in Air).

Release of the latch causes the billet to free fall down the chute and into the trough, from which it rolls over powdered glass on an inclined table to the loader. It is then lifted by the loader in front of the liner and pushed through the die by the stem.

testing, mode of fabrication, and finishing procedure, are shown in Table 10. In general, the Tee-shaped hole in the electrically conductive materials (the borides and carbides) was made by commercial electrospark discharge machining. The low expansion materials, the silicate and nitride composites, were hot pressed to "size" and no extensive finishing was required. After minimal grinding, the dies were assembled into steel die supports or casings with about a three mil shrink fit. An aluminum ring about 0.1 inch thick was present between the steel support or casing and ceramic die. (After die testing, the ring was to be dissolved in sodium hydroxide, freeing the nib for evaluation and the casing for another nib.) For additional support, a steel collar was shrunk around the casing to complete the assembly. The die support assembly and other extrusion tooling are shown in Figure 51. A Tee-shaped steel cone of cast H-13 tool steel, coated with 0.100 inch of stabilized ZrO_2 was used to prevent leakage of metal between the liner and the die holder, as shown in Figure 52. For most of the dies (Testing Conditions B and C, Table 11) a collar of steel was shrunk around the casing and an additional four-inch thick shaped bolster was used to ensure sufficient support for the nib and casing. For some dies (Testing Conditions A, Table 11) this was not used and casing failure occurred with the $MoSi_2$-Al_2O_3 die. Nibs prior to testing are shown in Figures 29, 30 and 31.

c. Performance of Tests

Billets were heated to 3400°F (Test Procedure A) and 3700°F (Test Procedures B and C), lubricated and extruded at 5 to 14 in./sec. as shown in Table 11. Upset and running pressures of 70 ± 20 and 60 ± 10 tsi, respectively, were obtained. This corresponds to upset and running extrusion constants of 28 ± 1 and 23 ± 3 tsi respectively.

3. Results

The results of the tests are listed in Table 11. Good extrusions and die performance were found with TiB_2-Al_2O_3, TiC and ZrO_2. Fair extrusions and die performance were found with Si_3N_4- 40 $^{v}/o$ W (20 $^{v}/o$ W powder + 20 $^{v}/o$ W fibers). Poor extrusions and die performance were found with $ZrSiO_4$-20 $^{v}/o$ W powder, $ZrSiO_4$-40 $^{v}/o$ W (20 $^{v}/o$ W fibers + 20 $^{v}/o$ W fibers), Si_3N_4-20 $^{v}/o$ W powder, TaC-W and ZrC. The silicate and nitride composites were at low theoretical density (93% or less) and lacked sufficient strength to resist wear. The heavily worn $ZrSiO_4$-20 $^{v}/o$ W powder die rated as poor, is shown in Figure 53 and the Si_3N_4-20 $^{v}/o$ W powder + 20 $^{v}/o$ W fibers rated as fair is shown in Figure 54. The ZrC and the TaC-W dies probably gave poor performance because they severely fractured at upset and partly broke apart, probably due to high residual stresses resulting from hot pressing. (The ZrB_2-Al_2O_3 die broke in Eloxing due to these stresses.) A front to rear comparison of their respective extrusions shows that the die material suffered little wear even though sharp protuberances of fragments were present. Since the properties of ZrC and TiC are similar (TiC is harder and ZrC is stronger) and a similar die performance was expected, the failure of ZrC due to fabrication history is more likely than failure due to an inherently poorer die material.

TABLE 10

CERAMIC TEE DIES PREPARED FOR TESTS WITH TZM BILLETS

Composition (v/o)	Diameter (in.)	Fabrication	Finishing	Status
ZrO_2	3.4	Sintered (1)	Ground	Tested
TiC	3.4	Hot Pressed	Eloxed	Tested
TiB_2	3.4	Hot Pressed	Eloxed	Broke in assembly (2)
ZrC	3.4	Hot Pressed	Eloxed	Tested
TaC-W	3.4	Hot Pressed	Eloxed	Tested
$ZrSiO_4$-20W	3.0	Hot Pressed	As pressed	Tested
$ZrSiO_4$-40W	3.0	Hot Pressed	As pressed	Tested
Si_3N_4-20W	3.0	Hot Pressed	As pressed	Tested
Si_3N_4-40W	3.0	Hot Pressed	As pressed	Tested
Si_3N_4-50W	3.0	Hot Pressed	As pressed	Abortive test (3)
ZrB_2-Al_2O_3	3.0	Hot Pressed	Eloxed	Broke in Eloxing (4)
TiB_2-Al_2O_3	3.0	Hot Pressed	Eloxed	Tested
$MoSi_2$-Al_2O_3	3.0	Hot Pressed	Eloxed	Abortive test (5)

(1) Pressed and sintered.

(2) Cracked during shrinking and broke.

(3) Billet stalled due to slow load.

(4) Residual stresses in compact caused it to crack open.

(5) Die broke during upset when the casing shattered.

