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Producers & Fabricators of

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- HAFNIUM

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TUNGSTEN EXTRUSION PROGRAM

A STATE-OF-THE-ART SURVEY

First Interim Technical Report
2 December, 1960 - 2 March, 1961

TUNGSTEN EXTRUSION PROGRAM

State-of-the-Art Survey

WAH CHANG CORPORATION

For Submission to

Manufacturing and Materials Technology Division
AMC Aeronautical Systems Center
Wright-Patterson Air Force Base, Ohio

on

Air Force Contract No. AF 33(600) - 42395

ABSTRACT

The state-of-the-art on the extrusion of tungsten and its alloys is reviewed. A resume of physical and mechanical property data and present industrial capabilities for ingot production and extrusion practice is presented.

Ingot and mill product preparation techniques are discussed. Both powder metallurgy and arc consolidation may be utilized to prepare small ingots. However, the consumable electrode arc-melting process is the only method being used to produce tungsten ingot stock of sufficient size to warrant consideration.

Limited mechanical property data is presented on tungsten and several tungsten base alloys. Recrystallization behavior and factors affecting ductile-to-brittle transition temperature are also discussed.

It is reported that conventional extrusion experience on tungsten and its alloys has been limited primarily to round bar at reduction ratios of less than 10:1. The major problem areas which must be overcome before a successful tungsten tee extrusion can be obtained are the limitations in die materials, lubrication, and insufficient billet heating capability.

NOTICE

FOREWORD

This Interim Technical Progress Report covers the work performed from 2 December, 1960 to 2 March, 1961 under Air Force Contract No. AF 33(600) - 42395. It is published for technical information only and does not necessarily represent the recommendations, conclusions, or approval of the Air Force.

This contract with the Wah Chang Corporation was initiated under AMC Manufacturing Methods Project No. 7-793, "Tungsten Extrusion Program". It is administered under the direction of Mr. T. S. Felker of the Metallic Materials Branch, Manufacturing and Materials Technology Division, AMC Aeronautical Systems Center, Wright-Patterson Air Force Base, Ohio.

The State-of-the-Art Survey was conducted by the Battelle Memorial Institute under subcontract from the Wah Chang Corporation. Personnel from Battelle Memorial Institute who contributed toward this survey were: Mr. T. G. Byrer, Mr. F. F. Schmidt, Dr. J. Maykuth, Mr. A. M. Sabroff and Mr. F. W. Boulger.

Mr. A. E. Riesen of the Wah Chang Corporation was the engineer in charge. Others who cooperated in the research and in preparation of this report were: Dr. J. Wong, Director of Metallurgical Research, Mr. S. Yih, President, Mr. Mark McNabb and Mr. E. Baroch. This report has been assigned the Wah Chang Research Project No. AF-42395.

Approved by:

JAMES WONG
Director of Metallurgical Research

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INTRODUCTION

Within the next decade, aircraft, missiles and spacecraft will be moving at speeds which will require engines and airframes capable of sustained operation in the temperature range of 3000 - 4000 F. At these operating temperatures, the use of refractory metals offers the most practical and immediate approach to meeting these structural and component requirements for strength and stability. Among the structural form requirements for the construction of future aerospace vehicles will be the need for refractory metal extruded shapes.

Tungsten and tungsten alloys have the greatest potential for high-strength high-temperature applications. At the present time, the extrusion industry cannot supply aircraft quality tungsten extrusions equivalent to those required and supplied in other metals. The Materials Advisory Board has indicated a potential need for structural shapes in tungsten within the next three years. Thus, the need is evident for the creation of the necessary capabilities to produce extrusions of tungsten and other refractory metals.

In view of this, the Air Material Command awarded the Wah Chang Corporation U. S. Air Force Contract No. AF 33(600)-42395 for the "Development of Techniques for the Production of Tungsten Extrusions". This program involves five phases as indicated below:

Phase I - State-of-the-Art Survey

Phase II - Billet Process Development

Phase III - Development of the Extrusion Operation

Phase IV - Verification of Process Uniformity and Development of Post-Extrusion Operations

Phase V - Pilot Production of the Target Section.

The target section has the following configuration and property requirements:

1. Maximum Tee section circumscribed within a 2-inch diameter circle
2. Minimum length of 10 feet
3. Width of flange equal to twice the depth of section.
4. Thickness of flange and stem of 0.250-inch ±0.010-inch
5. Surface finish of 150 rms minimum.
6. Target mechanical property of 0.2 per cent creep deformation at 3750 F after 100 hours under stress of 20,000 psi.

This report covers the Phase 1 effort. This survey was conducted by Battelle Memorial Institute under the direction of Wah Chang Corporation. The objectives of the survey were:

1. To determine the present state-of-the-art in the production and extrusion of tungsten and its alloys
2. To establish the most suitable materials and procedures presently available for carrying out the development effort in the program.

In conducting the survey, use was made of a questionnaire, plant visits, and an extensive search of the literature and the Defense Metals Information Center. The questionnaire and a partial list of the organizations contacted is given in Appendix I.

RECOMMENDATIONS AND CONCLUSIONS

On the basis of the findings of the survey, the following recommendations are made:

1. That unalloyed tungsten be selected as the base material for Phase II.

2. That consumable-electrode arc melting, alone or in conjunction with electron-beam melting, be used to prepare ingots from which the extrusion billets are to be prepared.

3. That the usefulness of grain refining additions, preferably 2 per cent molybdenum, be considered as part of the Phase II evaluation.

These recommendations are based upon the following conclusions reached as a result of this survey:

1. At the present time, thoriated tungsten (i.e., tungsten containing 1 to 2 per cent ThO_2) is the only alloyed form of tungsten shown to have a significant strength advantage over unalloyed tungsten at temperatures above 3500F.

2. The use of powder metallurgical techniques to prepare thoriated tungsten is essential.

3. The preparation of satisfactory quality extrusion billets, in the diameters of interest to this survey, by powder metallurgy techniques entails more difficulty than the use of melting techniques.

4. The production capability for thoriated tungsten extrusion billets, in diameters of 3-inches or greater, has not yet been established.

5. Generally, tungsten prepared from melted ingot is characterized by a higher total purity than can be presently obtained by powder metallurgy consolidation practices. The higher purity associated with the melted product may be expected to contribute to greater ductility in this material at elevated temperature. Conversely, the higher purity may lead to a reduction in the recrystallization temperature.

6. Electron-beam melting of tungsten is generally believed to yield an ingot of higher total purity than that which can be obtained by consumable-electrode arc melting. However, the purity advantage of electron-beam melting over consumable-electrode melting for tungsten has not yet been demonstrated. Moreover, ingots produced by electron-beam melting are characterized by appreciably larger grain sizes than are consumable-electrode arc melted ingots, a factor which is expected to decrease the extrudability of unalloyed electron-beam melted tungsten.

7. The grain size of electron-beam melted tungsten can be reduced significantly by grain refining additives, and at least one so-treated 3.35-inch-diameter tungsten ingot has been successfully extruded. However, the effects of the grain refining additive used on the subsequent properties of the metal have not yet been evaluated.

8. The largest diameter W ingots now being made by electron-beam melting are 4-inches, while unalloyed tungsten ingots as large as 9-inches in diameter have been made by consumable-electrode melting.

9. Conventional extrusion has been used to break down the as-cast structure of unalloyed tungsten and tungsten-molybdenum alloys with good success. Successful high-velocity extrusion of tungsten materials has also been reported. There is no known extrusion experience on structural shapes in tungsten materials.

10. Extrusion of cast tungsten and tungsten alloys at about 3000 F has required higher working pressures than most commercial molybdenum alloys. Extrusion of a Tee at ratios between 11:1 and 17:1 will probably require a billet temperature greater than 3500 F for cast billets. Pre-extruded and recrystallized billets may require lower temperatures and pressures.

PRODUCTION OF TUNGSTEN AND TUNGSTEN ALLOYS

The present source of all commercial tungsten products is tungsten powder. The method most commonly used in tungsten powder preparation is the hydrogen reduction of tungstic oxide. Ammonium paratungstate is an intermediate in the preparation of tungstic oxide, but may also be hydrogen reduced directly to tungsten powder.

The major producers and/or suppliers of tungsten powder in the United States include the following:

Belmont Smelting and Refining Works, Inc.
Cleveland Tungsten, Inc.
Fansteel Metallurgical Corporation
Firth Sterling, Inc.
General Electric Company
Kennametal, Inc.
Metal and Thermite Corporation
North American Phillips, Inc.
Reduction and Refining Company
Shieldalloy Corporation
Sylvania Electric Products, Inc.
Union Carbide Corporation
Wah Chang Corporation
Westinghouse Electric Corporation

Raw Materials

Tungsten powder is available in a wide range of average particle sizes, extending from about 0.015 to 500 microns. The particle size is dependent on a number of processing variables including size and purity of the original oxide, the reduction times and temperatures, and concentration of water vapor in the hydrogen reductant. Although the metal powder is available in a wide range of particle sizes, the preferred size range for consolidation to fabricable shapes and consumable-electrode stock extends from about 0.5 to 10 or 15 microns. The physical characteristics of several representative grades of commercial unalloyed tungsten powder are given in Table 1.

The current purity specifications of 14 different companies and organizations for tungsten powder have been listed according to end

TABLE I.

PHYSICAL CHARACTERISTICS OF SEVERAL GRADES OF UNALLOYED COMMERCIAL TUNGSTEN POWDERS
(Reference 1)

Powder Designation	Average Fisher Size	Bulk Density, gm/in^3	Particle Size Distribution, Per Cent by Weight In Micron Size Ranges					
			1	2	3	4	5	6
M-10	0.90	30	22	72	6			
M-20	1.6	40	5	60	32	3		
M-30	2.4	48	3	45	39	10	3	
M-40	4.2	65	2	7	26	35	21	9
M-50	3.7	48	1	7	32	37	18	5

usage in powder metallurgy or melted product in Table 2. Oxygen appears to be the largest single impurity in tungsten powder and current specifications for each of the other major impurities vary widely.

Several types of prealloyed tungsten powders are used in the preparation of tungsten alloy shapes. These include "doping", thoria, and metallic additions. Generally, the doped and thoriated powders are not marketed as such but rather are supplied as sintered or wrought product.

The doping additions generally consist of alkaline oxides with silica and/or alumina. These are normally added as soluble salts to the starting tungstic oxide for the purpose of producing an interlocking grain structure in the wrought and recrystallized metal. Typical doping combinations used include K_2O-SiO_2, $K_2O-Na_2O-SiO_2$, $K_2O-SiO_2-Al_2O_3$, and K_2O-SiO_2-CaO. Normally, these doping additions are added to tungstic oxide in amounts from 0.10 to 0.45 per cent (2). Due to their volatile nature, most of these additions are lost in processing the powder to wrought product.

Thoria is also most commonly added as a soluble salt prior to oxide reduction. The principal effects of thoria are to delay recrystallization and inhibit grain growth. Thoria additions of 0.75 to 2 per cent are most frequently used although, on an experimental basis, additions as high as 5 per cent have been investigated (3).

The only metallic prealloyed tungsten powders which have been used in substantial amounts to date are binary tungsten-molybdenum alloys. These are prepared by a proprietary reduction practice and can be made practically in any tungsten/molybdenum ratio desired. In practice; however, most of the producer's experiences have been with the 85W-15Mo and 50W-50Mo alloys.

Billet Preparation

All of the extrusion work with tungsten and tungsten alloys has been carried out with billets prepared by powder-metallurgical techniques or by consumable-electrode or electron-beam melting practices. Because these consolidation practices exert an appreciable effect on the composition and metallurgical properties of tungsten, the status of these practices is reviewed in some detail. While hot

TABLE 2. CURRENT PURITY SPECIFICATIONS FOR TUNGSTEN POWDER

	Impurity Content, weight per cent										
	O	N	H	C	Al	Ca	Fe	Mo	Ni	Si	W
Producers:					For Use in Powder Metallurgy Product						
Cleveland Tungsten	0.20	- - -	- - -	0.01	- - -	- - -	0.01	0.005 0.030	- - -	0.01	- - -
Fansteel	0.10	0.005	0.005	0.02	0.005	- - -	0.02	0.02	0.02	- - -	- - -
Firth Sterling	0.30	- - -	- - -	0.01	0.01	0.01	0.02	0.01	0.02	0.01	- - -
General Electric (Lamp Metals and Components Department)	0.01	- - -	- - -	0.003	<0.001	- - -	0.002	0.003	<0.001	<0.001	- - -
Reduction and Refining	0.05	- - -	- - -	0.001	0.002	- - -	0.001	0.05	0.002	0.003	- - -
Sylvania	0.15	- - -	- - -	- - -	- - -	- - -	0.002	- - -	0.001	- - -	- - -
Wah Chang	0.030	- - -	- - -	0.002	<0.002	- - -	0.003	0.0500	<0.001	<0.003	- - -
Westinghouse	- - -	- - -	- - -	- - -	0.005	- - -	0.005	0.002	0.002	0.01	- - -
Consumers:											
Aerojet General (Sacramento)	0.02	0.02	0.01	0.025	- - -	- - -	0.05	0.015	- - -	0.05	- - -
Allison Division of General Motors	0.5	0.03	- - -	0.001	<0.001	- - -	<0.001	<0.003	<0.001	<0.001	- - -
General Electric Research Lab.	0.01	0.001	- - -	0.005	<0.001	- - -	<0.001	0.003	<0.001	<0.001	- - -
Raytheon	- - -	- - -	- - -	- - -	0.01	- - -	0.03	0.01	0.03	0.01	- - -
Universal Cyclops	0.015	0.003	- - -	0.005	- - -	- - -	0.0005	0.0025	0.0004	0.0003	- - -
						For Use In Melted Product					
General Electric Research Lab.	0.01	0.001	- - -	0.005	<0.001	- - -	<0.001	0.003	<0.001	<0.001	- - -
Oregon Metallurgical Corp.	0.03	- - -	- - -	0.01	- - -	- - -	- - -	- - -	- - -	- - -	99.9 min.
Universal Cyclops	0.01	0.003	- - -	0.005	0.001	- - -	0.003	0.001	0.001	0.002	- - -
Wah Chang Corp.	0.010	- - -	- - -	0.002 0.010	<0.002	- - -	0.003	0.05	<0.001	<0.003	- - -
Westinghouse (Blairsville)	- - -	- - -	- - -	- - -	0.002	- - -	0.002	0.01	0.002	0.002	- - -

pressing, plasma-jet spraying, and slip casting techniques are being investigated by various groups for the consolidation of tungsten, none of these techniques have been used to prepare extrusion billets of tungsten or its alloys. Hence, these consolidation practices are not considered pertinent to this survey report.

Powder Metallurgy

Compacting. A wide variety of shapes and sizes have been made from tungsten powders, These include rectangular and cylindrical bars, slabs, tubes, rings, rocket nozzles, vanes, and other contoured shapes. The compacting techniques most commonly used are mechanical pressing in tool steel dies or explosive or isostatic pressing in rubber or soft plastic containers.

Mechanical pressing is generally limited to the production of simple shapes, i.e., rectangular pieces or short tubes and cylinders. Explosive compacting of tungsten, on the other hand, has generally been limited to the production of simple cylindrical rounds. At present, the greatest variety of shaped tungsten powder products has been obtained with isostatic pressing. Facilities already exist for the production of cylindrical rounds, by isostatic pressing in diameters up to 14 inches and in lengths to 15 feet.

Generally, pressures in the range of 30 to 50 ksi are used for compacting tungsten. While as-pressed densities may range from 50 to 75 per cent of theoretical, densities of the order of 60 per cent are most commonly attained.