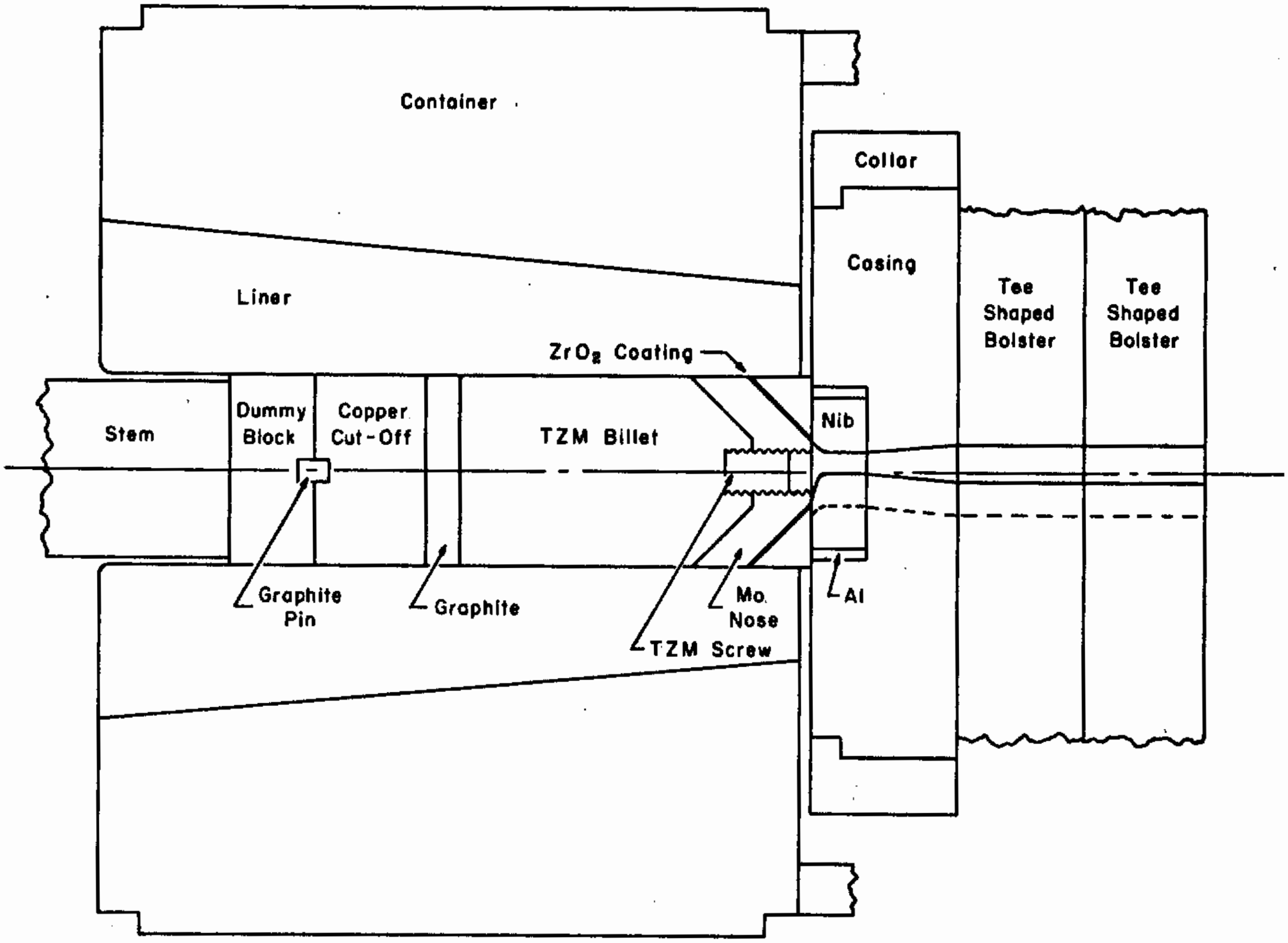
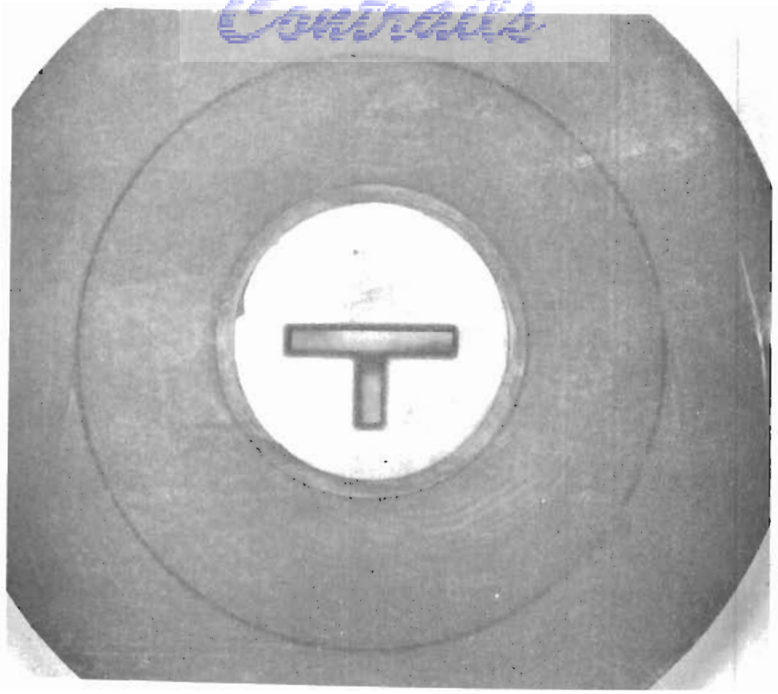


Figure 51. Tooling for Testing Tee Dies with TZM Billets.

Two shaped bolsters and a collar (shrunk around the casing) were used to gain greater support for the nib. (Failure of the casing had occurred while testing the $MoSi_2$-Al_2O_3 die without one bolster and the collar). A ZrO_2 coated steel cone with Tee-shaped orifice prevented the leakage of metal between the nib and the liner. A sintered molybdenum nose was used to reduce the upset force, in test procedure B and C, while none was used in test procedure A (see Table 11). For the ZrO_2 and TaC-W dies aluminum-graphite rather than copper-graphite cut-offs were used.



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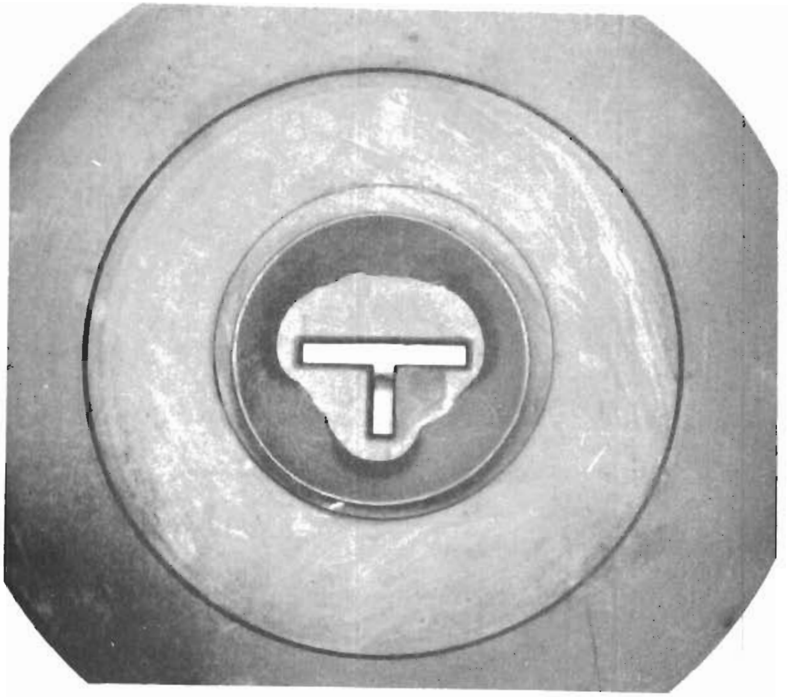

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Figure 52. Cone and Support for Ceramic Tees.

The assembly at the top is the ZrC die within its steel casing which has been shrunk into the steel collar. The same assembly is shown at the bottom with the ZrO_2 coated cone in place.

TABLE 11

CERAMIC TEE DIE TESTS WITH TZM BILLETS

Conditions				Results$^{(1)}$									
Die				Die		Extrusion							
Composition v/o	Density (%)	Testing Conditions	Ram Speed (in/sec)	Wear$^{(2)}$ (in)	Rating	Front			Rear			Overall Quality
						Rt Fl	Lt Fl	St	Rt Fl	Lt Fl	St	
$ZrSiO_4$-20 W_P	93	A	14	.043	Fair	G	G	T	W	WS	WS	Poor
$ZrSiO_4$-40 $W^{(3)}$	92	A	5	.071	Fair	G	G	G	W	WS	WS	Poor
Si_3N_4-20 W_P	93	A	7	.053	Fair	G	R	T	G	WS	WS	Poor
Si_3N_4-40 $W^{(3)}$	93	A	6	.028	Fair	T	G	G	WS	R	R	Fair
TiB_2-20 Al_2O_3	95	B	8	.012	Good	G	G	G	G	G	TS	Good
TaC-W	--	C	12	--	Poor	FS	FS	FS	FS	FS	FS	Poor
ZrC	94	B	12	--	Poor	FS	FS	FS	FS	FS	FS	Poor
TiC	96	B	11	.007	Good	G	G	G	G	G	G	Good
ZrO_2	95	C	10	.008	Good	G	G	G	G	G	G	Good

(Table continued on next page).

(1) Rt = Right G = Good R = Rough
Lt = Left T = Torn S = Striated
St = Stem W = Worn (or washed) F = Fracture (of die) Fl = Flange

(2) Maximum wear measured at radii of intersection of stem at flange.

(3) 20 $^v/_o$ W powder + 20 $^v/_o$ W fibers.

TABLE 11 (continued)

Other Conditions

Billet

Temperature = A-3400, B and C-3700°F
Diameter = 3.400 inches
Cutoff = Cu-Gr
Length = A-9, B-10, C-12 inches
Nose = Sintered Mo in B and C
Lubrication = A - covered with Glass 5
B - rolled in Glass 7 in air
C - rolled in Glass 7 in argon
Heat time = 14 min.
Soak time = .5 min.
Heat atmos. = Argon except vacuum in C
Unextruded = .50 inches

Die

Support = A - no collar
B and C - collar
Reduction = 14:1
Temperature = 200°F estimated
Lubrication = A - Glass Pad 4
B and C - Glass Pad 4 plus
preform of Glass 2

Extrusion

Upset Force = 700 ± 200 tons
Upset Pressure = 70 ± 20 tsi
Upset Constant = 28 ± 1 tsi
Running Force = 600 ± 100 tons
Running Pressure = 60 ± 10 tsi
Running Constant = 23 ± 3 tsi
Length = A -126 in.
B -140 in.
C -168 in.

Liner

Diameter = 3.545 in.
Temperature = 900°F
Condition = Fair
Identity = 34-A
Load Time = A - 5 sec.
B and C - 15 sec.
Lubrication = Oil dag

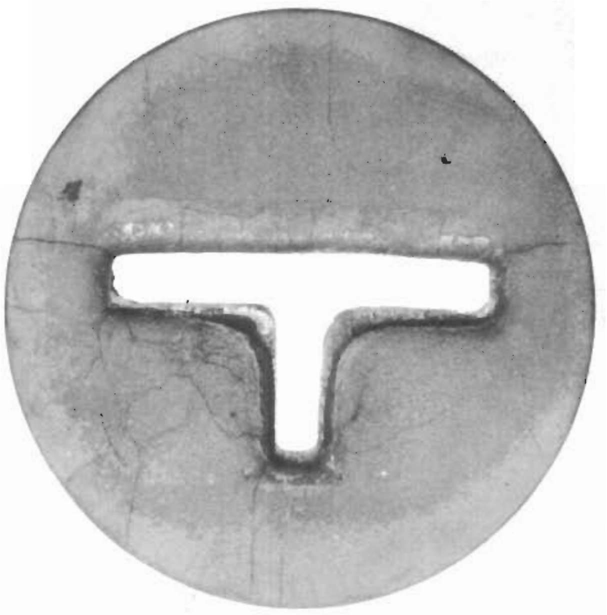


Figure 53. Heavily Worn $ZrSiO_4$-W Powder Die. Magnification: about actual size.

This material, only 93% dense, was rated as poor. Presumably a denser body would yield better performance.

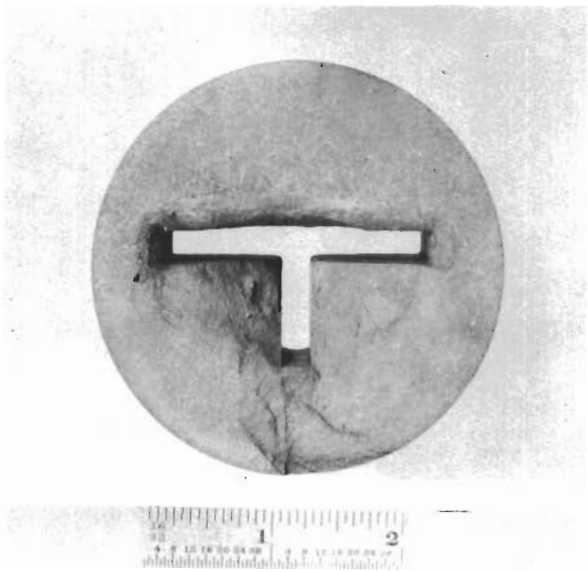


Figure 54. Tested Si_3N_4 - 20 $^v/o$ Powder - 20 $^v/o$ W Fibers.

This die with 93% theoretical density was rated as fair. Optimization of die fabrication parameters and increases in theoretical density may cause better performance.

A comparison of section sizes of the four best dies before and after extrusion, and a front to rear comparison of the dimensions of their respective extrusions are shown in Table 12. The TiC and ZrO_2 dies were found to wear the least. Their extrusions indicated their good performance with a front to rear dimensional run-out of 0.003 inch each over a 140 and 168-inch length respectively. Although there were local areas of tearing, the surfaces of the Tee bars from the ZrO_2 and TiC dies were at least 63 rms at the front and rear. The local tears were believed to be caused by imperfect lubrication and/or extrusion speed and not inadequate die performance. The radius of .063 inch at the intersection of the flange and stem of the Tee was maintained from front to rear of the extruded Tee bar. The ZrO_2 and TiC dies after extrusion are shown in Figures 55, 56 and 57. Representative cross-sections from front and rear of the extrusion from ZrO_2 and TiC dies are shown in Figures 58 and 59 respectively.

TABLE 12

DIMENSIONS OF CERAMIC TEE DIES AND TEE BARS(1)

(Dimensions are in inches)

Composition	Theo. Den. (%)	Tee Die								Tee Bar						
		Size Before				Size After				Station	Position				Surface Smoothness (rms)	Length (in)
		Lt Fl	Rt Fl	St	Rs	Lt Fl	Rt Fl	St	Rs		Lt Fl	Rt Fl	St	Rs		
$ZrSiO_4$-W_p	93	.249	.250	.219	.062	.285	.261	.248	.158	Front Rear	.260 .280	.262 .253	.240 .284	.062 .158	62 125-250	126
TiB_2- Al_2O_3	94	.252	.255	.250	.062	.260	.254	.262	(2)	Front Rear	.263 .260	.260 .261	.259 .259	.062 .125	62 62-125	140
TiC	95	.260	.260	.260	.062	(3)	-	-	-	Front Rear	.250 .248	.254 .241	.257 .258	.062 .075	$62^{(4)}$	140
ZrO_2	95	.260	.257	.265	.062	.260	.265	.261	(2)	Front Rear	.262 .265	.262 .265	.270 .270	.062 .075	$62^{(4)}$	168

(1) Rt = Right
Lt = Left
St = Stem
Rs = Radius
Fl = Flange

(2) Corner broke off when butt was removed from die.

(3) Butt stalled in die.

(4) Some local tearing due to nonoptimum extrusion conditions.

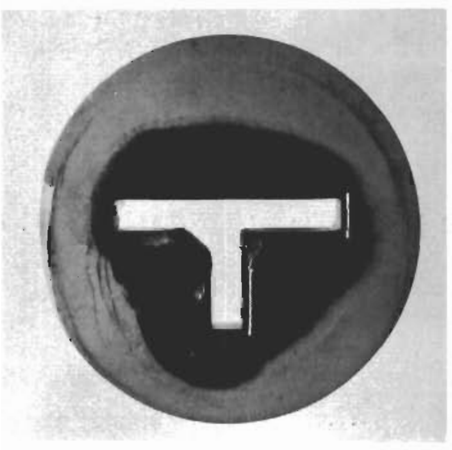


Figure 55. ZrO_2 Tee Die after TZM Test.

The dark color around the orifice of the die was caused by lubrication. When the unextruded butt was removed from the die a portion of the die was broken from the flange and stem intersection.