Sintering. The densification of tungsten in sintering depends upon a number of factors including initial powder purity, particle size, compacting pressure, and sintering time and temperature. The available information on current commercial sintering practice has been summarized in considerable detail in the First Interim Report on the AMC Tungsten Sheet Rolling Contract, AF 33(600)-41917, by Battelle in their survey for The Universal-Cyclops Steel Corporation (4). Summarily, experience has shown that fine powders (average particle size of 1 micron) sinter more readily than coarse powders (average particle size of 5 microns). Also, higher purity accelerates the consolidation rate. Thus, dopes and/or thoria additions retard densification such that appreciably longer sintering times or higher temperatures are required.

The specific time-temperature combinations used to sinter tungsten vary widely and depend on both the end use of the compacts as well as the facilities available at the individual organizations.

Where the sintered compacts are to be used for consumable-electrode arc melting although densities in the range of 70 to 90 per cent theoretical are preferred, densities as low as 60 per cent theoretical appear permissible. For compacts to be worked directly, e.g., sintered extrusion billets, appreciably higher densities (of the order of 90 per cent theoretical) are required. Densities of this order can be achieved with unalloyed tungsten powder in a relatively short time (1 to 2 hours) at temperatures of 4400 to 4600°F. The most practical means of attaining these temperatures in commercial practice has been through the use of self-resistance heating. This technique, however, is limited primarily to bars with small section sizes (i.e., of about 2 square inches in section). As a consequence, induction or radiation heating, under vacuum or in hydrogen have been the principal methods of heating pressed electrode bars and extrusion billets for sintering.

The available data on the types and capabilities of sintering furnace equipment available are summarized in Table 3. Details of current sintering practices for large diameter unalloyed tungsten bars are given in Table 4. Relatively little information is available on the time-temperature combinations necessary to achieve high densities in large section-size compacts of doped and/or thoriated tungsten, although information of this type is now being generated by Fansteel on their tungsten sheet rolling program for the US Navy (5). Each of the powder producers has acknowledged the fact that it would be difficult to economically sinter large diameter bars of thoriated tungsten to a minimum density of 90 per cent theoretical. This is primarily due to the lack of adequate high-temperature sintering furnaces. The General Electric Company has, however, prepared at least two 3-inch diameter bars of the $W-1ThO_2$ composition, using proprietary methods(4).

Density variations within large sintered tungsten compacts represent one possible problem area with these materials. A number of forging companies (6) have reported forging difficulties with sintered billets in which the failures were at least partially attributed to large density variations between the center and surfaces of the billet. Information received from the major powder producers for this survey

TABLE 3.

SINTERING-FURNACE FACILITIES AVAILABLE FOR TUNGSTEN COMPACTS

Organization	Type Of Heating	Maximum Temperature, F	Billet-Size Capability	Atmosphere	
				Vacuum Pressure, microns	Inert Gas
Fansteel	Induction	4350	5"	--	H_2
Firth-Sterling	Indirect	--	4"	5	--
	Indirect	--	12" x 15"	--	H_2
	Indirect	--	5" x 10"	--	H_2
General Electric	Radiation	3270	12" x 35"	--	H_2
Kennametal, Inc.	Induction	4500	6" x 18"	Vac.	--
	Radiation	4500	6" x 18"	Vac.	--
NASA	Radiation	4000	2-1/2" x 6"	0.01 - 0.5	--
Sylvania	Electric	3270	10" x 48"	--	H_2
	Electric	3810	--	Vac.	--
Wah Chang	Induction	--	4" x L	<100	--
Westinghouse (Blairsville)	Resistance	3180	8" x L	--	H_2
	Induction	3540	5" x L	--	Argon

TABLE 4.

DETAILS FOR SINTERING OF UNALLOYED TUNGSTEN POWDER-METALLURGY COMPACTS

Organization	Size of Billet	Time, Hours	Temperature, F	Atmosphere	Density, per cent theoretical	
					Initial	Final
Fansteel	--	--	--	Dry H_2	--	90-92
Firth-Sterling	--	--	--	--	51-61	87-94
General Electric	6" Ø	--	>3270	H_2	60-70	92-97
Kennametal, Inc.	4" Ø	2	4200	Vac.	55	84.5
NASA	1" - 2"	4-24	3000-3500	H_2	--	70-75
	2-1/2" x 6"	2-16	3500-4000	Vac.	--	--
Sylvania Electric	--	--	3270	--	60-65	92 ± 2
Wah Chang Corp.	2	--	2550	Vac.	--	85-87
Westinghouse Electric	1" - 3"	20-100	3090	H_2	--	92-98

indicate that a 2 per cent tolerance on center-to-surface density variation can now be met for sintered unalloyed tungsten bars in diameters up to 6 inches. No comparable experiences or data for doped and/or thoriated tungsten bars are available.

Laboratory studies at Battelle(7) and General Electric(8) have shown that appreciable purification of tungsten from interstitial impurities can be achieved by vacuum or hydrogen sintering small diameter bars (maximum of 3/8 x 3/8 inch section) at high temperatures. In both studies, self-resistance heating was used to reach the temperatures (4600 F and greater) where the purification reactions were completed readily (i.e., within 2 hours). The lowest interstitial level achieved(8) was 2 ppm oxygen, 1 ppm nitrogen and <1 ppm hydrogen after vacuum sintering 2 hours at 5610°F. However, the author admits an analytical range of ± 5 ppm.

Melting

Two methods of melting have been used in the preparation of extrusion billets of tungsten and its alloys. These include cold-mold consumable-electrode arc melting and electron-beam melting.

Consumable-Electrode Melting. Experience in the consumable-electrode arc melting of tungsten is being accumulated rapidly. At least seven organizations have used this procedure to produce good-quality unalloyed tungsten ingots in diameters of 4 inches or greater. Unalloyed tungsten ingots as large as 9 inches in diameter have been made. So far as tungsten alloys are concerned, production melting capabilities have been demonstrated for only a few binary tungsten-molybdenum compositions, most notably the 85W-15Mo alloy which is now being melted by several producers in diameters up to 12 inches. The facilities at which tungsten and tungsten alloy ingots, in diameters of 4 inches or larger, have been melted are listed in Table 5.

Consumable-electrode-melting practices for seven organizations are summarized in Table 6. In most instances, simple round or cylindrical electrode sections are used. For small electrode sections (1-1/2 inches in diameter or less), successful joining by inert atmosphere arc welding has been employed although weld cracking introduces some difficulties. For larger sections, threading and tapping are almost universally used to assemble the electrodes. Best results are obtained by a combination of threading, tapping and welding to improve both strength and electrical conductivity.

TABLE 5.

FACILITIES AT WHICH TUNGSTEN AND/OR TUNGSTEN ALLOY INGOTS IN DIAMETERS OF 4-INCHES OR LARGER HAVE BEEN PREPARED BY COLD MOLD CONSUMABLE ELECTRODE ARC MELTING

	Maximum Ingot Diameter, inches	
Facility	Unalloyed Tungsten	Tungsten Alloy
Climax Molybdenum Company	9	12 (85W-15 Mo)
General Electric Corporation	4	--
Oregon Metallurgical Corp.	4	12 (85W-15 Mo)
Universal-Cyclops Steel Corp.	8	12 (85W-15 Mo)
Wah Chang Corporation	4-1/2	4 (85W-15 Mo)
Westinghouse Electric Corp.	8	--
U.S. Bureau of Mines (Albany)	5	--

TABLE 6. SUMMARY OF DATA FOR CURRENT CONSUMABLE-ELECTRODE ARC MELTING PRACTICE AS APPLIED TO TUNGSTEN

		Melting Conditions						
Organization	Electrode Configuration	Electrode Material	Ingot Diameter Inches	Electrode/Mold Diameter Ratio	Voltage	Amperage	Polarity	Furnace Atmosphere
Climax Molybdenum Company	Round	W	2 - 5	0.23-0.56	--	--, AC	--	Vac.
	Hexagonal	W	5 - 9	0.24-0.42	--	--, AC	--	Vac.
		85W - 15 Mo	12	--	--	--, AC	--	Vac.
General Electric Company Research Laboratory	--	W	4	0.25-0.35	40	5000, DC	--	Vac. (2)
NASA (Lewis Research)	Round	W	2-1/2	0.40-0.50	28-30	4500		
						4700, DC	Reverse	Vac.
Oregon Metallurgical Corporation	Square	W	4	0.50	--	--	--	--
	Round	85W - 15 Mo	12	--	25	30000, DC	Straight	Vac. (20)
Union Carbide Metals Co.	Square	W	1-1/2	0.25	23-25	2200, AC	--	Vac.
	Round	W	2	--	25-26	3500, AC	--	Vac.
Universal-Cyclops Steel Corporation	Round	W	4 - 8	0.38-0.50	30-35	4000 - 15000, DC	Straight	Vac. (10)
		85W - 15 Mo	11-3/4	0.51	42	23000, DC	Straight	Vac. (10)
U. S. Bureau of Mines, Albany, Oregon	Square	W	2-1/2	0.40	30-32	3500 - 4000, DC	Straight	Vac. (150)
	Round	W	3	0.33	30-32	4800 - 5000, DC	Straight	Vac. (150)
		W	4	0.63	30-32	4000 - 4500, DC	Straight	Vac. (150)
		W	5	0.60	30-32	7500 - 8000, DC	Straight	Vac. (150)
Wah Chang Corporation	Square Round	W	3-1/2	0.30-0.57	24-30	6000 - 6500, DC	Straight	Vac.
Westinghouse Electric Corporation (Metals Plant)	Round	W	4	0.25-0.50	30-32	5500, DC	Straight	Vac. (10)
		W	6	0.33	40	7000 - 10,000 DC	Straight	Vac. (10)

In melting, direct current, with straight polarity, is preferred by the majority. Voltages increase, with ingot diameter, over the range of 20 to 40 volts, while current requirements span the range from 2,200 to 30,000 amperes for ingots from 1-1/2 to 12 inches in diameter, respectively.

An appreciable degree of purification can be effected in tungsten by consumable-electrode arc melting. This subject was recently reviewed by Carnahan(9) who pointed out that:

(1) The elimination of carbon as CO is thermodynamically favorable at the melting point of tungsten with pressures on the order of 100 microns.

(2) The volatility of WO can be 100 times greater than the volatility of tungsten metal at this temperature.

(3) The elimination of tungsten nitride and hydride is also favored due to their instability under these conditions.

(4) The majority of the metallic impurities common to tungsten should also be evaporated under these conditions.

Among various factors associated with purification during melting, melt-off rate and vacuum pressure are recognized as being among the most important. The effect of variable melt-off rates is shown in Table 7. These data generally show that purification in melting decreases with increasing melting rate and suggest that, with melt-off rates of about 5 pounds per minute or greater, little or no purification can be obtained.

Comparative purification data obtained on vacuum sintered and arc cast carbon-reduced tungsten powder samples are also given in Table 8. In this work(13), AC melting was used. These investigators found no improvement in ingot chemistry when hydrogen, at reduced pressures, constituted the melting atmosphere. Similarly, the use of zirconium and manganese as gettering additions had no effect upon ingot chemistry at melting pressures of 100 mm argon. Boron additions gave a large increase in hardness and refined the grain structure, but did not deoxidize the metal.

TABLE 7.

PURIFICATION ACHIEVED BY SINGLE CONSUMABLE-ELECTRODE ARC MELTING OF TUNGSTEN UNDER THREE DIFFERENT CONDITIONS

	Analysis, ppm by weight					
Impurity	Electrode	Ingot	Raw Powder	Ingot	Electrode	Ingot
C	< 60	10	260	30	22	26
O	≲100	<1	540	20	24	20
N	≲ 10	<1	60	3	18	18
H	≲ 5	<1	11	1	--	--
Al	--	--	--	--	<5	<5
Ca	--	--	--	--	<10	<10
Cr	--	--	--	--	<5	<5
Cu	--	--	4	2	<5	<5
Fe	--	--	450	40	30	10
K	--	--	--	--	<10	<10
Mo	--	--	--	--	50	50
Na	--	--	--	--	<10	<10
Ni	--	--	30	5	<5	<5
P	--	--	<10	<10	--	--
S	--	--	120	10	--	--
Si	--	--	70	26	<5	<5
Th	--	--	--	--	<50	<50
Arc Melting Conditions	General Electric(10)		Union Carbide(11)		NASA(12)	
	Hydrogen-sintered electrode Straight polarity 200 flowing hydrogen 4-inch-diameter ingot from 2-inch-diameter electrode Melting rate: 0 33 lb/min.		Slip cast or pressed-and-sintered electrode, 85% theoretical density a-c melting 1 impurity gases 1-inch-diameter ingot from 5/8-inch diameter electrode Melting rate: 1 lb/min		Sintered and swaged electrode Reverse polarity (electrode +) <10 impurity gases 1-1/2-inch-diameter ingot from 3/4-inch-diameter electrode Melting rate: 5 lb/min	

TABLE 8.

COMPARATIVE IMPURITY CONTENTS OF SINTERED* AND OF ARC-CAST TUNGSTEN FROM TWO LOTS OF POWDER (13)

	Impurity Element	Raw Powder	Impurity Content, PPM Sintered (1930 F) Vacuum (0.1) 5 hr.	Impurity Content, PPM Sintered (3810 F) Vacuum (0.1) 4 hr.	Vacuum Arc-Cast
Lot A	Carbon	580	280	60	10
	Oxygen	2200	1800	50	40
	Nitrogen	50	24	11	5
	Hydrogen	--	7	3	1
Lot B	Carbon	630	170	90	30
	Oxygen	600	690	100	20
	Nitrogen	40	17	4	1
	Hydrogen	16	4	4	1

* 3/4" thick compacts

The grain size of tungsten ingot is of considerable importance with regard to subsequent fabricability(4). It is known; for example, that impurities present on solidification tend to concentrate at grain boundaries where they introduce a source of weakness. For a given impurity level, embrittlement effects are maximized where the ratio of grain surface area to unit metal volume is low, i.e., in large-grained structures. In large diameter*, consumable-electrode arc melted tungsten ingots, coarse columnar grain structures are obtained in which the diameters of individual grains may reach sizes of the order of 1/4-inch. Under these conditions, the only hot working method which has been consistently successful in breaking down the cast structure is extrusion. This fabrication process is favored since it is characterized by the relative absence of large tensile forces (in the outer fibers of the billet) in the initial deformation stages which frequently result in intergranular fracture.

In this connection, grain refinement in arc-cast ingots is of extreme interest. Here, the somewhat fortuitous effect of molybdenum has been noted and exploited, with varying degrees of success, by various organizations. This effect of molybdenum was probably first proven, in large ingot sizes (7 to 9 inches in diameter) by Climax(14) who found additions of 5 to 70 per cent tungsten substantially decreased the as-cast grain size of both tungsten and molybdenum. Data presented in Table 9 show that additions of as little as 2 per cent molybdenum have a significant grain refining effect.

The effectiveness of boron additions in refining the cast grain structure of arc-melted tungsten has been reported by both Morgan and Schottmiller(13) and the U. S. Bureau of Mines(4). In the former case, a large hardness increase resulted from the boron additions while in the latter "inferior fabricability" resulted. Significant grain refinement in unalloyed tungsten has also been observed by Semchyshen and Barr(16) with binary additions of cobalt (up to 2.8 per cent) and vanadium (additions in excess of 2 per cent). A proprietary grain refining addition for arc-melted tungsten has also been developed by General Electric(4). Reportedly, this addition reduces the grain size and workability is correspondingly improved.

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*Two inches and above.

TABLE 9.