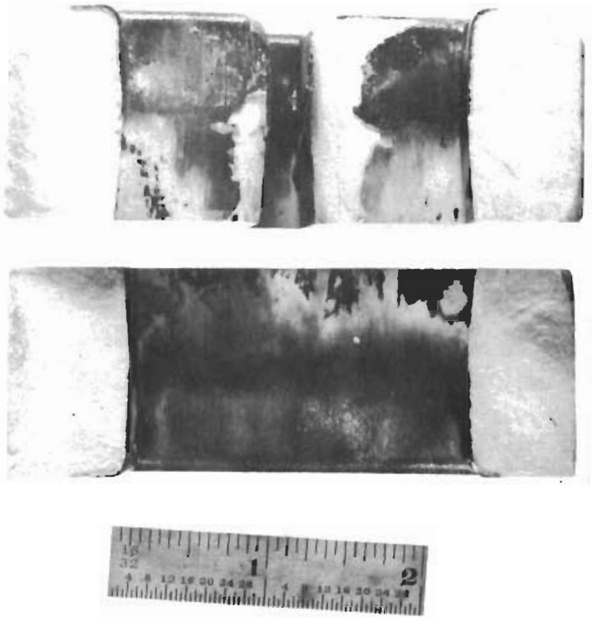


Figure 56. Internal View of Tested ZrO_2 Die.

The die broke in half during testing. The dark color is due to residual lubrication. The surfaces over which the metal pressed are about 63 rms, except for the portions which have spalled off.

Figure 57. TiC Tee Die after TZM Test.

The die broke when the unextruded butt was removed. A section of the extrusion remained in the die.

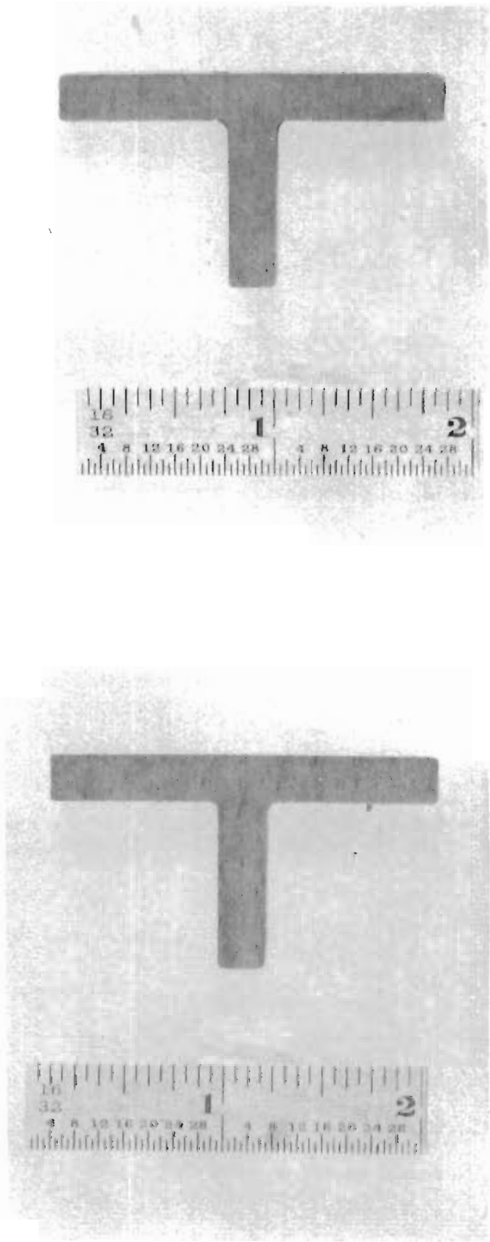


Figure 58. Sections of TZM Tee from ZrO_2 Die.

The section thicknesses and radii are maintained from front to rear as seen by comparisons between the front section (top) and rear section (bottom). Some glass is present on the extruded surfaces of the specimens.

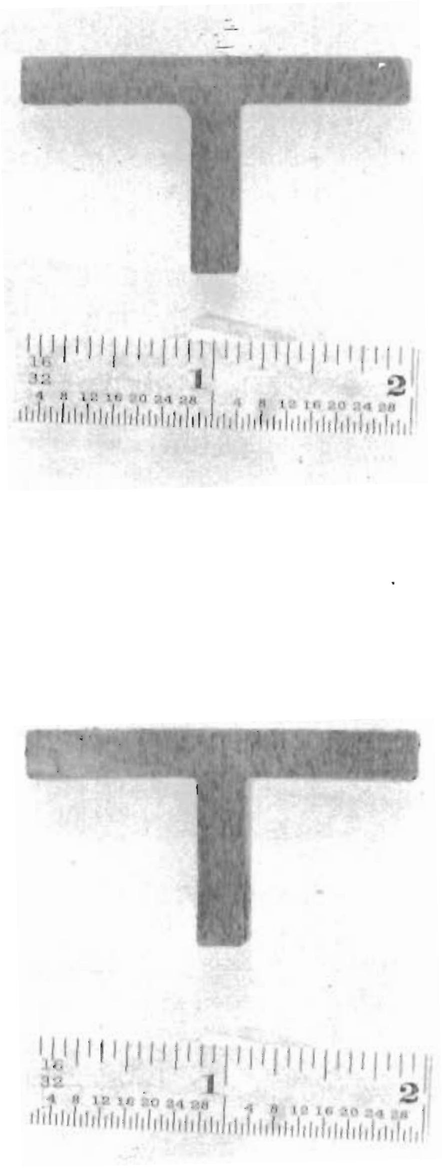


Figure 59. Sections of TZM Tee from TiC Die.

The section thicknesses and radii are maintained from front to rear as seen by comparisons between the front section (top) and rear section (bottom). Some glass is present on the extruded surfaces of the specimens.

G. Testing of Coated Dies with Steel Billets

1 Purpose

Instead of testing experimental dies under standard extrusion conditions, proven dies were used in an experiment to determine near optimum extrusion conditions for thin-wall Tees of 4340 steel. This scope change, in compliance with instructions from the Project Engineer, Mr. T. Felker, MATB, was based on his judgment that the solution to the problem of extruding thin sections of structural steels would be achieved by optimization of extrusion parameters, and extrusion practice.

The extrusion practice prescribed by the Project Engineer for this particular purpose involved the combination of techniques described in the following:

(1) The billets were to be heated rapidly in argon to avoid contamination. Oxide scale seriously disturbs the surface metal flow of the billet, and reacts or interacts with the glass lubrication. These factors lead to rough extruded surfaces.

(2) The billets were to be heated bare, that is, without glass coating. The glass lubrication coated on the billets prior to heating (as in Section D) tends to run off because the desired viscosity of the glass at the billet temperature is only 200 poise.

(3) After heating, the billets were to be rolled several revolutions down an inclined plane covered with a glass powder of a specific composition. Rolling coats the cylindrical surface of the billet uniformly; the composition of the glass and the billet temperature control glass viscosity.

(4) The billets were to be transferred within about 8 seconds to the extrusion liner. Short transfer times are required to minimize the loss of heat and stiffening of the billet.

(5) The liner was to be coated with oil-dag; glass wool pads and glass preform were to be placed ahead of the die. The oil-dag prevents sticking of the freezing glass on the liner surface and allows easy butt removal; the glass wool pad lubricates the front of the billet; and glass preform assumes a near conical shape, contributing to the ease of metal flow, and functions as a reservoir of glass during extrusion.

(6) Alumina-coated tool steel dies were to be used. Al_2O_3 resists the erosion of the extruding steel and reduces the passage of heat to the supportive steel substrate below its surface.

The extrusion parameters to be varied, as prescribed by the Project Engineer, were billet temperature and extrusion speed. Higher billet temperatures and low extrusion speeds lead to die failure, because of softening of the die. Low billet temperatures and low speeds lead to high extrusion stiffness and stalling, because of cooling of the billet. High speed extrusion also leads to the problem of decelerating the extrusion without deforming it. However, of primary interest to the Project Engineer was the effect of a given billet temperature and extrusion speed on the viscosity and extrusion behavior of the glass lubricant, which largely controls extruded surface quality.