EFFECT OF MOLYBDENUM ON THE GRAIN SIZE AND MELTING POINT OF ARC-MELTED TUNGSTEN (15)

Molybdenum Content, weight per cent	Grain Size	Melting Point, F
0	3.8 mm x 36 mm columnar in 2-in.-diameter ingot	6152
2	1.5 mm diameter in 3-inch diameter ingot	6125
7	1.5 mm diameter at surface, 1.5 mm x 20 mm columnar at mid radius, 2.5 mm diameter at center in 3-1/2 inch-diameter ingot	6062
15	1 mm diameter	5972
40	0.25 mm diameter in 6-inch-diameter ingots	5612
50	0.25 mm diameter in 6-inch-diameter ingots	5550

A number of alloying additions to arc-cast tungsten have been investigated. Some difficulty has been experienced in the retention of iron, cobalt, nickel, vanadium, titanium, and zirconium (4). Several investigators have shown that binary additions of tantalum or columbium, in amounts above about 5 per cent, have an embrittling effect. Cracking in ingots of the 85W-15Mo alloy has also been reported, although this problem has apparently been alleviated by a stress-relief heat treatment (4).

Electron-Beam Melting. Electron-beam melting of unalloyed tungsten ingots, in diameters up to 4-inches, has been successfully accomplished by the Stauffer Metals Company and the Wah Chang Corporation. For 4-inch diameter ingots, approximately 15,000 volts dc are required with corresponding currents of 1 to 10 amperes. Furnaces are operated at about 10^{-2} micron pressure, a condition under which significant purification occurs during melting (Table 10).

For unalloyed tungsten, the as-cast grain size in electron-beam-melted ingots is extremely large, e.g., grain diameters in excess of 1-inch in ingots of 3-inch diameter have been obtained. Stauffer has developed a proprietary grain refining addition which reduces the grain diameter of their electron-beam-melted ingots appreciably although no grain size advantage over that which can be obtained by ordinary consumable-electrode melting has been demonstrated. Nevertheless, some successes in the direct forging of grain refined, electron-beam-melted ingots have been reported (4).

PROPERTIES OF TUNGSTEN AND TUNGSTEN ALLOYS

Physical and Thermal Properties

Selected physical and thermal property data for pure, unalloyed tungsten are given in Table 11. No comparable physical property data are available for any tungsten alloys.

Softening and Recrystallization Behavior

The hardness of high-purity tungsten can be increased from minimum values of 340-360 Vhn for the dead-soft, fully recrystallized condition, to values in excess of 500 Vhn through hot-cold working (4,7). Interstitial and metallic contaminants, in the ranges normal for present commercial sintered product, have little or no significant effect on hardness (7).

TABLE 10.

TYPICAL ANALYSIS OF ELECTRON-BEAM MELTED TUNGSTEN (17)

Impurity Element	Content, weight per cent
C	0.002
O	0.006
H	<0.001
N	0.001
Al	<0.001
Ca	<0.001
Si	<0.001
Mo	0.001
Fe	0.002
Cr	<0.001
Ni	<0.001
Mn	<0.001
Cu	<0.001
Mg	<0.001
Sn	<0.001
W	99.95

TABLE 11.

SELECTED PHYSICAL PROPERTY DATA FOR UNALLOYED TUNGSTEN (18)

Melting point, F	6170
Boiling point, F	9900
Density, lb/in^3	0.697
g/cm^3	19.3
Crystal structure	Body-centered-cubic
Lattice parameter, A^{o}	3.158(3)
Specific heat, cal/g C	20C (70 F): 0.033 1000 C (1830 F): 0.041 2000 C (3630 F): 0.047
Thermal conductivity cal/sec-cm-C	20 C (70 F): 0.31 1000 C (1830 F): 0.27 2000 C (2910 F): 0.25
Linear coefficient of Expansion, 10^{-6}	20 C (70 F): 4.43 1000 C (1830 F): 5.17 2000 C (2910 F): 7.24

The recrystallization temperature of tungsten is dependent on several factors of which the degree of cold working and metal purity appear predominant. For severely cold-worked structures (i.e., small diameter tungsten wires), recrystallization can occur at temperatures as low as 1830°F. For rod or sheet stock, cold worked to reductions in the range of 50 to 75 per cent, temperatures of 2200-2400°F are normally required for complete recrystallization in one hour.

Interstitial contaminants, in the ranges normal for sintered product, have no significant effect on the recrystallization temperature(7). Conversely, trace metallic impurities appear to exert a far more potent effect. Work by two laboratories has indicated that reduction of metallic impurities, through zone or inert electrode arc melting, can lower the recrystallization temperature of tungsten by several hundred degrees Fahrenheit(3,7).

Additions of doping agents or thoria to tungsten increase the recrystallization temperature of tungsten(4,7).

Ductile-to-Brittle Transition Behavior

The ductile-to-brittle transition temperature of tungsten has been shown to be sensitive to a number of factors including grain shape and size, strain rate, and metal purity. Generally, elongating the grain shape through cold working, decreasing grain size or strain rate, or improving metal purity all tend to lower the transition temperature.

Recrystallized tungsten, tensile tested at strain rates around 10^{-3} sec^{-1} shows a ductile-to-brittle transition at temperatures near the high end of a range extending from 350 to 850 F(3,19,20). With decreasing strain rates, the transition temperature is lowered (Figure 1). For wrought tungsten rod, the tensile transition temperature is lower (in the range of 300 to 400F) than that obtained for recrystallized rod tested at the same strain rate(20,22).

Attempts to correlate the effects of impurities on the mechanical properties of tungsten have been hampered by the lack of suitable analytical techniques for accurately measuring impurity elements in amounts below about 10 ppm Nevertheless, Westinghouse (3) has shown that lower tensile transition temperatures for tungsten are achieved with increasing metal purity. In this study, variations in oxygen (from 1-15 ppm), nitrogen(2-29 ppm),

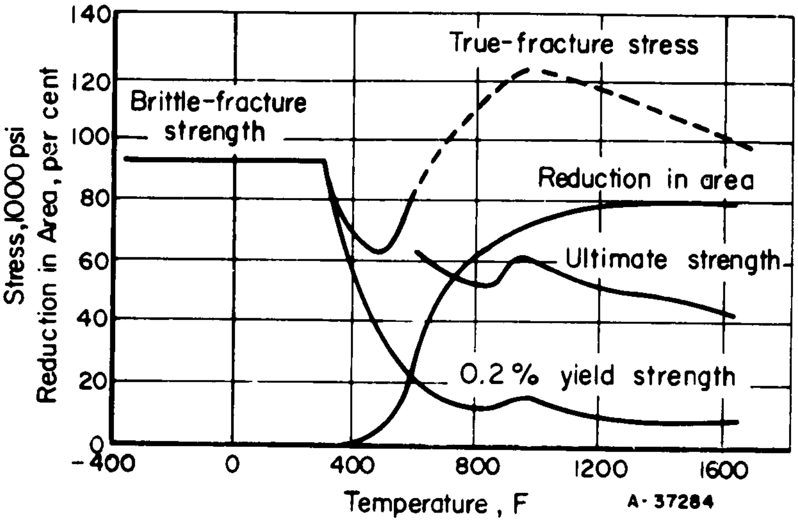


FIGURE 1. EFFECT OF TEMPERATURE ON THE TENSILE PROPERTIES OF RECRYSTALLIZED TUNGSTEN ROD (0.00028 SEC-1 STRAIN RATE)(19)

or hydrogen (from 1-3 ppm) appeared to have little effect. Rather, increases in the amount of trace metallic impurities, especially nickel, appear definitely detrimental.

The effectiveness of several alloying additions on the transition temperature of tungsten has been investigated(23) with the results shown in Table 12. As indicated, a W-1.11 Re alloy had the lowest transition temperature obtained. Other work has shown that rhenium additions in the range of 26 to 28 per cent are optimum for ductilizing tungsten in that room temperature ductility can be achieved for such alloys, even in the fully recrystallized condition.

The W-15Mo alloy, as extruded at a 6:1 ratio at 4000F, has a ductile-to-brittle transition temperature of about 500 F for both arc-melted and powder metallurgy material (33).

Strength Properties

At temperatures up through about 2500F, the tensile strength of tungsten appears quite sensitive to processing variables, e.g., type of consolidation method and degree of cold working. At 2500F, strengths from 25,000 to 50,000 psi has been reported as shown in Figure 2. With increasing temperatures, the effect of processing variables on tensile strength appears less marked, and above about 3500F, the ultimate strength of unalloyed tungsten appears to be essentially independent of both the consolidation practice used and prior thermal history.

On the other hand, the type of consolidation practice apparently has a marked effect on the degree of high-temperature tensile ductility obtained as shown in Figure 3. Thus, above about 2500F, the tensile ductility of powder-metallurgical product decreases rapidly while that of arc-melted product is maintained at high levels to at least 4000F. This loss in ductility of the powder-metallurgical product has been attributed to its higher total impurity content.

Most of the available elevated-temperature tensile data for wrought tungsten alloys are summarized in Figure 4. For the most part, these data represent the results of tests from single bars or heats.

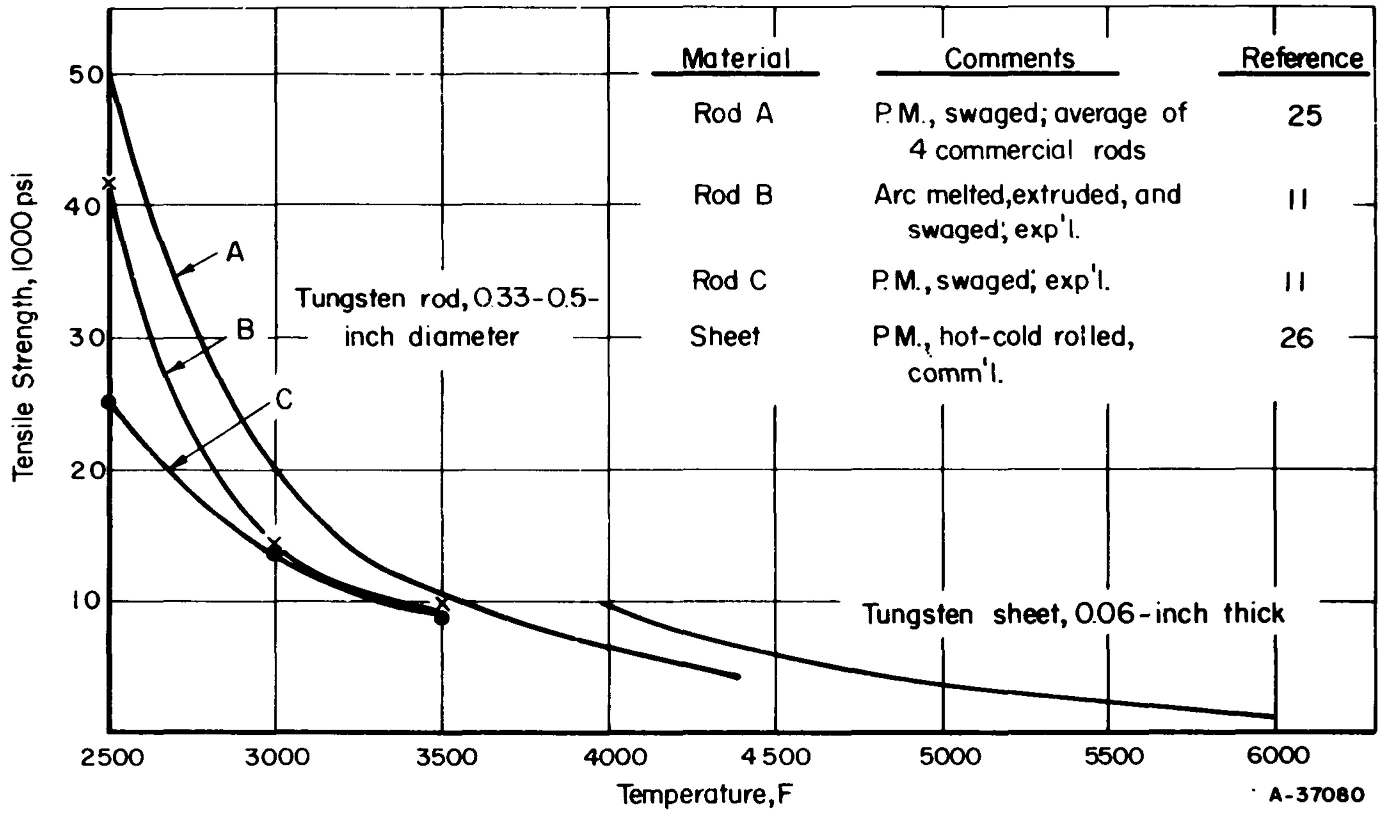
TABLE 12.

TENSILE TRANSITION TEMPERATURES OF SWAGED, POWDER-METALLURGY TUNGSTEN ALLOY RODS

Alloy Addition, Per Cent	Interstitial Content, ppm				Sintering Condition (a)		Tensile Transition Temperature, (b) F
	C	O	N	H	Type of Heating	Temp., F	
None	10	6	<5	0.2	Radiation	2200-4200	265
0.10 Ti	15	640	<5	1	Radiation	2200-4200	275
0.014 Ti	10	96	<5	1	Radiation	2200-4200	210
1.11 Re	10	13	<5	<1	Radiation	2200-4200	160
2.98 Re	30	5	<5	<1	Radiation	2200-4200	275
0.95 Ta	10	65	<5	<1	Radiation	2200-4200	250
0.065 Zr	10	230	<5	1	Radiation	2200-4200	275
None	10	22	<10	0.7	Resistance	4710	317
None	--	--	--	--	Resistance	4350	327
0.25 Y	11	21	<10	0.1	Resistance	3990	418
0.07 Ti	9	34	<10	1	Resistance	3990	351
0.21 Zr	10	66	<10	0.8	Resistance	3990	312

(a) Vacuum atmosphere used

(b) Designated arbitrarily as lowest temperature at which 5 per cent elongation was obtained



Material	Comments	Reference
Rod A	P.M., swaged; average of 4 commercial rods	25
Rod B	Arc melted, extruded, and swaged; exp'l.	11
Rod C	P.M., swaged; exp'l.	11
Sheet	P.M., hot-cold rolled, comm'l.	26

FIGURE 2. EFFECT OF TEMPERATURE ON THE TENSILE STRENGTH OF WROUGHT, UNALLOYED TUNGSTEN ROD AND SHEET

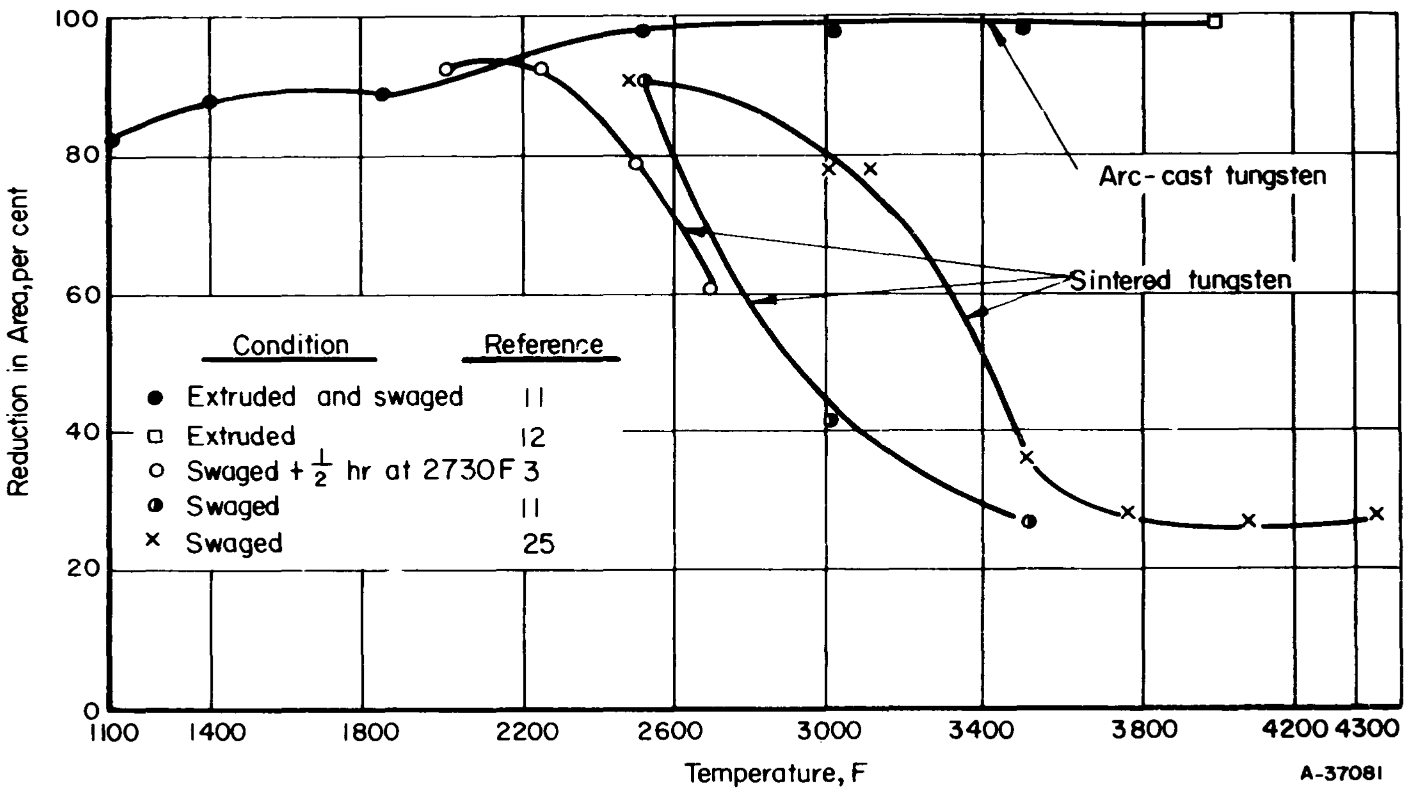
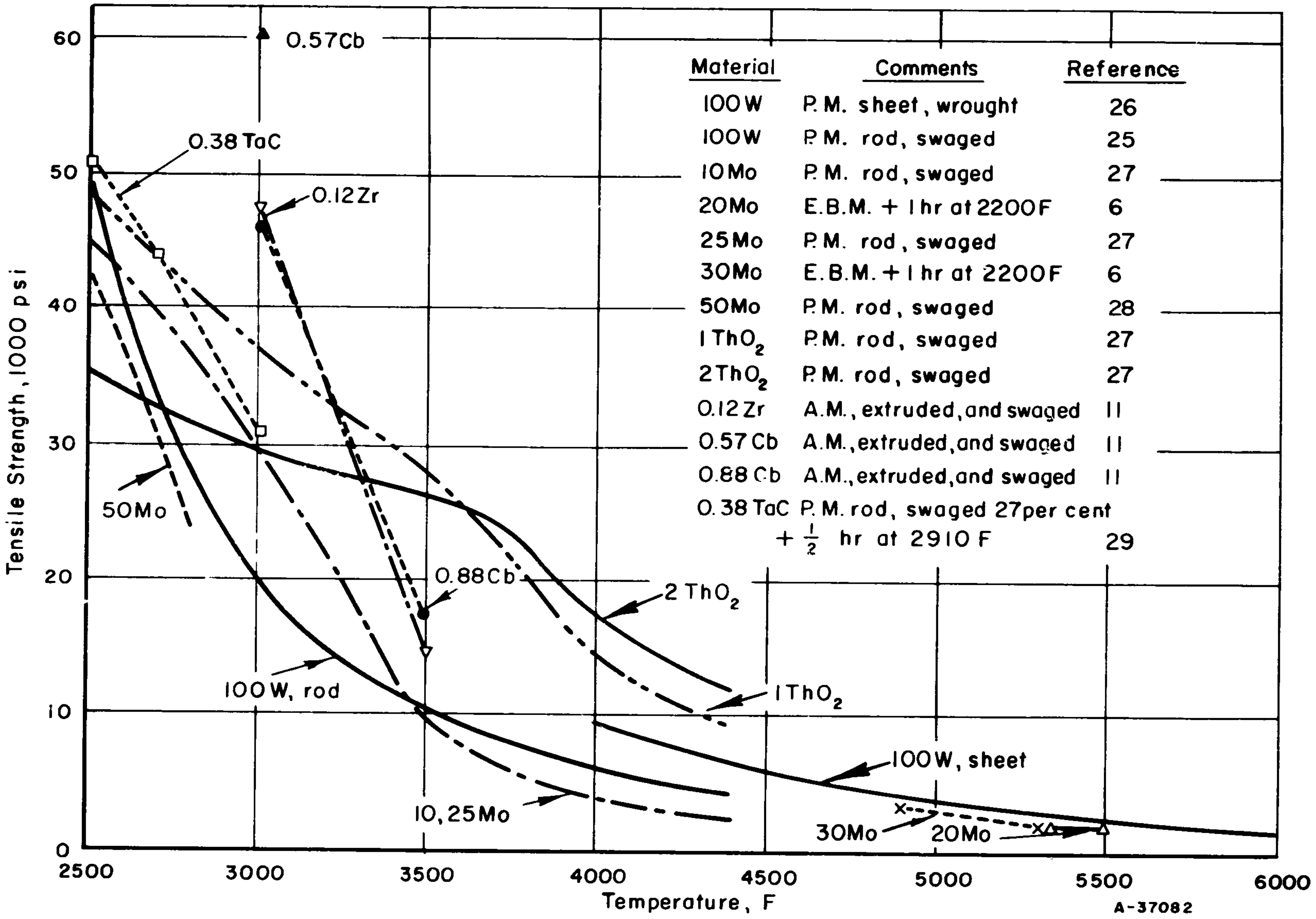


FIGURE 3. EFFECTS OF TEMPERATURE ON THE DUCTILITY OF ARC-CAST AND POWDER-METALLURGY TUNGSTEN(6)



Material	Comments	Reference
100W	P.M. sheet, wrought	26
100W	P.M. rod, swaged	25
10Mo	P.M. rod, swaged	27
20Mo	E.B.M. + 1 hr at 2200F	6
25Mo	P.M. rod, swaged	27
30Mo	E.B.M. + 1 hr at 2200F	6
50Mo	P.M. rod, swaged	28
1ThO$_2$	P.M. rod, swaged	27
2ThO$_2$	P.M. rod, swaged	27
0.12Zr	A.M., extruded, and swaged	11
0.57Cb	A.M., extruded, and swaged	11
0.88Cb	A.M., extruded, and swaged	11
0.38TaC	P.M. rod, swaged 27 per cent + $\frac{1}{2}$ hr at 2910 F	29

FIGURE 4. EFFECT OF TEMPERATURE ON THE TENSILE STRENGTH OF TUNGSTEN ALLOYS

As indicated, most of the alloys show a significant strength advantage over unalloyed tungsten at temperatures to about 3500F. At higher temperatures, the only addition shown to improve the strength of tungsten is thoria, in amounts from 1 to 2 per cent.

Stress-rupture properties have been determined only for unalloyed tungsten and several thoriated alloys. These data are summarized in Figures 5 and 6. The Westinghouse work (3, 29) with thoria additions of 2, 4 and 5 per cent generally showed that, at 2500F, superior tensile and creep strengths were obtained in the W-2ThO$_2$ alloy, apparently as a result of a better thoria dispersion. This suggests that little further advantage would be gained by increasing the amount of thoria beyond 2 per cent.

Oxidation Behavior

Upon heating in air, tungsten begins to tarnish at around 570F, and starts to oxidize rapidly at temperatures in the range of 750F to 1000F. As long as an underlying protective lower oxide remains on the metal surface, the oxidation rate curves are parabolic. However, within a short time above about 1300F, complete conversion of the oxide to the yellow WO$_3$ occurs and oxidation rates become linear.

The oxidation rate of tungsten is senstive to increase in oxygen or water vapor partial pressure. Tungsten is, however, relatively insensitive to attack by nitrogen.

During heating in air, oxidation of massive tungsten is apparently confined to the oxide-metal intersurface, and internal oxidation or contamination does not appear to represent serious problems.

Few oxidation data are available on any wrought tungsten alloys. Simple tests at Climax (14) on binary tungsten-molybdenum alloys showed that molybdenum additions over the range of 2 to 40 per cent actually increase the oxidation rate of tungsten at 1750F. Thoria additions up to 4 per cent have no significant effect on the oxidation behavior of pure tungsten at 2500F (3).

It does not appear likely that any other dilute, wrought tungsten-base alloys will show significantly improved oxidation resistance over unalloyed tungsten. For this reason, coatings will almost certainly be required to adequately protect tungsten and its alloys in service in oxidizing environments at high temperatures.

EXTRUSION OF TUNGSTEN MATERIALS

Over the past few years, a sizeable amount of work has been done on the extrusion of unalloyed tungsten and tungsten alloys, both sintered and cast. A total of 10 organizations are known to have extruded arc cast billets of a variety of compositions. In addition, four organizations are known to have extruded tungsten materials at high velocities

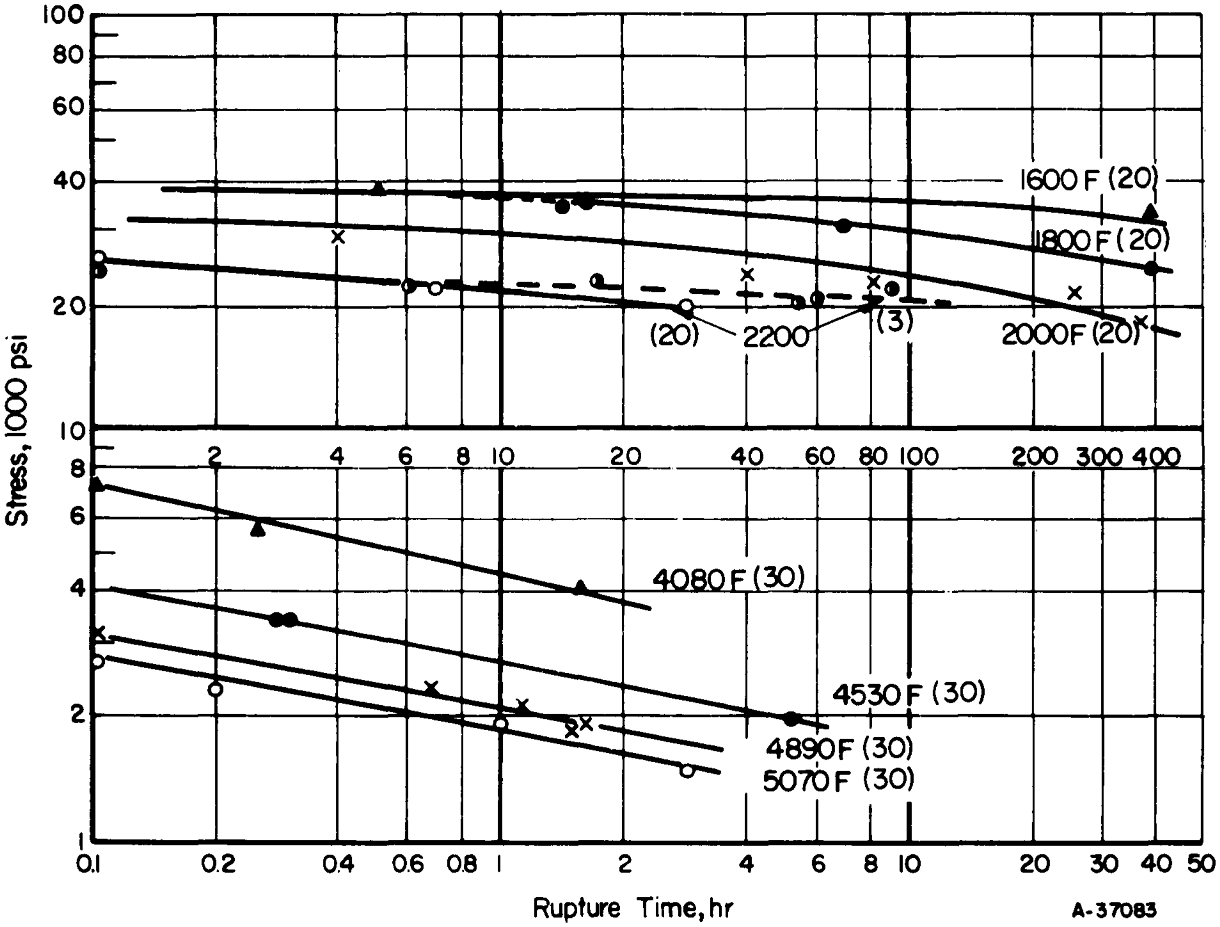


FIGURE 5. STRESS-RUPTURE PROPERTIES OF UNALLOYED TUNGSTEN ROD

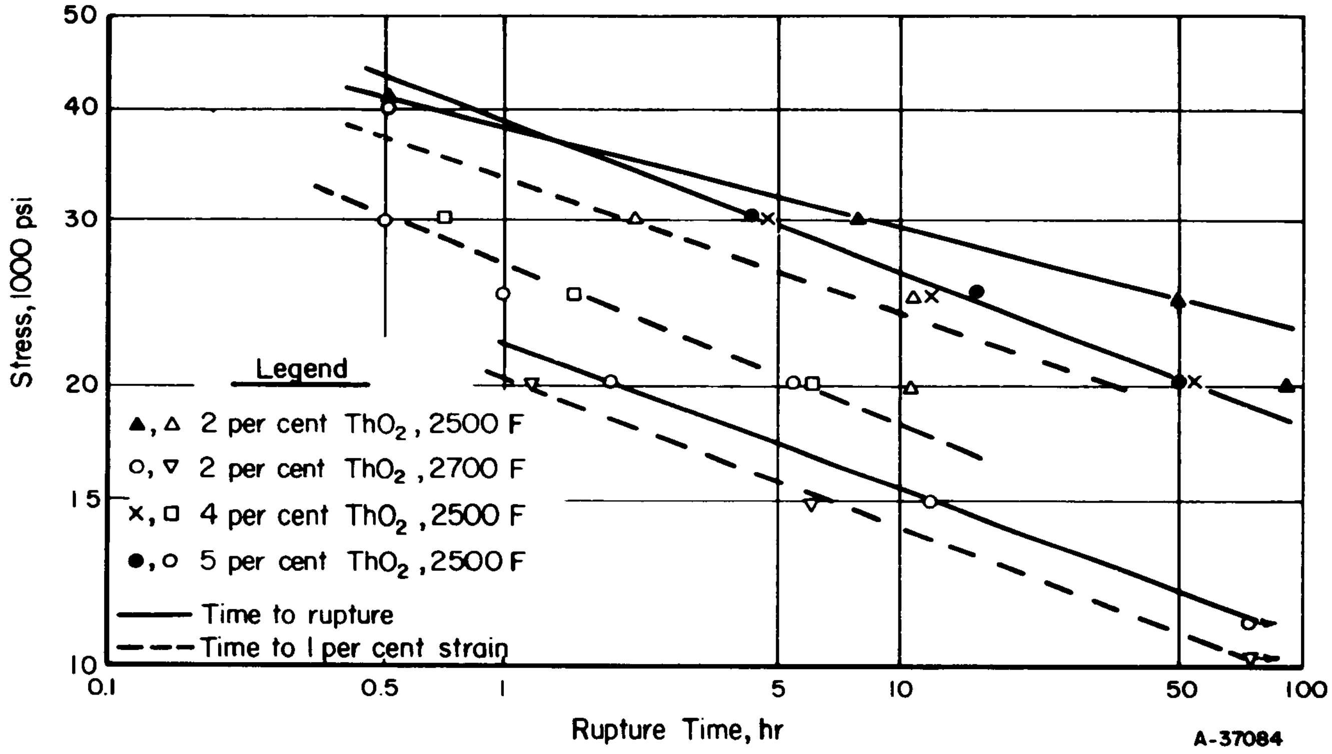


FIGURE 6. CREEP RUPTURE PROPERTIES OF THORIATED TUNGSTEN BAR ALLOYS(3)

on Dynapak machines. Table 13 lists these extruders and summarizes the work reported to date.