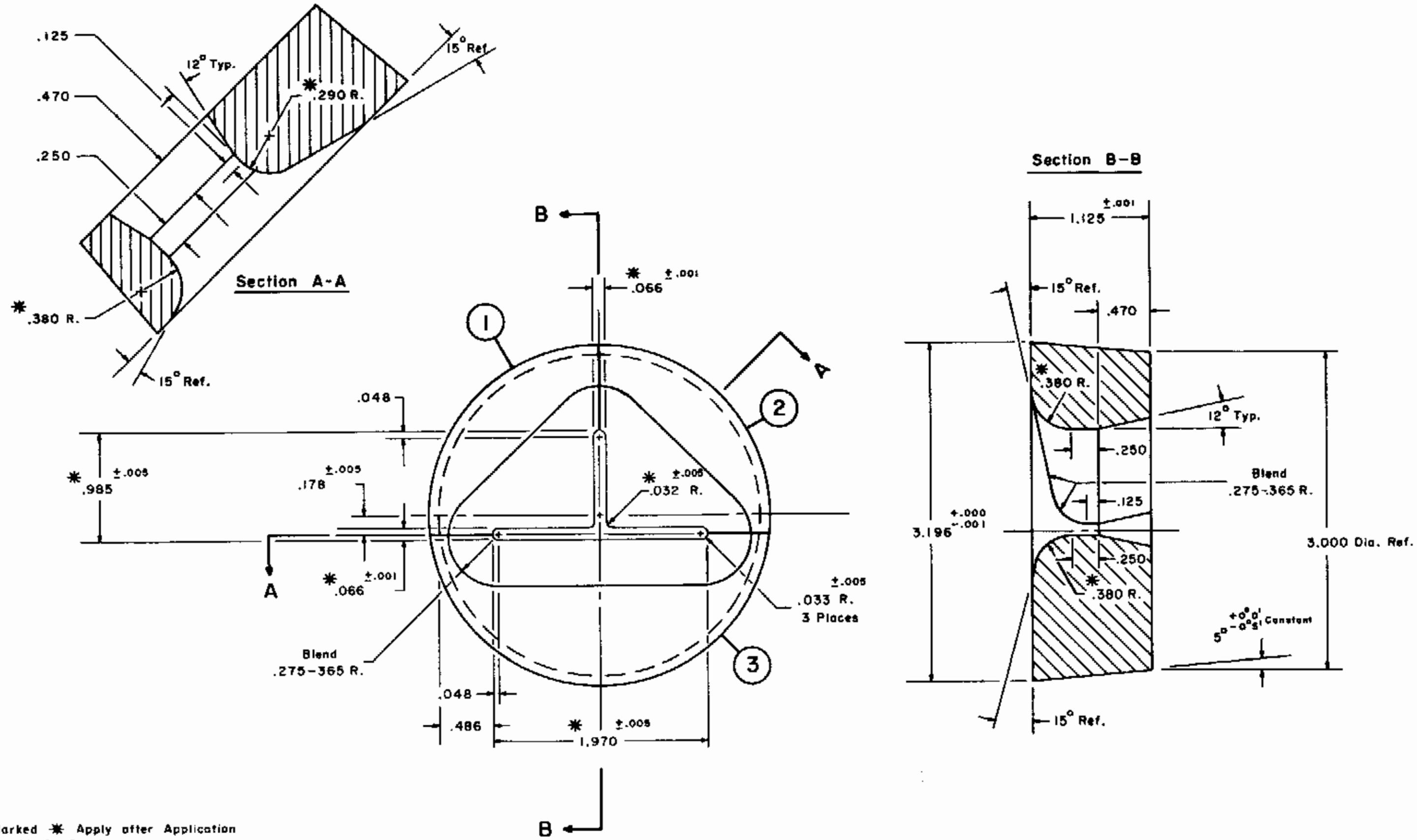
According to glass lubrication theory, the glass selected has a relatively low viscosity (essentially "liquid") at the temperature of the metal extruded, but exceedingly high viscosity (essentially "solid") at the temperature of the tools. As the metal billet is elongated in extrusion, all the shearing action in the glass takes place in the low viscosity or "liquid" layer of glass between the extruding metal and the layer of high viscosity glass.

The force of friction during the extrusion is the force required to shear the glass within the "liquid layer." This force is defined by the relation $F = \eta A v/T$ where F = force, η = viscosity of the glass, A = area of the surface lubricated, v = extrusion speed and T is the thickness of the liquid layer. The viscosity, η, changes with glass composition and billet temperature. The thickness of the shearing layer within the glass increases as billet temperature increases, since hotter billets will cause wider as well as steeper thermal gradients within the glass "liquid" layer.

The purpose of the 4340 steel extrusions programmed by the Project Engineer was to determine the range of billet temperature and extrusion speed which produces optimum glass lubrication behavior and consequent improved extruded surfaces for his particular application.

2. Conditions

The designs of the dies, which were segmented or split, were of two types, one with a long stem (1.63 inches) and one with a short stem (0.985 inch) as shown in Figure 60. The three segments were placed into a steel ring, and the resulting assembly was set into a casing similar to that used for the ceramic-based dies for tests with TZM billets. The billets used for this experiment were ten inches overall, including a 90° nose composed of unalloyed iron to reduce the upset force. Extrusion parameters varied, according to direction by the program's Project Engineer, were (1) billet temperature (2300°F to 1900°F) and extrusion speed (25.0 to 0.75 in./sec.). Other changes, such as the difference in die design (long and short stems), and number of glass preforms (one or two) are not presumed to be significant variables. Billets were heated to temperature in about six minutes in the furnace shown in Figure 50, under argon, and soaked



Note

Dimensions Marked ✱ Apply after Application of .015 inches Al_2O_3 Coating.

Figure 60. Design of Coated 0.062-inch Tee Dies.

The design shown is that of dies C, CC, DD, EE, FF, and GG. The design for 14, 18, 19, and 20 is not shown. It differed from the first design by having a 1.625-inch stem and the orifice of the flange set 0.410 inch below the center line of the die.

three minutes. Within three seconds of emerging from the furnace they were coated with glass by rolling down the inclined table covered with powdered glass. Within another eight seconds, they were upset in the liner. The extrusion conditions are shown in Table 13.

3. Results

Good extrusions were obtained at higher billet temperature (2300°F) and moderate speed (4 to 7 in./sec.). Higher and lower speeds at this temperature led to tearing or rippling of the flanges. Good extrusions were also obtained at lower temperature (2000°F) and slower speed (2.0 in./sec.). Extrusion speeds below 2.0 in./sec. even at 1900°F led to die failure. The use of one or two glass preforms did not contribute important differences in extrusion flow shape as revealed by comparison of extrusion butts shown in Figure 61.

Correlations exist between extrusion forces (pressures), uniformity of glass coverage and flange condition. Higher speeds (17 and 25 in./sec.) at the higher temperature (2300°F) induced higher pressures, produced extrusions with minimal glass coverage, particularly at the edges of the flanges, and rippled or torn flanges. Lower speeds (1.5 to 2 in./sec.) at this temperature induced lower forces and excessive glass coverages in local areas and rippled or torn flanges. Nonuniformity of glass coverage presumably led to rippling or tearing of the flanges, because medium speeds (2 to 7 in./sec.) at this temperature produced good extrusions with uniform glass coverage.

At lower billet temperature (2000°F) a good extrusion with uniform glass coverage was obtained at a lower speed (2 in./sec.) -- a speed which produced a poor extrusion at higher temperature (2300°F).