The extrusion of tungsten and tungsten alloys, however, must still be considered as experimental in its present state of development. All of the work on conventional presses has been on conversion of billets to simple rounds either as a primary breakdown operation or for the preparation of test materials.

Sintered billets of unalloyed tungsten, W-1ThO$_2$ and W-15 Mo have been extruded to rounds successfully at ratios up to about 6:1 at temperatures as high as 3900 F. This work has been done with billets up to 3 inches in diameter.

Arc-cast billets of unalloyed tungsten and a variety of alloys have been extruded to rounds at ratios up to about 8:1, using billet temperatures up to 4000F. This work has been divided about equally between unalloyed and alloyed tungsten. The alloys extruded have included mainly the "commercial" W-15 Mo alloy and a number of experimental W-Mo-base compositions. The largest cast billets extruded have been a 5-5/8-inch diameter x 20-inch billet of unalloyed tungsten (Canton Drop Forge) and a 5-1/2-inch diameter x 14-3/8-inch billet of W-5Mo alloy (Allegheny-Ludlum). Most of the other work reported has been with billets about 3-inches in diameter by about 6-inches long.

Extrusion of a 3.35-inch-diameter electron-beam melted billet of unalloyed tungsten was reported by General Electric (31). This was also the only report of the extrusion of a tungsten sheet bar. The billet was extruded to a 0.6 x 2.87 x 27-inch rectangular section (5:1 ratio) at 3000 F.

The major efforts in extrusion of tungsten materials have been made by Canton Drop Forge, Materials Central, and Tapco. Materials Central has probably been the most active in development of extrusion procedures.

Extrusion Practices

Only the information reported on extrusion of tungsten and tungsten alloys by conventional methods under established practices is discussed herein. Information and data on high-velocity extrusion on Dynapak equipment is not too applicable to this program, but is also included.

Several extrusion practices have been evolved and are in current use for extruding tungsten materials. The most pertinent of these are briefly outlined below:

TABLE 13.

ORGANIZATIONS THAT HAVE EXTRUDED TUNGSTEN BASE MATERIALS

Organization	Press Facilities[a]	Alloys Extruded[b]	Extrusion Conditions Ratio	Temp.F	Shapes
Allegheny Ludlum	1778 H 500 V	W-5Mo	2.1:1	2300	Rounds
Canton Drop Forge	3000 V 5500 H	100W	4:1	2350	Rounds
		W-10Mo	3:1	2350	
		W-15Mo	3:1	2350	
		W-30Mo	3:1	2350	
		W-50Mo	4:1	2350	
General Electric	1250 H	100 W	5.5:1	3000	Rounds
		100W (EB melted)	5:1	2980	Sheet - bar
Jet Propulsion Lab.	Dynapak 1800	100W	5.16:1	3500	Rounds
Materials Central[c] (Harvey Aluminum)	700 H	100W	4:1	3000	Rounds
		W-7Mo	5:1	3400	
		W-10Mo-0.01C	4:1	3000	
		W-15Mo-0.01C	4:1	3000	
		W-30Mo-0.01C	4:1	3000	
		W-50Mo-0.01C	4:1	2200	
		W-10Cb-0.01C	4:1	3400	
		W-2Ta-0.01C	4:1	3200	
		W-5Ta-0.01C	4:1	3200	
		W-15Mo-0.01B	4:1	3400	
		W-15Mo-0.05Zr-0.01B	4:1	3400	
		W-15Mo-0.5Cb-0.01C	4:1	3600	
		W-33Mo-0.5Ta-0.02B	4:1	3200	
		W-33Mo-0.05Zr-0.01B	4:1	3200	
		W-0.6Cb	4:1	3400	
		W (sintered)	4:1	3000	
		W-1ThO$_2$ (sintered)	6:1	3400	

TABLE 13. (continued)

			Extrusion Conditions		
Organization	Press Facilities[a]	Alloys Extruded[b]	Ratio	Temp.F	Shapes
NASA	1020 V	100W	8:1	3400	Rounds
	Dynapak				
	1800	100W	7.4-45:1	3000-3800	
		100W (sintered)	9.5-35:1	3000-3500	
Nuclear Metals	1000 H	100W	4:1	2300	Rounds
			20:1 max.	3550	Rounds
Sylvania	Dynapak	100W			
	1800	100W (sintered)			
		100W (EB melted)			
Tapco	700 H	100W	5.5-8:1	2300-4000	Rounds
	150V	W-15Mo	6:1	4000	
		W-0.5Mo	8:1	3700	
		W-2.5Mo	8:1	3700	
		W-12Mo	8:1	4000	
		W-25Mo	8:1	3500	
		W-1Ta	N/A	N/A	
		W-3.5Ta	N/A	N/A	
		100W (sintered)	4-6:1	2300-3500	
		W-15Mo (sintered)	5.5:1	3900	
Westinghouse	700 H	100W (sintered)	N/A	N/A	Rounds
	Dynapak	100W	N/A	N/A	Sheet bar
	1210	W-Re	N/A	N/A	

(a) Number indicates capacity in tons, H = horizontal, V=Vertical

(b) All material is arc cast except where indicated.

(c) Most of the extrusion work on W at Materials Central has been for other organizations, including Climax Molybdenum, Universal-Cyclops, Republic Aviation, General Electric, and Manufacturing Laboratories

Canton Drop Forge	(1) Salt bath heating, 2350 F-max. (2) Grease lubrication (3) Die material and design proprietary
Materials Central (also Tapco, NASA	(1) Induction heating under inert atmosphere (2) Glass-coated, tapered-nose billets (3) Alumina-coated conical dies (4) Follower block behind billet
General Electric Tapco, NASA	(1) Induction heating under inert atmosphere (2) Billet jacketed in molybdenum, columbium, or steel (3) Glass lubrication
Allegheny Ludlum	Ugine-Sejournet glass-lubricant process: (1) Glass coated billets (2) Inert atmosphere heating (3) Glass pad lubrication of die (4) Flat dies with radiused orifice.

The most reliable techniques for extrusion at temperatures of 3000 F or above have included the use of (1) induction heating under inert atmosphere, (2) billets precoated with glass, and (3) dies flame sprayed with alumina, as developed by Materials Central. Recent modifications at that laboratory have involved the use of zirconia-coated dies, tapered steel nose blocks ahead of a radiused billet, and steel follower blocks instead of graphite.

The only information on extrusion of tungsten by the Sejournet glass-lubricant process from any of the sources contacted during the survey was the ingot breakdown extrusion by Allegheny Ludlum. The successful extrusion of the target Tee section in molybdenum at Allegheny Ludlum, however, should serve as a guide for extrusion of a tungsten-base material in this configuration by the Sejournet glass-lubricant process.

Billet Preparation and Inspection

The preparation of cast billets involves cutting off the top and bottom of the ingot and lathe turning the surface to remove all defects. The cutting of tungsten is best done with an abrasive cut-off wheel following the recommendations of the abrasive manufacturers.

The machining of tungsten is accomplished with some difficulty. Low machining speeds produce a fine, pulverized chip and cause considerable chatter of the tool. High speeds produce a dark, red-hot continuous chip which is desirable, but care must be taken to prevent welding of the chip to the tool. Typical cutting speeds for rough machining are 100-150 spfm with feeds of .005 - .020-inch per revolution.

Turning operations are best accomplished without lubrication using grade C-2 carbide tools. Throwaway carbide inserts set in negative rake tool holders are generally used for single point turning. A typical tool geometry for tungsten uses back and side rake angles of -5 degrees with end and side relief angles of 5 degrees.

Grinding is recommended for finish machining. Silicon carbide wheels (60 grit) are preferred. Best results are obtained with light wheel pressures and flooding of the workpiece with a water-soluble detergent type coolant.

In recent years, several new processes for removing material have been developed for processing such materials as carbides, ceramics, and high-strength and heat-resistant alloys, which are difficult to cut. These techniques include:

(1) Electrical-discharge machining
(2) Electrolytic grinding and machining
(3) Ultrasonic machining
(4) Electron-beam machining
(5) Plasma-arc cutting.

A summary and bibliography prepared by Boulger (32) describes these processes and indicates potential application of one or more to metal removal of tungsten.

There are no data available on either the optimum surface finish requirements for extrusion billets or on what effect the finish has on the surface quality of the extrusion. It is expected that such an effect does exist, but no specific data has been reported.

The best inspection techniques appear to be a combination of ultra-sonic methods for checking internal defects plus one of a variety of penetrant methods (check, zyglo, etc.) for checking surface quality. Tapco (33) has now set up ultrasonic standards for internal inspection of arc-cast billets. Varying size voids were drilled in dummy billets and ultrasonic measurements taken for use in evaluating soundness of regular ingot material. Frequencies and other variable adjustments are set to the maximum sensitivity possible, which is just below the level at which grain size effects interfere with soundness measurements. This limits the ability to detect very small material defects, but the over-all process has worked satisfactorily.

Normal practice has been to extrude the top end of the ingot first since grain orientation tends to curve up in this direction toward the center of the ingot, simulating the natural metal flow in extrusion. The only work reported on the actual evaluation of the effect of grain orientation on extrusion was by Harvey (34). Two Mo-base alloys were extruded - one top end first, the other bottom end first. The latter showed severe nose bursts but a lower breakthrough pressure than the billet extruded with the ingot top as the nose end. These results are being investigated further to evaluate the effect on recrystallization and reworking of the material.

Billet Heating

The recent Materials Advisory Board Survey Reports (35,36) and the molybdenum-extrusion survey by Santoli (37) have both pointed up the lack of adequate refractory metal heating facilities, particularly for production extrusion presses. This is understandable inasmuch as nearly all of these present heating facilities were designed for the extrusion of steel, copper, etc. Santoli's compilation (37) of present facilities from the standpoint of type of heating media available and maximum temperature possible is given in Table 14.

For the temperature ranges believed necessary in this program (3000 F and above), induction heating was indicated by most extruders to be a definite requirement. Billet size capacities of induction heating units reported in this survey are indicated in Table 15.

TABLE 14.

SUMMARY OF BILLET HEATING FACILITIES (Reference 37)

Source	Status	Type of Equipment	Maximum Temperature (F)	Atmosphere Added	Remarks
A	Production	Induction	2400	Yes	Special induction coil to 3500 F
B	Production	Salt	2300	---	Special induction coil to 3000 F
		Induction	2300	Yes	
		Muffle furnace	1900	Yes	
C	Laboratory	Salt	2300	---	---
		Induction	3500	Argon	
		Muffle furnace	3200	Hydrogen	
D	Production	Salt	2300	---	---
E	Production	Salt	2300	---	Installing induction to 3500 F
F	Laboratory	Muffle furnace	3360	Hydrogen	---
G	Laboratory	Air furnace	2300	---	---
		Salt bath	2300	---	---
		Induction	4200	Argon	
H	Production	Air-gas furnace	3500	---	---
I	Job Shop	Induction	4500	---	---
J	Laboratory	Induction	4800 ± 300	Vacuum	---
K	Laboratory	Induction	3800	Argon	Maximum billet preheat temperature - 3400 F for extrusion

TABLE 15. CAPABILITIES FOR HEATING TUNGSTEN BILLETS BY INDUCTION

Company	Maximum Billet Size	Maximum Temperature, F
A	4-inch diameter x	4000
	1-3/4" diameter x 4" long	3100
B	4" diameter x 6" long	4000
C	5" diameter x 6" long	3600
D	3-1/8" diameter x 12" long	4000

In addition, two large induction-heating facilities -- one at Curtiss Wright, for billets up to 28 inches in diameter x 60 inches long -- the second at du Pont, for billets up to 9 inches in diameter, are now under construction.

The extrusion work reported to date on tungsten indicates that the combination of induction heating in a protective atmosphere with glass-coated billets is the best technique for heating billets. The fast opening units now in use at Materials Central and Tapco provide rapid heating plus the ability to get the billet out of the unit and into the press container within a few seconds. Other methods of heating under inert atmosphere may also be applicable, but the shorter heating time required by induction is favored since little information is available on the reactivity of coating glasses with tungsten base materials.

Lubrication

At the temperatures required for extrusion of tungsten structural shapes, the work reported thus far indicates glass will provide the best lubrication during extrusion. The application of glass coatings to the billet in combination with the alumina-coated dies have produced the best results to date in extrusion of tungsten or tungsten alloys(34). The use of glass pads for the die as practiced by Sejournet should be of even greater benefit for extruding a tungsten Tee section. Graphitic lubricants have little effectiveness at these temperatures, other than to prevent the glass from adhering to the container liner.

Thus, the role of glass in extrusion of tungsten will, of necessity, be a major one. One of the problems with present commercial glasses is their lack of sufficient viscosity above 2800 - 3000 F.

This can be seen in Figure 7 which shows the effect of temperature on the viscosity of several typical glass compositions. Above 3000 F, very little viscosity data is available and then only for a few of the glass compositions shown.

Tooling Materials and Design

The predominant materials used for press tooling are the hot work tool steels such as Types H11, H12, H13 and H21. These are generally designed and heat treated for maximum stem stresses of 180,000 - 200,000 psi. With the exception of die design, extrusion tooling is very similar in design and application among the extruders surveyed. A summary of the data obtained on tooling materials used in extruding tungsten or tungsten alloys is given in Table 16.

Die design and die material for the extrusion of tungsten structural shapes have not yet been evaluated. However, the extrusion of refractory metals for ingot breakdown strongly indicates that the standard tool steel extrusion die is not applicable at extrusion temperatures much above 2400 F - 2500 F, even with glass coated billets. The use of flame-sprayed, alumina coatings on dies has been most successful to date for extrusion temperatures up to about 3400 F from the standpoint of die life. Three pushes per coating -- 35-50 pushes per die have been reported.

Further refinements in application of the coating and design of the die are being made. The application of alumina by the plasma-jet process shows promise of improving adhesion of the alumina to the die. The use of higher melting point refractories such as zirconia have only been investigated recently, but look highly promising for extrusion temperatures above 3400 F. Zirconia-coated dies were used in two recent extrusion trials conducted by Harvey Aluminum at Materials Central on Mo-25W-0.1Zr-0.2C alloy rounds at 3600 F with an 8:7:1 ratio and at 4125 F ·

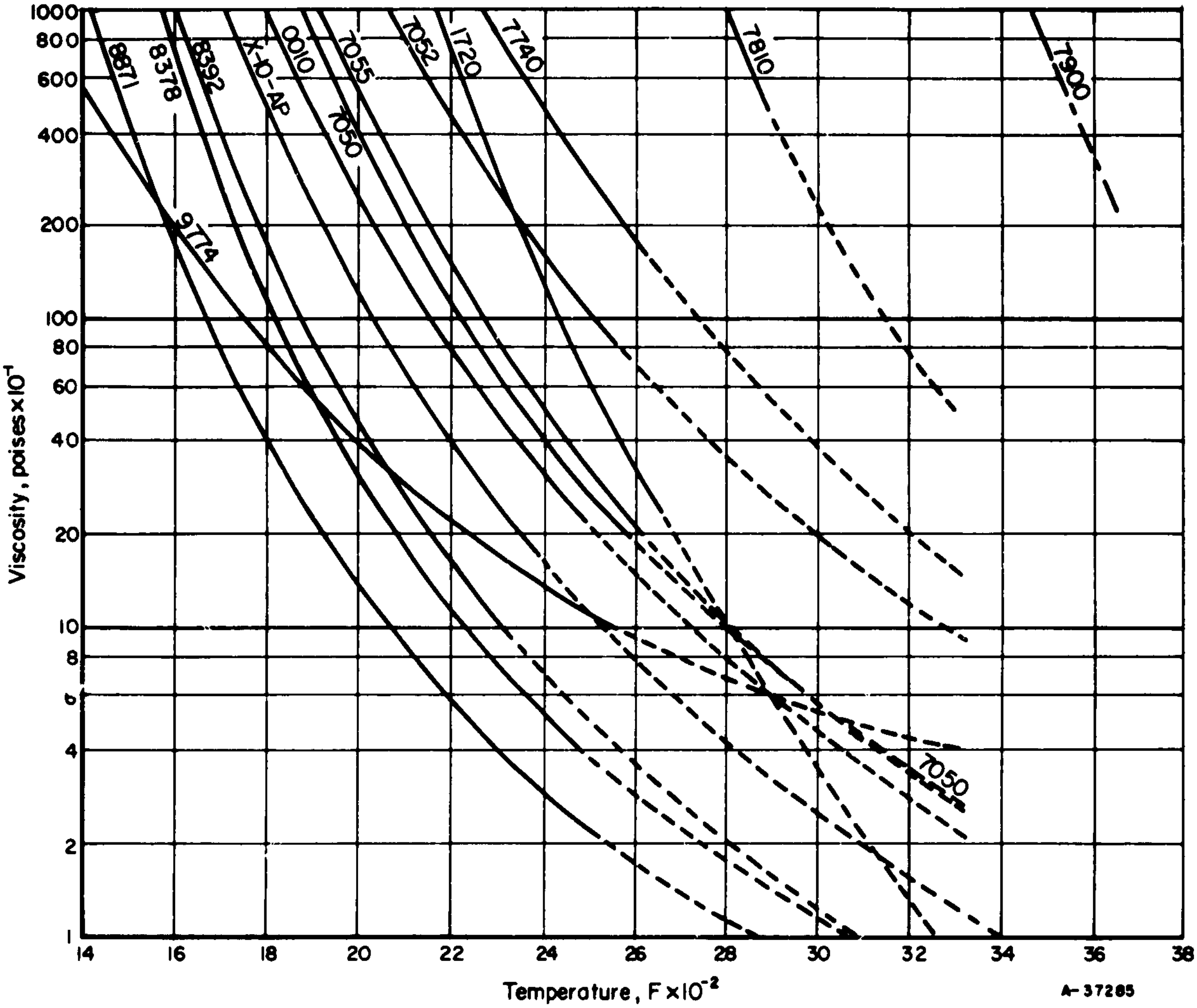


FIGURE 7. VISCOSITY VERSUS TEMPERATURE OF SOME COMMERCIAL GLASSES BELOW THE WORKING POINT

(Courtesy Corning Glass Works)

TABLE 16.

SUMMARY OF DATA ON TOOLING MATERIALS USED IN EXTRUSION OF TUNGSTEN

	Tooling Materials, AISI Type					
Company	Stem	Container Liner	Die	Dummy	Type of Press	Maximum Allowable Stem Stress, psi
A	H12	Ajax 4-2	H11, H21	---	1020 ton V	180,000
	H11	4340	4340, 01	H21	1800 Dynapak	N/A
B	H13	H12, H13	H21	N/A	1780 ton V	190,000
C	H13	4340	H13	N/A	700 ton H	200,000
D	H11	H11	Carbide	Graphite	1800 Dynapak	N/A
E	H11	H11	H11	Armco Iron	1800 Dynapak	200,000
F	H12	H11, H12	Stellite(a)	N/A	3000 ton V 5500 ton H	N/A
G	H12	CMV	Alumina(b)	N/A	700 ton H	196,000

(a) Applied as a hardfacing on H-12 tool steel

(b) Applied by flame or plasma spraying as coating on H-13 tool steel with an 8.1:1 ratio. The dies, coated with 0.030 to 0.050 inch zirconia, showed only slight wear--0.010 to 0.014 inch, and were reusable without recoating. The billets used in these trials were coated with glass.

It is generally agreed that uniform metal flow must be achieved for satisfactory extrusion of refractory metals. There is quite a variation of the die design used in the extrusion of rounds. Conical dies with included angles of 90 to 120 degress have been used with success, but no single conical design has been proven best as yet.

Two alternatives appear possible for extrusion of the Tee section:

(1) A contoured, conical design based on the use of glass-coated billets and alumina-or zirconia-faced dies.

(2) An essentially flat-faced design based on the use of the Sejournet glass-lubricant procedures, wherein a pad of glass is positioned between the billet and die to provide a lubricant reservoir.

It could well be that alumina or zirconia facings on the dies will be required in the latter method also to achieve satisfactory die life at the high temperatures anticipated.

Consideration should also be given to the possible use of entirely new die materials. Allegheny-Ludlum is currently doing some work along these lines (37) with vanadium-modified H13 tool steel, cast cobalt and nickel base alloys, and with tungsten carbide coatings. Extrusion trials with molybdenum dies at both Allegheny(37) and H. M. Harper(37) resulted in upsetting of the die at extrusion pressures of 170,000 - 180,000 psi. Tapco(38) has machined an unalloyed tungsten die but no extrusion has been made.

It is expected that the development work now underway at Allegheny-Ludlum for extrusion of the molybdenum Tee section will serve as a good basis for the extrusion of the tungsten Tee section.

EXTRUDABILITY OF TUNGSTEN AND TUNGSTEN ALLOYS

Table 17 presents extrusion conditions and pressure requirements for extrusion of cast tungsten and tungsten alloy billets. Unsuccessful

TABLE 17. EXTRUSION DATA FOR TUNGSTEN-BASE MATERIALS

Company	Material[(1)]	Billet Size Diameter x Length	Extrusion Ratio	Die Design[(2)]	Lubricant	Extrusion Temp. F	Maximum Ram Speed, ipm	Extrusion Pressure, 1000 psi		Resistance to Deformation, K[(3)]
								Maximum	Minimum	
Allegheny Ludlum	W-5 Mo	5-1/2" x 14-3/8"	2.1:1[(4)]	A	Glass	2300	760	124	91.5	- -
Canton Drop	W	5-5/8" x 20"	3.8:1	B	Grease	2350	200	N/A	N/A	- -
	W	4-7/8" x N/A	2.9:1	B	Grease	2350	200	71.5	N/A	- -
	W-10Mo	5" x N/A	3:1	B	Grease	2350	200	92	N/A	- -
	W-15Mo	5" x N/A	3:1	B	Grease	2350	200	N/A	N/A	- -
	W-30Mo	5" x N/A	3:1	B	Grease	2350	200	N/A	N/A	- -
	W-50Mo	5-5/8" x N/A	3.8:1	B	Grease	2350	200	N/A	N/A	- -
General Electric	W	3-1/2" x 2-1/2"	3.18:1	N/A	N/A	3000	N/A	84	N/A	- -
	W	3-1/2" x 2-1/2"	5.5:1	N/A	N/A	3000	N/A	108	N/A	- -
	W(EB)	3-11/32" x 6"	5:1 Rect.[(5)]	C	Glass	2980	360	135	124	- -
Jet Propulsion Lab.	W	1-3/8" x 2"	13.5:1	D	None	3500	26000	- -(Dynapak)- -		- -
	W	15/16" x 1-9/16"	16:1	D	None	3500	N/A	- -(Dynapak)- -		- -
Materials Central	W[(7)]	3" x 6-1/2"	4:1	E	Glass	2800	N/A	167	N/A	121000
	W	3" x 5-7/8"	4:1	E	Glass	2800	N/A	155	N/A	112000
(Harvey Aluminum)	W	3" x 5-7/8"	4:1	E	Glass	2800	N/A	153	N/A	110000
	W	3" x 6"	4:1	E	Glass	2800	N/A	170	N/A	122500
	W	3" x 4-7/8"	4:1	E	Glass	3000	N/A	170	N/A	122500
	W	3" x 5-1/2"	5:1	E	Glass	3000	N/A	150	N/A	93500
	W	3" x 6-7/16"	6:1	E	Glass	3000	N/A	187	N/A	104000
	W[(8)]	2-7/8" x 4-31/32"	4:1	E	Glass	3000	N/A	125	N/A	90500
	W	2-3/8" x 5-17/32"	4:1[(9)]	E	Glass	3000	N/A	133	N/A	96000
	W(s)[(10)]	3" x 5-1/4"	4:1	E	Glass	3000	N/A	155	N/A	112000
	W(s)	3" x 5-7/32"	4:1	E	Glass	3000	N/A	138	N/A	995000
	W-1ThO$_2$(s)	3" x 5-7/8"	4:1	E	Glass	3100	N/A	156	N/A	112500
	W-1ThO$_2$(s)	3" x 5-1/2"	6:1	E	Glass	3400	N/A	145	N/A	81000
	W-7Mo[(11)]	2-7/8" x 2-7/8"	5:1	E	Glass	3400	N/A	124	N/A	77500
	W-7Mo	3" x 5-1/2"	5:1	E	Glass	3400	N/A	165	N/A	97000
	W-10Mo[(7)]	3" x 5-15/16"	4:1	E	Glass	2800	N/A	159	N/A	115000
	W-10Mo	3" x 6-1/4"	4:1	E	Glass	3000	N/A	146	N/A	105000
	W-10Mo	3" x 6-3/16"	4:1	F	Glass	3000	N/A	173	N/A	125000
	W-15Mo[(7)]	3" x 6-3/16"	4:1	E	Glass	3000	N/A	197	N/A	142000
	W-15Mo	3" x 5-7/16"	4:1	E	Glass	3000	N/A	167	N/A	120500
	W-15Mo	3" x 6-7/16"	4:1	E	Glass	3000	N/A	178	N/A	128000
	W-15Mo	3" x 5-1/8"	4:1	E	Glass	3200	N/A	190 (1/3 extruded)		137000
	W-15Mo	3" x 5-3/32"	4:1	E	Glass	3400	N/A	188 (1/10 extruded)		136000

Footnotes will appear at the end of the table.

TABLE 17. (continued)

Company	Material[1]	Billet Size Diameter x Length	Extrusion Ratio	Die Design[2]	Lubricant	Extrusion Temp. F	Maximum Ram Speed, ipm	Extrusion Pressure 1000 psi Maximum	Extrusion Pressure 1000 psi Minimum	Resistance to Deformation, K[3]
(Harvey Aluminum)	W-30Mo[7]	3" x 5-13/16"	4:1	E	Glass	2400	N/A	188	N/A	136000
	W-30Mo	3" x 5-5/8"	4:1	E	Glass	2600	N/A	178	N/A	128000
	W-30Mo	3" x 5-1/4"	4:1	E	Glass	3000	N/A	182	N/A	131000
	W-30Mo	3" x 5-3/8"	4:1	E	Glass	3200	N/A	142	N/A	102500
	W-30Mo	3" x 5-5/16"	5:1	E	Glass	2900	N/A	173	N/A	107500
	W-30Mo	3" x 5-1/8"	5:1	E	Glass	3200	N/A	174	N/A	108000
	W-50Mo[7]	3" x 5-3/16"	4:1	E	Glass	2000	N/A	168	N/A	121000
	W-50Mo	3" x 6-1/2"	4:1	E	Glass	2200	N/A	169	N/A	122000
	W-50Mo	3" x 4-3/8"	6:1	E	Glass	3200	N/A	204	N/A	114000
	W-10Cb	3" x 6"	4:1	E	Glass	3400	N/A	172	N/A	124000
	W-2Ta	3" x 6-1/16"	4:1	E	Glass	3200	N/A	150	N/A	108000
	W-2Ta	3" x 6-7/16"	4:1	E	Glass	3200	N/A	149	N/A	107500
NASA	W	3" x N/A	8:1	G	Glass-Grease	3400	240	102	102	49100
	W(s)	1-21/32" x N/A	10:1	G	Glass-Grease	3500	64000	- (Dynapak)	-	- -
	W(s)[12]	1" x 1"	16:1	A	None	3500	N/A	- (Dynapak)	-	- -
	W	1" x 1"	40:1	A	None	3800	N/A	- (Dynapak)	-	- -
	W	1" x 1"	45:1	A	None	3800	N/A	- (Dynapak)	-	- -
Nuclear Metals	W(s)[13]	2-1/4" x 3-1/2"	4:1[14]	N/A	Glass-Grease	2300	70	152	N/A	- -
	W(s)	2-1/4" x 3-1/2"	5.35:1	N/A	Glass-Grease	2300	145	150	N/A	- -
	W[15]	3" x 4-1/2"	4:1[16]	N/A	N/A	2300	20	86	84	- -
	W	3" x 4-1/2"	6:1	N/A	N/A	2300	20	72	N/A	- -
Tapco	W-15Mo	3" x 6"	5.5:1	H	Grease	4000	7600	102	102	60000
	W-15Mo	3" x 6"	6:1	H	Grease	4000	7600	110	97	61500
	W-15Mo	3" x 6"	6:1	H	Grease	4000	640	124	106	69000
	W-15Mo	3" x 6"	6:1	H	Grease	4000	480	156	133	87000
	W-15Mo(s)	3" x 6"	6:1[17]	H	Grease	3800	600	170	129	95000
	W-15Mo(s)	3" x 6"	5.5:1	H	Grease	3900	600	142	115	83000
	W[13]	2-1/8" x 2-3/4"	5.5:1[14]	H	Grease	2300	70	128	N/A	- -
	W	2-1/8" x 2-3/4"	5.5:1	H	Grease	2300	70	132	N/A	- -
	W	1-3/16" x 2-1/2"	5.5:1[18]	H	Grease	3100	94	104.5	N/A	- -
	W	1-3/16" x 2-1/2"	8:1	H	Grease	3100	94	110	N/A	- -
	W	1-3/8" x 2-1/2"	5.5:1	H	Grease	3200	94	113	N/A	- -
	W	1-3/8" x 2-1/2"	8:1	H	Grease	3200	94	126	N/A	- -
	W-0.5Mo[13]	2" x N/A	8:1	N/A	Grease	3700	300	138	N/A	66500
	W-2.5Mo	2" x N/A	8:1	N/A	Grease	3700	330	129	N/A	62000
	W-12Mo	2" x N/A	8:1	N/A	Grease	4000	310	129	N/A	62000
	W-12Mo	2" x N/A	8:1	N/A	Grease	4000	370	135	N/A	65000
	W-25Mo	2" x N/A	8:1	N/A	Grease	3500	270	138	N/A	66500
	W-25Mo	2" x N/A	8:1	N/A	Grease	3500	240	144	N/A	69500
Westinghouse	W	4" x N/A	N/A	I,D	Grease	N/A	N/A	N/A	N/A	- -
Allegheny Ludlum	Mo[19]	3-23/32" x 4-9/16"	10.9:1 Tee	J	Glass	2850	450	157	141	59000
		3-23/32" x 6-1/16"	10.9:1 Tee	J	Glass	2850	325	157	143	60000

Footnotes will appear at the end of the table.

FOOTNOTES FOR TABLE 17.

1. Materials designated by (s) sintered, (EB) electron-beam melted, all others consumable-electrode arc cast.

2. Die nomenclature given as follows:
   - A. Flat-faced, uncoated
   - B. Proprietary conical design-hardfaced (stellite)
   - C. Rectangular opening 3/4" x 2-7/8"
   - D. 90 degree Conical, uncoated
   - E. 90 degree, Al_2O_3 coated
   - F. 110 degree Conical, Al_2O_3 coated
   - G. 90 degree, 120 degree, 130 degree conicals used - no indication which were used here
   - H. 120 degree conical - Al_2O_3 coated
   - I. 130 degree conical, uncoated
   - J. Flat, 0.315 x 1.24 x 2.48-inch Tee

3. K = resistance to deformation. Due to insufficient reporting of minimum extrusion pressures under equivalent extrusion conditions, the maximum pressure p has been used to calculate K. $K = \frac{P\ max}{\ln\ Extrusion\ Radio}$

4. Billet canned in stainless steel.

5. Molybdenum clad electron-beam melted ingot.

6. Similar tests made at 5:1 and 10:1 extrusion ratios.

7. This series extruded for Climax Molybdenum.

8. This series extruded for Universal Cyclops.

9. Billet canned in steel jacket with steel nose plug.

10. This series extruded for General Electric.

11. This series extruded for Republic Aviation.

12. This series extruded <u>at</u> Dynapak Convair.

13. This series extruded for NASA.

FOOTNOTES FOR TABLE 17. (Continued)

14. This series all canned in stainless steel. Overall extrusion ratio given.

15. This series extruded for Union Carbide.

16. Billets canned first in stainless steel - then mild steel. Actual billet diameter = 1-3/4".

17. Both sintered billets canned in steel.

18. Entire series canned in columbium. Overall extrusion ratio given.

19. Wrought and recrystallized billet.

extrusion trials, etc., have been deleted from this table inasmuch as they are described in detail in the data referenced throughout this report. Data from Allegheny-Ludlum for extrusion of the target Tee in molybdenum are included for comparison.