These results indicate, as anticipated by Mr. Felker, that the quality of extruded surface was controlled by (among other things) glass lubrication behavior, that this behavior was sensitive to extrusion speed, and that for a given extrusion speed a specific range of billet temperature was indicated.

The radii and thickness of the three best extrusions (those extruded through Dies 7C, 7FF and 14) were found to be uniform to within ± 0.002 inch over the entire length of the extrusion, 34 feet in one case and 42 feet in another; the 0.102 inch radius indicated by the die was maintained from front to rear of the extrusion. The general appearance of the extrusions is summarized in Table 14. Representative front and rear sections from the extrusions formed by Dies 7C and 14 are shown in Figure 62. In almost all areas the surface of these extrusions was found to be 16 rms or better in the longitudinal (extruded) direction, and 63 rms in the transverse direction. An oblique view of a typical section from the extrusion through Die 14 is shown in Figure 63. A front to rear measurement of section thickness from the middle third of a large stem extrusion (pushed through Die 7C) and short stem extrusion (pushed through Die 18) are shown in Table 15.

TABLE 13

TEE DIE EXTRUSIONS WITH 4340 ALLOY STEEL BILLETS

Conditions						Results (1,2)										
		Extrusion					Extrusion									
Die No.	Billet Temp. (°F)	Speed (in/sec)	Time (sec)	Pressure (tons)	K (3) (tsi)	Die (4) Hardness (R_c)	Front			Middle			Rear			Overall Quality
							Lt Fl	Rt Fl	St	Lt Fl	Rt Fl	St	Lt Fl	Rt Fl	St	
7EE	2300	17.0	0.6	57	15	27	G	G	G	T	G	G	T	G	G	Poor
7GG	2300	25.0	0.4	56	15	26	G	G	G	G	G	G	T	T	T	Fair
7C	2300	7.0	1.4	33	8.9	27	G	G	G	G	G	G	G	G	G	Good
7CC	2300	2.0	5.0	27	7.3	35	G	G	G	G	G	R	R	R	R	Poor
7DD	2300	1.5	6.6	32	8.9	29	G	G	G	G	T	G	T	T	G	Poor
7FF	2000	2.0	5.0	56	15	31	G	G	G	G	G	G	T	G	G	Good
14	2300	4.0	2.5	33	8.4	36	G	G	G	G	G	G	G	T	G	Good
18	1900	1.5	6.6	58	15	31	G	G	G	G	G	G	W	W	W	Poor
19	2000	0.75	13.0	50	13	30	G	G	G	W	W	W	W	W	W	Poor
20	1900	0.75	13.0	65	17	32	G	G	G	G	T	G	W	W	W	Poor

Other Conditions

Billet

Cutoff = 3 in. Cu-.5 in. Gr.
Reduction = 40:1 with No. 7 dies
50:1 with Nos. 14-20
Length = 9 in. + 90° Nose
Unextruded = .5 in.
Heating time = 7 min. (approx.)
Soaking time = 3 minutes
Heating atmos = Argon (flowing)
Lubrication = Glass 6
Material = 4340 steel plus iron nose
Diameters = 3.400 in.

Die

Temperature = 200°F (estimated)
Stem Length = 1.50 in. No. 7 dies
.875 in. Nos. 14-20
Coating = Al_2O_3
Type = External
Lubrications = 1. Aquadag with Glass 4 plus one glass preform for dies 7EE, GG, C, CC, DD and 14.
2. Aquadag with glass 4 plus 2 glass preforms for dies 7FF, 18, 19, 20.

Liner

Diameter = 3.545 in.
Temperature = 900°F
Condition = Fair
Identity = 34-A
Load Time = 8 secs.
Lubrication = Oil Dag

(1) G = Good, R = Rippled, T = Torn, W = Wash (die)
(2) Lt = Left, Rt = Right, Fl = Flange, St = Stem
(3) K = P/ ln R
(4) At intersection of flange and stem (coating removed) Hardness of die in zone unaffected by heat (back) was 50-52 Rc

Figure 61. Comparison of Extrusion Butts.

Although two glass preforms were used for the butt shown at left, and one glass preform for the butt shown at right, they have substantially the same shape.

TABLE 14

SUMMARY DIMENSIONS OF COATED DIES AND BEST STEEL TEES(1)

(Dimensions are in inches)

Die				Extrusion					
No.	Size Before			Station	Position			Surface Smoothness (rms)	Length (feet)
	Left Flange	Right Flange	Stem		Left Flange	Right Flange	Stem		
7C	.060	.062	.062	Front Middle Rear	.060 .060 .058	.063 .063 .061	.058 .060 .058	63-125 63-125 125-250	34
7FF	.054	.061	.061	Front Middle Rear	.050 .050 .055	.060 .060 .064	.065 .065 .067	63-125 63-125 63-125	34
14	.061	.059	.061	Front Middle Rear	.060 .055 .058	.059 .055 .055	.063 .057 .060	63-125 125-250 125-250	42

(1) The 102 in. radius at the intersection of the flange and stem was maintained from front to rear of the extruded length.

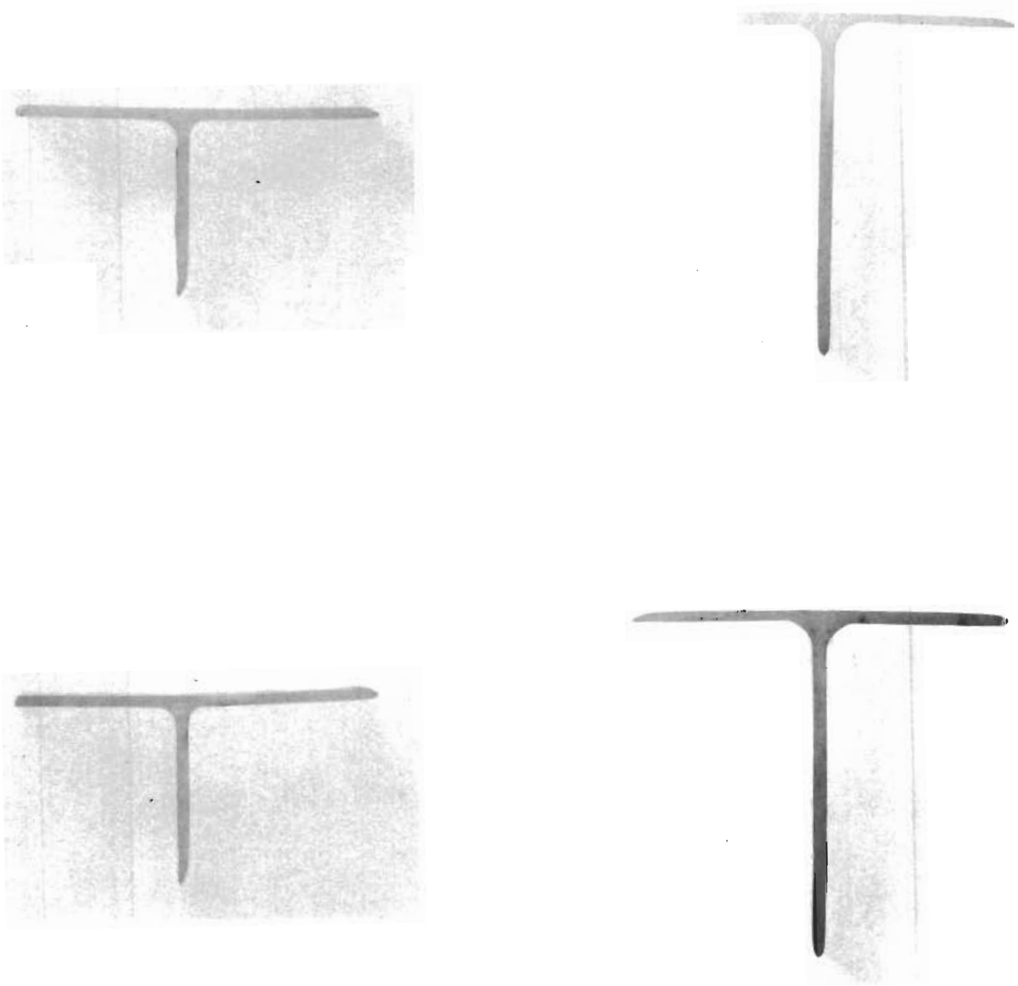


Figure 62. Representative Steel Sections through Coated Dies.