The data from Materials Central, which has also extruded a number of molybdenum and tantalum alloys under essentially the same conditions, probably gives the best indication of the relative extrudability of tungsten materials. At temperatures of about 3000 F, for example, unalloyed tungsten and tungsten molybdenum alloys require higher extrusion pressures than either molybdenum or tantalum alloys. Extrusion pressures for unalloyed tungsten ranged from 153,000 to 170,000 psi at 4:1 ratio. It is interesting that the work done at Materials Central has required higher extrusion pressures than that done either at General Electric or Canton Drop Forge. It may be that the 90 degree die angle used at Materials Central is not optimum for extruding tungsten materials. Recent work at Materials Central has shown some very marked effects on extrusion parameters with the substitution of boron for carbon in certain zirconium bearing tungsten base alloys. This data is summarized below:

Alloy	Extrusion Ratio	Extrusion Temp., F	Maximum Press Load Tons	Result
W-0.1Zr	4:1	3600	718	Stuck
W-0.05Zr-0.003C	4:1	3000	660	Stuck
W-0.05Zr-0.01C	4:1	3400	670	Stuck
W-15Mo	4:1	3400	665	10% extruded
W-15Mo-0.01B	4:1	3400	511	100% extruded
W-15Mo-0.05Zr-0.01C	4:1	3200	665	Stuck
W-15Mo-0.05Zr-0.01B	4:1	3400	515	100% extruded

The alloys containing additions of only zirconium or zirconium and carbon exhibited very poor extrudability. Severe cracking was observed with these materials even on initial upset. With boron additions, however, brittle fracture did not occur and complete extrusion was obtained at rather low extrusion pressures.

Two other comments on the results of the Materials Central extrusion work should be made. The extrusion procedure used at WADD is probably the most consistent and reproducible process developed to date. However, break through extrusion pressures show considerable variation under identical conditions. This situation may merely indicate the critical nature of the entire process at these high temperatures, or it may suggest variation in billet quality or purity.

NASA has investigated the widest variety of extrusion conditions in their early work at Nuclear Metals, Tapco, and Dynpak Convair. Both arc cast and sintered billets have been extruded using no lubricant at all as well as canning, glass, and grease lubricants. Extrusion ratios have varied from 4:1 to 45:1 at ram speeds of 70 ipm to over 60,000 ipm. Although sufficient data under any one set of conditions has not been generated to indicate any advantage of one process or technique over another, this work does represent a significant contribution to present tungsten extrusion technology.

Extrusion of the tungsten Tee at ratios between 11:1 and 17:1, as proposed, will obviously require considerable modification in extrusion practice, particularly the billet condition, extrusion temperature, lubrication, and die design. With cast billets, temperatures near 4000 F will probably be required. Although not under ideal conditions, Tapco's experience in extruding a Ta-10W alloy Tee substantiates this. An arc-cast ingot was extruded at 3950 F at an extrusion ratio of 16:1 Maximum extrusion pressure was 165,000 psi.

Allegheny-Ludlum has just completed initial Tee section extrusion trials on the molybdenum extrusion program with good success. A photograph of the first extrusions made are shown in Figure 8. Surface finish was in the range of 125-300 rms. Prior billet history involved extrusion from an 11-inch diameter ingot (cleaned up from 12-inch diameter arc-cast ingot) to a 6-inch billet, which was then cleaned up, recrystallized, and rolled at 2250° F to 3-3/4-inch-thick-bar. Extrusion billets were then obtained from this material.

Resistance to Deformation

Any consideration of the extrudability of a material must involve the resistance to deformation or K factor, which is an expression for the ease of extrusion of the material. The goal in this program is the extrusion of a tee section of the following specifications:

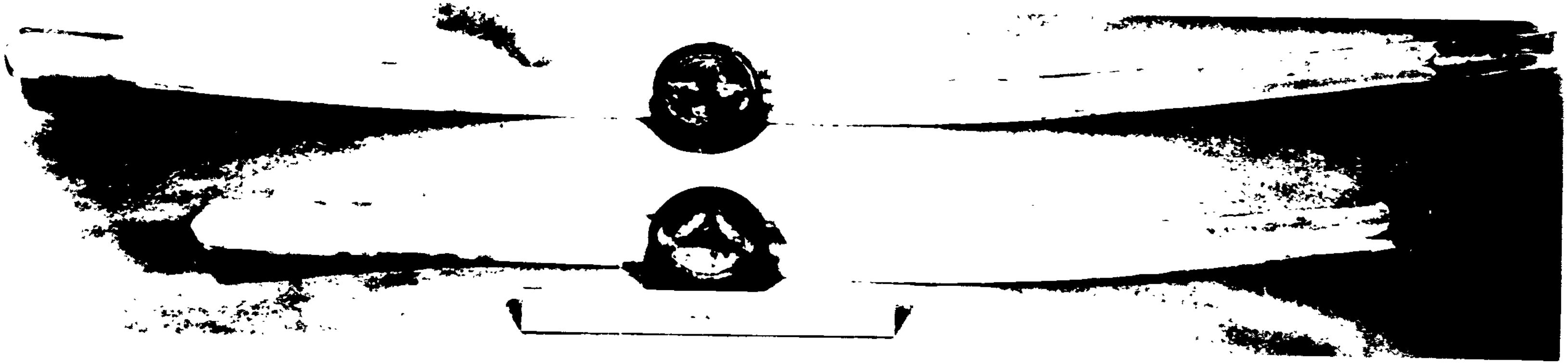


FIGURE 8. UNALLOYED MOLYBDENUM TEE SECTIONS EXTRUDED AT 10.9:1 REDUCTION RATIO

(Courtesy Allegheny Ludlum Steel Corp.)

(1) Flange width equal to two times the section depth
(2) Flange and stem thickness of 0.250 inch
(3) Circumscribed circle of 2 inches in diameter

Santoli's work(37) has indicated that this Tee configuration will result in a 17:1 reduction ratio from the contemplated container size of 3.875-inches in diameter. With a maximum stem stress value of 190,000 psi and ram-container clearance with 0.150 inch, the maximum liner pressure possible will be about 176,000 psi. Subsequent calculations showed that:

(1) At a K value of 50,000 psi, the target could be achieved with a 4-inch-long billet and a friction coefficient of 0.03 or an 8-inch billet with a friction value of 0.01.

(2) At a K value of 70,000 psi, there appeared to be little chance of achieving the target shape regardless of billet length or lubrication.

Thus, a "target" K value of 50,000 psi would seem to be a good yardstick in determining the extrusion conditions necessary for producing the target shape.

The calculation of resistance to deformation can be obtained from Sejournet's (39) equation, $p = K \ln A^\circ/A1 \ e \ 2fL/r$. The term $e \ 2fL/r$ expresses the resistance to billet movement along the liner wall only. The resistance to metal movement in the die itself is then incorporated in the K factor. In the absence of accurate values for the coefficient of friction, the best method for calculation of the K factor is as follows:

(1) When the billet length $L = O$, liner wall friction, f, also $= O$, and the extrusion pressure, P, is at a minimum.

(2) Thus, $P_{min.} = K \ln \frac{A^\circ}{A1} \ e^\circ$

or

$$K = \frac{P_{min.}}{\ln \frac{A^\circ}{A1}} = \frac{P_{min.}}{\ln \text{ extrusion ratio (R)}}$$

While this method for calculation of the K factor is recommended, the majority of the data which is reported on tungsten extrusion states only the maximum or break through extrusion pressure. Thus, the curves of K versus temperature developed in Figure 9 are based on the equation $K = \frac{P \text{ max.}}{\ln R}$ and are approximately 10 to 20 per cent higher than would be shown if the preferred calculation could be made.

Figure 9 shows the curves for unalloyed tungsten and the W-15Mo alloy with miscellaneous single K values also included for the modified W-15Mo alloy with boron additions. From the limited tests made, these alloy additions show a very favorable effect on the workability of the W-15Mo alloy. There is quite a variation between the K values developed for W-10Mo at WADD in the temperature range of 3000-3400 F and those obtained by Tapco at 4000F. However, the WADD work was done with a 90° conical die and the Tapco extrusion with a 120° die. K values based on maximum pressures required for extruding the Allegheny and Tapco Tee sections are also included in Figure 9.

Extrapolation of the present curve for unalloyed tungsten substantiates the earlier statement that temperatures in the order of 4000 F will be required to extrude the target Tee shape.

Preworking of Extrusion Billets

Although there has been considerable speculation that extrudability can be improved by using a preworked and recrystallized billet in place of a cast billet, there are no specific data available to resolve the question. Experience in forging has shown that even a partially worked and recrystallized billet exhibits better workability than cast material. Also, both the columbium and molybdenum extrusion programs are using preworked and recrystallized stock for extrusion of the Tee shape.

POST EXTRUSION FINISHING

The only post extrusion processes reported for tungsten which may be applicable for finishing a structural section are those for drawing, primarily of filament wire.

After swaging at temperatures of 2200-2900 F, tungsten is readily drawn through carbide and diamond dies to wire as small as

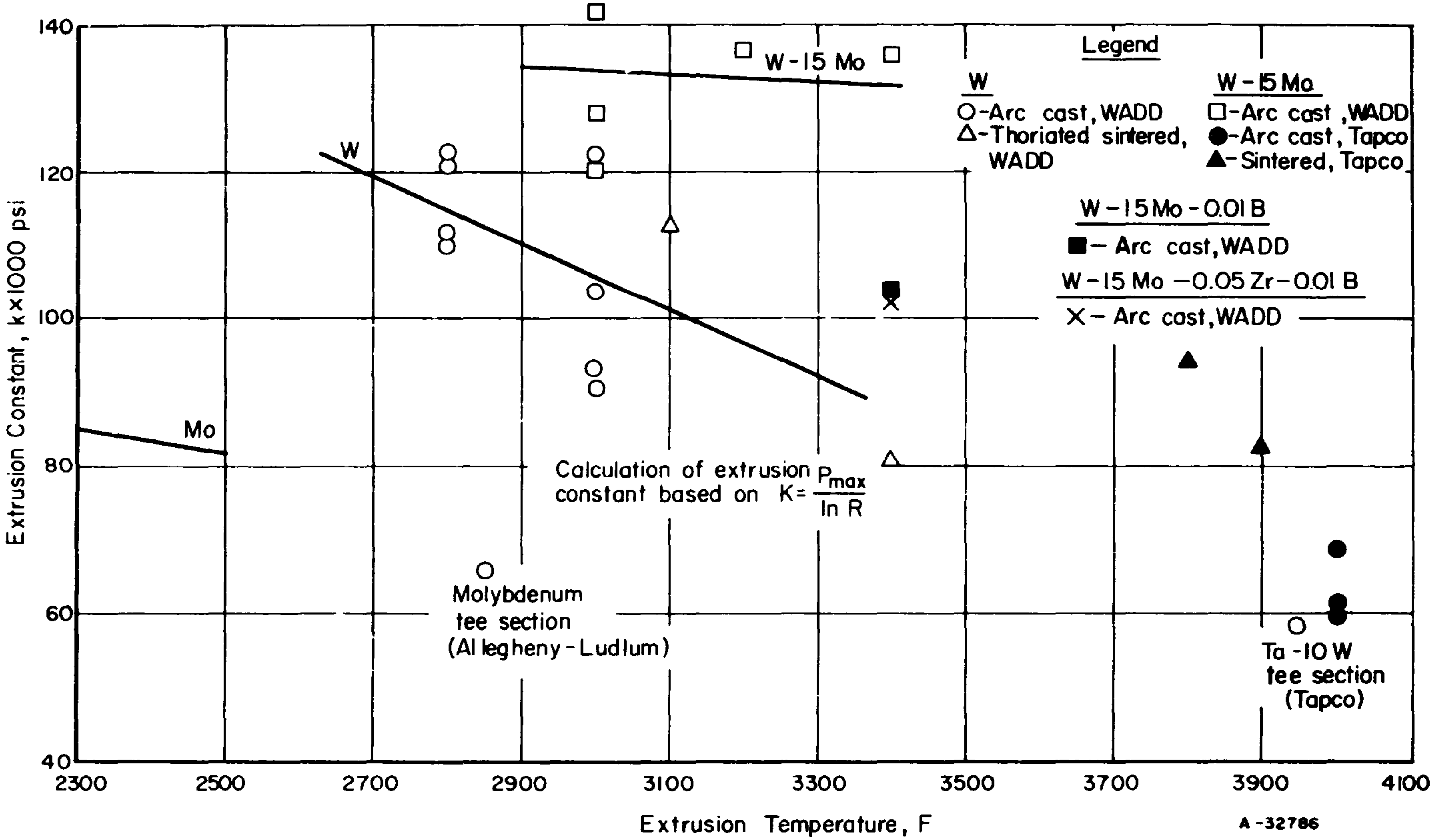


FIGURE 9. EXTRUSION CONSTANT FOR MISCELLANEOUS TUNGSTEN-BASE MATERIALS AT VARIOUS TEMPERATURES

0.0004 inch diameter(40). Table 18 taken from Li's book on Tungsten(41) illustrates a typical wire drawing schedule. As working progresses, lower die temperatures and faster drawing speeds are possible because of the progressive strengthening of the wire during drawing. Both Smithells (42) and Li and Wang(41) give complete details on drawing procedures.

No information is reported on the degree of surface contamination of tungsten extrusions or on methods for pickling or cleaning the extruded surface. Methods for cleaning tungsten have been developed, however, and several techniques are listed below:

(1) Immersion in 20% sodium hydroxide solution for 15 minutes

(2) Electrolyze in 0.1N sodium hydroxide solution with tungsten as the anode

(3) Immersion in boiling 5% solution of hydrogen peroxide (27%)

(4) Immersion in 5 nitric-3 sulfuric-2 water solution followed by a rinse in chromic acid.

The application of these techniques to extruded material has not been made.

Current practice on extrusion run-out is to plunge the hot extrusion into a box or tube filled with some inert material, such as Silocel, etc., in order to slow cool the extrusion and protect it from the air. There is no data reported on the degree of possible contamination from these materials except for a Westinghouse report(44) that Vermiculite contaminated the extrusion during cooling. Thus, consideration should be given to possible means for preventing contamination after extrusion in the course of developing extrusion techniques.

TABLE 18.