The section thicknesses and radii are maintained from front to rear as seen by comparison between the front section (top) and rear section (bottom).

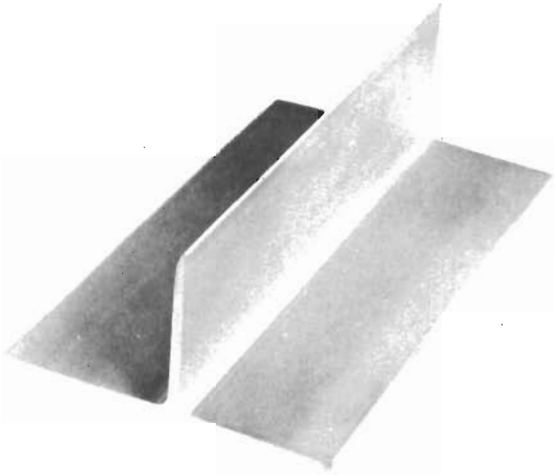


Figure 63. Representative Cropped Section of 4340 Steel Tee.

This section was extruded through Die 14 at 2300°F and 4 in./sec. ram speed.

TABLE 15

SECTION THICKNESSES OF TEES FORMED BY DIES 7C AND 18[1]

(Dimensions are in inches)

Tee Formed By Die 18				Tee Formed By Die 7C			
Distance From Front	Right Flange	Stem	Left Flange	Distance From Front	Right Flange	Stem	Left Flange
3 in.	.054	.057	.055	3 in.	.063	.062	.061
6	.054	.057	.055	6	.064	.062	.061
9	.054	.057	.055	9	.063	.062	.061
12	.054	.056	.055	12	.064	.062	.061
15	.054	.056	.055	15	.063	.062	.060
18	.062	.056	.055	18	.063	.062	.060
21	.054	.057	.055	21	.063	.062	.061
24	.053	.057	.055	24	.063	.062	.061
27	.055	.057	.055	27	.064	.062	.061
30	.055	.057	.055	30	.064	.062	.060
33	.055	.057	.055	33	.064	.062	.060
36	.055	.056	.055	36	.063	.062	.061
39	.054	.057	.054	39	.063	.062	.060
42	.056	.057	.054	42	.064	.062	.061
45	.055	.057	.055	45	.063	.063	.060
48	.055	.057	.054	48	.063	.062	.060
51	.055	.057	.055	51	.064	.062	.060
54	.055	.057	.054	54	.064	.062	.060
57	.054	.058	.054	57	.064	.062	.060
60	.054	.056	.055	60	.064	.062	.060
63	.055	.057	.054	63	.064	.061	.061
66	.054	.056	.055	66	.064	.061	.060
69	.054	.057	.054	69	.064	.062	.060
72	.054	.056	.054	72	.063	.062	.060
75	.054	.057	.054	75	.063	.062	.060
78	.054	.057	.054	78	.063	.062	.060
81	.054	.057	.055	81	.063	.063	.060
84	.054	.057	.055	84	.063	.062	.061
87	.054	.056	.055	87	.064	.063	.060
90	.054	.057	.055	90	.063	.062	.060
93	.054	.057	.055	93	.063	.062	.060
96	.054	.056	.055	96	.064	.062	.060
99	.055	.056	.055	99	.064	.062	.061
102	.054	.057	.055	102	.064	.062	.060
105	.054	.056	.055	105	.063	.062	.060

(1) Taken from the middle third of the extrusions.

(Table continued on next page.)

TABLE 15 (Cont.)

Tee Formed By Die 18				Tee Formed By Die 7C			
Distance From Front	Right Flange	Stem	Left Flange	Distance From Front	Right Flange	Stem	Left Flange
108 in.	.054	.057	.055	108 in.	.063	.062	.060
111	.055	.056	.055	111	.064	.062	.060
114	.055	.056	.055	114	.064	.062	.060
117	.054	.057	.054	117	.064	.062	.060
120	.055	.056	.054	120	.064	.062	.061
123	.055	.056	.054	123	.064	.062	.061
126	.054	.056	.055	126	.064	.062	.061
129	.054	.056	.055	129	.064	.062	.061
132	.054	.056	.054	132	.064	.062	.061
135	.054	.056	.054	135	.064	.062	.060
138	.054	.056	.054	138	.064	.062	.061
141	.055	.056	.054	141	.064	.062	.061
144	.054	.056	.054	144	.064	.063	.061
147	.054	.056	.054	147	.064	.063	.062
150	.055	.056	.053	150	.064	.063	.062
153	.055	.056	.053	153	.064	.063	.062
156	.055	.056	.053	156	.064	.063	.061
159	.054	.056	.053	159	.063	.063	.062
162	.054	.056	.053	162	.063	.063	.062
				165	.064	.063	.062

In those dies which produced good extrusions, die coating failure occurred when the unextruded butt was removed from the die. The loss of hardness of the steel substrate (see Table 13), including the dies which produced good extrusions, is believed to be caused in part by the 0.5-inch long, hot, unextruded butt left in the die after extrusion of the Tee section. However, the uniformity of the extrusions produced during the optimum combination of speed and temperature indicate that the die did not reach an adverse tempering exposure. This implies that a greater total length of extrusion could have been fabricated either by extruding a greater volume of metal or larger billets or by re-using the die if a technique can be developed to prevent this die coating from failing.

The dies and rear section of one good (14) and one bad (19) extrusion are shown in Figure 64.

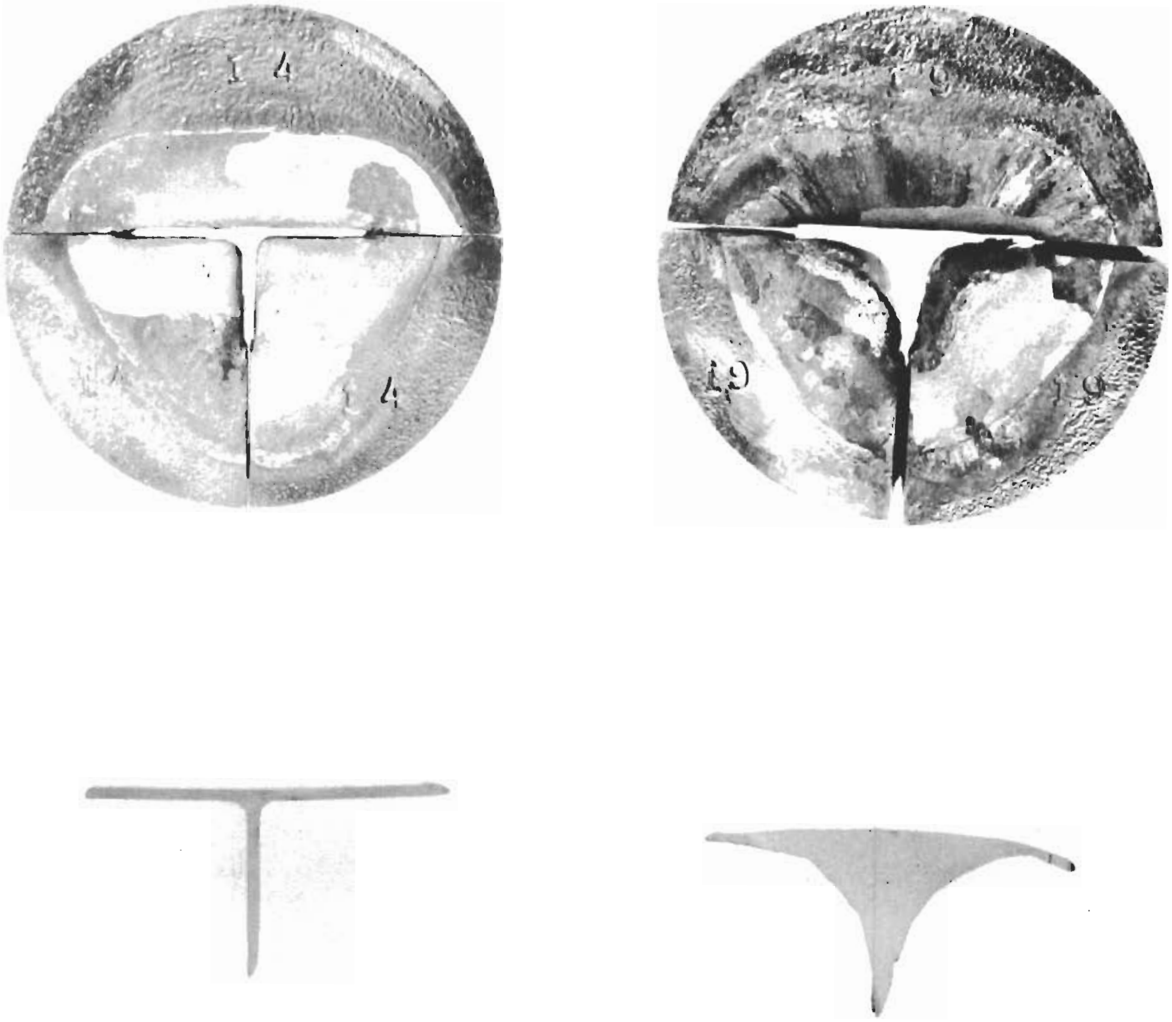


Figure 64. Extremes in Die Degradation by Steel.

Die No. 14 (top left) produced 42 feet of good extrusion at 2300°F and 4 in./sec. A section of the rear of the extrusion is seen at bottom left. Die No. 19 (top right) produced about 25 feet of good extrusion at 2000°F and 0.75 in./sec. The entire rear half of the extrusion was poor, as shown by the Tee section (bottom right). Die about 2/3 size; extrusions about actual size.

III. CONCLUSIONS

A. Problems Solved

The major feature of success of this work was the development of the massive refractory ceramic based materials which could withstand the temperatures and forces necessary for extrusion of complex shapes of refractory metals. Dimensional variation of less than 0.003 inch and surface smoothness of 63 rms was achieved. The front to rear scores present in extrusions with coated dies are not present in extrusions through massive ceramic dies. Massive refractory dies thus avoid the inherent weakness of coated die systems. When the entire die body can be made of a material at least as refractory, hard and erosion-resistant as the coating on a steel die, two problems are eliminated; first, the problem of coating adherence; and second, the problem of softening of the steel due to the passage of heat through the coating.

The materials which have been found capable of withstanding the extrusion conditions of shaped refractory metals are the hard, chemically inert, high melting ceramic materials of great compressive strength. Specifically, these were: ZrO_2 and TiC, as well as mixtures like TiB_2 - Al_2O_3. The part metal-part ceramic group, which gave promising performance, are the metal fiber and powder reinforced ceramics. Specifically, these are $ZrSiO_4$-20^v/o W fibers - 20^v/o W powder and $ZrSiO_4$-20^v/o W fibers and 20^v/o W powders.

There is experimental evidence from this program (as well as property data in the literature) which indicates that demonstration of success with ZrO_2 and TiC means that a wide variety of materials should make successful dies. These include the simple substances or alloys of refractory oxides, borides, nitrides, carbides and, perhaps, silicides and sulfides of the transition metals in Group IV, Group V, and Group VI of the Periodic Table. A partial list would include TiC, ZrC, HfC, TiB_2, ZrB_2, HfB_2, CbC, TaC, CbB_2, TaB_2, TiN, ZrN, WB, WC, ZrO_2, ThO_2, HfO_2, $MoSi_2$, and WSi_2.

By optimization of extrusion parameters, as specified by the Technical Monitor, structural shapes of alloy steel can be extruded with a dimensional variation of 0.002 inch and surface smoothness of 125 rms in lengths of at least 40 feet. Optimum conditions specified involve glass lubrication, fast billet transfer, Al_2O_3 coated steel dies, billet temperatures of 2300°F and ram speeds of 8 to 15 in./sec.

The results with massive ceramic dies with TZM extrusion (negligible die wear and absence of scores on the extrusions) imply that even better steel extrusions could be obtained if massive ceramic dies were used instead of ceramic coated dies.

Results of the program also led to other achievements or conclusions. A process was developed for the production of short lengths of tungsten wire or fibers for use in composite dies. Special supports and fabrication procedures were developed for ceramic dies.

B. Problems Remaining

Several major problems require solution before optimum dies can be developed from these promising materials, and then utilized in hot extrusion practice. These are in the areas of composition, fabrication, and design of dies.

The promising compositions of the massive, pure compound dies suffer from thermal shock. The promising metal-ceramic dies tried in this program may not have been fabricated from even near-optimum constituents in desirable volume fractions. The massive ceramic die compositions, which produced the best extrusions, are those for which the most information regarding fabrication conditions was known. The conditions used for the ceramic metal compositions were, generally, merely technical judgments and not based on experimental studies. Optimum conditions for those materials are unresolved. The shape of the ceramic die, although important, was not studied extensively in the program. Improved die performance requires investigation of die design.

IV. RECOMMENDATIONS

The experimental results summarized above indicate considerable improvement in refractory die performance could be obtained by study and optimization of die composition, fabrication, and design.

A. Die Composition

The most favored die compositions for study should be based on the simple substances or alloys of the refractory oxides, carbides, borides, nitrides, and possibly silicides of the transition metals, either self bonded or bonded with a refractory constituent. Less favored die compositions for study would include the metal-ceramic composites such as the macrolaminates or metal-fiber-ceramic composites.

B. Fabrication

Conditions and procedures for converting the initial constituents of the dies, generally powder, into a dense fine-grained body should be studied and continued for specific die compositions. In the case of the dies composed of simple substance, the initial particle size, shape, and distribution, the hot pressing or pressing and sintering conditions require study. In the case of ceramic alloys, the procedure for compositional addition adds another parameter for study. The metal-ceramic composites introduce the additional variables of preparing the fibers, and mixing the fibers with ceramic, aside from the time-temperature-pressure study needed for optimum hot pressing. Optimization in hot-press equipment such as improved alignment, special atmospheres or vacuum and improved hot-press tooling may be necessary.

C. Die Design

Investigations are needed which make design comparisons between conical and shear die entries, segmented (split) and integral dies, and various methods of die support.

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