TYPICAL WIRE DRAWING SCHEDULE FOR TUNGSTEN (Reference 41)

Wire Size Rating, mg/200 mm	Tensile Strength, g/mg wt	Die Temperature, C	Drawing Speed, ft/min.
190.0	--	--	--
163.5	59.6	650	145
140.0	61.3	650	145
120.0	62.8	650	162
105.0	63.6	650	178
93.0	64.7	650	178
80.5	66.9	600	196
69.0	68.6	600	210
58.8	71.6	600	230
50.8	73.1	600	230
45.2 (anneal)	50.0	600	260
40.0	51.5	600	260
35.59	52.5	600	290
31.23	54.3	600	320
27.92	56.2	550	320
23.67	57.6	550	320
21.66	58.8	550	320
18.34	60.5	550	320
16.42	62.2	550	320
14.09	64.2	550	320
12.54	64.9	550	320
11.50	66.1	550	320
9.60	68.5	550	320
8.52	71.3	550	320
7.63	73.0	550	320
6.53	74.1	550	320
5.44	76.9	500	320
5.06	77.0	500	320
4.67	78.7	500	320
4.22	79.4	500	320
3.60	81.6	500	320
3.00	83.3	500	320
2.60	84.1	500	320
2.30	87.1	500	320
1.92	90.3	500	320
1.72	93.3	500	320
1.42	96.7	450	320
1.26	99.0	450	300

Proposed Program for Phase II

Introduction

Phase II work is directed primarily towards the development and refinement of tungsten billet production processes to obtain a high integrity extrusion billet. An additional important aspect of this phase will be the establishment of tests and testing procedures to verify satisfactory uniformity of extrusion billets. The major emphasis will be on relating complete destructive macro examination of cast ingots with ultrasonic inspection results. A process will be developed for the production of high quality extrusion billets on a reproducible basis.

Consolidation

Approximately six small ingots of W-2Mo weighing fifty pounds each will be arc cast into a 4-1/2-inch diameter mold varying the following parameters to obtain optimum ingot quality.

(1) Electrode configuration
(2) Hydrogen versus vacuum sintered electrode stock
(3) Sintered electrode density
(4) Melting rates (amperage and voltage)
(5) The use of stirring coils
(6) Electrode polarity
(7) Any other parameters affecting ingot quality which may prove to be effective during the melting of these ingots.

All of the initial six ingots will be melted at a rate not to exceed 2-1/2 pounds per minute in order to obtain good purification. In addition, the vacuum near the top of the mold will be held at less than 100 microns.

The first six ingots will be inspected as follows: each ingot will be cropped top and bottom to obtain solid metal and a quarter inch slab will be then taken from the top and bottom and examined using the following procedures:

(1) Macro grain size determination
(2) Dye penetrant inspection
(3) Grain size and orientation determination
(4) Faces machined for chemical analysis at both top and bottom
(5) Hardness traverse across each slab
(6) In addition, the major body of the ingot will be machined smooth and dye penetrant inspected as well as ultrasonic inspected for internal defects.

After completion of the inspection of the slabs in the major portion of the ingot, each ingot will be quartered and each quarter-slab tested as follows:

(1) Visual examination for cracks
(2) Dye penetrant inspection for micro and macro cracks
(3) Longitudinal and transverse hardness readings
(4) Grain size determination
(5) Radiographic and ultrasonic inspection
(6) Chemical analysis at the centers, mid-radius and edges of sample pieces.

It is anticipated that examination of the initial six ingots will reveal an optimum melting practice for producing sound ingots. This practice will then be utilized to consolidate four additional 100-pound ingots for extrusion work. An additional 1000 pounds of ingot stock will be produced in the optimum fashion to be used for melt material to produce large diameter tungsten ingots. The large tungsten ingots will be extruded from a billet diameter of 6.55-inch with a reduction ratio of approximately 2.8:1. Minimum extrusion billet lengths shall be 8-inches, the initial ingot diameter will be 8-inches In the event that a good sidewall is obtained the cast diameter will be reduced to 7-1/4-inches thus reducing material required and reducing machining time to obtain extrusion billets.

It is anticipated that the large diameter tungsten ingots will be machined into extrusion billets and broken down at about 3500°F using glass lubrication. The extrusion will then be cropped to obtain sound material. Two small extrusion billets will be machined from each extrusion from large diameter ingots. The primary extrusion will be machined, ground, pickled, and inspected ultrasonically and radiographically. Metallographic samples will be obtained from the end sections to determine grain size and distribution and extrusion soundness.

Extrusion Work

It is anticipated that a total of eight final extrusions to round shape will be conducted under Phase II of this program. Four of the billets will be extruded from the as-cast condition utilizing a 3.70-inch diameter extrusion billet. The other four extrusions will be obtained from the initial extrusion of the 6.55-inch diameter billets. These also will be machined to 3.70-inch diameter round. These extrusions will be conducted at two temperatures, 4000°F, and a second temperature on the order of 3500°F.

The extrusions will also be conducted at two different reduction ratios of 12:1 and 18:1. It is anticipated that this preliminary extrusion work will point the way for further extrusions with optimum reduction ratios utilizing best starting material to produce the desired surface finish of a 125 RMS or better.

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TGB/FFS/DJM/AMS/FWB:mmg

APPENDIX

In the performance of the Phase I state-of-the-art survey, contacts through questionnaires and/or personal visits were made with industrial concerns, research organizations and government agencies. A breakdown of the number of questionnaires mailed out on the tungsten survey and the response thereto is given below:

SURVEY SUMMARY

Number of Questionnaires Mailed	191
Total Number of Replies	102
Number of Replies Containing Information	34*

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*Includes information received on plant visits which were not put on questionnaires

Personal visits were made to the following companies:

Company	Location
National Aeronautics & Space Administration	Cleveland, Ohio
Canton Drop Forging & Manufacturing Company	Canton, Ohio
Bridgeport Brass Company	Riverside, California
Climax Molybdenum Company of Michigan	Detroit, Michigan
Wolverine Tube Div., Calumet & Hecla, Inc.	Detroit, Michigan
Jet Propulsion Laboratory, California Institute of Technology	Pasadena, California
Hayes-Stellite Div., Union Carbide Corp.	Kokomo, Indiana
Fansteel Metallurgical Corporation	N. Chicago, Illinois
Stauffer Metals Company	San Francisco, California
Aerojet-General Corporation	Sacramento, California
Oregon Metallurgical Corporation	Albany, Oregon
Bureau of Mines, U. S. Dept. of Interior	Albany, Oregon
Wah Chang Corporation	Albany, Oregon
National Research Corporation	Cambridge, Massachusetts
Tapco Division, Thompson-Ramo Wooldridge Co.	Cleveland, Ohio
Materials Central, WADD	Dayton, Ohio
Allegheny-Ludlum Steel Corporation	Brackenridge, Pennsylvania
Westinghouse Electric Corporation	Monroeville, Pennsylvania

DISTRIBUTION

AMC Aeronautical Systems Center
ATTN: LMBML
Wright-Patterson Air Force Base
Ohio

Commander
Air Research & Development Command
ATTN: RDTDEG, Mr. Kniffen
Andrews Air Force Base
Washington 25, D. C.

Armed Services Technical Information Agency
Document Service Center
Arlington Hall Station
Arlington 12, Virginia

Bureau of Naval Weapons
Department of the Navy
ATTN: Mr. S. E. Sanfilipo, AE 155
Washington 25, D. C.

Chief, Bureau of Naval Weapons
Department of the Navy
Washington 25, D. C.

Mr. Harold Bernstein
Special Projects Office
Department of the Navy
Washington 25, D. C.

National Aeronautics & Space Administration
Lewis Research Center
ATTN: Mr. George Mandel, Chief, Library
2100 Brookpark Road
Cleveland 25, Ohio

National Academy of Science
National Research Council
Division of Engineering and Industrial Resources
ATTN: Mr. E. V. Bennett
Washington 25, D. C.

U. S. Atomic Energy Commission
Technical Information Services Extension
ATTN: Mr. Hugh Voress
P. O. Box 62
Oak Ridge Tennessee

Commanding Officer
ATTN: Mr. S. V. Arnold, Associate Director
Watertown Arsenal Laboratories
Watertown 72, Massachusetts

Wright Air Development Division
ATTN: WWRCEP
Wright-Patterson Air Force Base
Ohio

Wright Air Development Division
ATTN: WWRCMA
Wright-Patterson Air Force Base
Ohio

Advanced Technology Laboratories
Division of American Standard
ATTN: Mr. W. C. Wolff
Contracts Manager
369 Whisman Road
Mountain View, California

Aerojet General Corporation
P. O. Box 296
Azusa, California

Aerojet General Corporation
Solid Rocket Department
P. O. Box 1947
Sacramento, California

Allegheny Ludlum Steel Corporation
ATTN: Extrusion Plant
Watervliet, New York

Hubert J. Altwicker
Lebanon, Ohio

Aluminum Company of America
ALCOA Building
ATTN: Mr. R. W. Andrews
Pittsburgh, Pennsylvania

Armour Research Foundation of
Illinois
Institute of Technology
Metals Research Department
ATTN: Mr. Frank A. Crosley
3350 South Federal Street
Chicago 16, Illinois

AVCO Corporation
Research & Advanced Development
Division
ATTN: Mr. John V. Erickson, Manager
Contracts & Administrative
Services
201 Lowell Street
Wilmington, Massachusetts

Babcock & Wilcox Company
ATTN: Mr. James Barrett
Beaver Falls, Pennsylvania

Baldwin-Lima-Hamilton Corporation
ATTN: Mr. Fred A. Fielder
Philadelphia 42, Pennsylvania

Defense Metals Information Center
Battelle Memorial Institute
505 King Avenue
Columbus 1, Ohio

Boeing Airplane Company
ATTN: Mr. Vince A. Dornes, Manager
Manufacturing Development Section
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Seattle, Washington

Jet Propulsion Laboratory
California Institute of Technology
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General Dynamics Corporation
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Crucible Steel Company of America
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Curtiss-Wright Corporation
Metals Processing Division
ATTN: Mr. Reese Williams
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Curtiss-Wright Corporation
Wright Aeronautical Division
ATTN: Mr. R. J. Moran, Manager
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Wood Ridge, New Jersey

Douglas Aircraft Company, Inc.
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ATTN: Dr. T. E. Leontis
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E. I. DuPont De Nemours & Company, Inc.
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ATTN: Mr. F. M. Mahla
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North Chicago, Illinois

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AiResearch Manufacturing Division
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3851 Sepulveda Boulevard
Los Angeles 45, California

General Electric Company
Aircraft Gas Turbine Division
ATTN: Mr. G. J. Wile, Engineering Mgr.
Metallurgical Engineering Operations
Large Jet Engine Department, Building 501
Cincinnati 15, Ohio

Grumman Aircraft Engineering Corporation
Manufacturing Engineering
ATTN: Mr. W. H. Hoffman, Vice President
Plant 2
Bethpage, Long Island, New York

H. M. Harper Company
ATTN: Mr. E. A. Channer
Vice President - Sales
Lehigh Avenue & Oakton Street
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Harvey Aluminum, Inc.
ATTN: Mr. G. A. Moudry
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Jones & Laughlin Steel Corporation
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ATTN: Mr. F. M. McGuire, Jr.
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Lockheed Aircraft Corporation
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Manufacturing Methods Division
Burbank, California

Lockheed Aircraft Corporation
ATTN: Mr. Alfred Peterson
Manufacturing Methods Division
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Magnethermic Corporation
ATTN: Mr. J. A. Logan
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16555 Saticoy Street
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The Martin Company
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The Martin Company
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McDonnell Aircraft Corporation
Lambert - St. Louis Municipal Airport
ATTN: Mr. C. E. Zoller
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NORAIR Division
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Hawthorne, California

North American Aviation, Inc.
ATTN: Mr. Walter Rhineschild
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Los Angeles 45, California

Nuclear Metals, Inc.
ATTN: Mr. Klein
Vice President
Concord, Massachusetts

Oregon Metallurgical Corporation
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Vice President & Sales Manager
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Republic Aviation Corporation
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Director of Manufacturing Research
Farmingdale, Long Island, New York

Republic Steel Corporation
Republic Research Center
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Cleveland 31, Ohio

Reynolds Metals Company
Dayton Sales Office
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11 W. Monument Building
Dayton, Ohio

Rohr Aircraft Corporation
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Manufacturing Research
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Chula Vista, California

Ryan Aeronautical Company
ATTN: Mr. L. J. Hull, Chief Metallurgist
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Lindberg Field
San Diego 12, California

Sandia Corporation
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Sandia Base
Albuquerque, New Mexico

Sandia Corporation
Livermore Laboratory
ATTN: Mr. M. W. Mote, Jr.
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Solar Aircraft Company
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U. S. Bureau of Mines
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United Aircraft Corporation
Pratt & Whitney Aircraft
Division
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United States Steel Corporation
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Universal Cyclops Steel Corporation
Refractomet Division
ATTN: Mr. P. C. Rossin
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Chance-Vought Aircraft Corporation
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Wah Chang Corporation
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Westinghouse Electric Corporation
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QUESTIONNAIRE
ON THE
STATE-OF-THE ART SURVEY
ON
EXTRUSION OF TUNGSTEN

Prepared by Battelle Memorial Institute
Contract No. AF 33(600)-42395

Company ______________________________ Date ____________

SECTION I. ORGANIZATION

A. Please indicate your organization's role in the development, production, fabrication, or use of W or W alloys:

(1) Producer ________
  a. Powder ________
  b. Sintered Billet ___
  c. Cast Billet _____
  d. Other ________

(2) Fabricator _____
  a. Extruder ____
  b. Forger _____
  c. Other _____

(3) User ________
  a. Extrusions ____
  b. Forgings ____
  c. Sheet _____
  d. Other _____

(4) R&D ______________
  a. Alloy Development ____
  b. Billet Production ______
  c. Fabrication _________
  d. Application _________

B. If an extruder, what W shapes have you produced?

Shape	Application

C. If a user of W extrusions, what shapes and for what applications?

Shape	Application

NOTE: If your organization is a supplier of W products, will you please attach any literature available describing these products.

Completed by ______________________

Title ______________________

Company ______________________________ Date ______________

SECTION II. APPLICATIONS AND REQUIREMENTS FOR W EXTRUSIONS

A. Are you aware of any specific needs for W extrusions:

(1) At present? Yes ______ No ______
(2) In 3 years? Yes ______ No ______
(3) In 3-5 years? Yes ______ No ______

B. If yes to any of the above, please provide the following information:

	Unalloyed	Alloyed
(1) Applications (aircraft, missiles, etc.)		
(2) Shapes (cross section and lengths)		
(3) Minimum Property Requirements		
Ultimate tensile strength		
Yield tensile strength		
Elongation		
Reduction in area		
Creep strength		
Rupture strength		

C. If possible, please attach drawings or sketches of any components which you have listed above (Part B) that show dimensions, including section thicknesses and length of extrusion desired.

Completed by ______________________________

Title ______________________________

COMPANY ______________________________ Date ______________

SECTION III. BILLET PREPARATION - POWDER TECHNIQUES

A. Raw Materials

(1) Do you specify impurity level for the extrusion billets used?

Yes ____ (see below) No ___

	Tungsten Powder	Sintered Tungsten Billet
C		
H		
N		
O		
Cb		
Fe		
Ni		
Si		
Ti		
Others ____		
____		
____		
____		
____		

(2) What alloying additions have you investigated in extrusions?

Element	Amount Added, weight per cent	Form of Addition	Purpose of Addition

B. Consolidation Practice

(1) General procedures used:

Mechanical die pressing _______
Hydrostatic pressing _________
Others ____________________

(Continued)

SECTION III. (Continued)

(2) Details on pressing procedure:

Method	Pressure	Billet Shape	Dimensions of Largest Billet Produced	Density

(3) Sintering-furnace equipment:

			Atmosphere	
Type of Heating	Maximum Temperature	Billet-Size Capability	Vacuum Pressure	Inert Gas (Specify)

(4) Sintering details:

				Density	
Size of Billet	Time	Temperature	Atmosphere	Initial	Final

(5) What methods are used to measure density?

Method	Limitation

(Continued)

SECTION III. (Continued)

(6) What density variations are encountered on sintered billets?

Billet Diameter, inches	Density Variation, Surface to Center
<3	______
3-4	______
4-5	______
5-6	______
6-8	______
>8	______

(7) What inspection methods do you use for sintered billets?

a.	Penetrant methods	Yes ______	No ______
b.	Magnetic particle	Yes ______	No ______
c.	Ultrasonics	Yes ______	No ______
d.	Magnetic susceptibility	Yes ______	No ______

e. Other ______________________________

(8) Which inspection method is best and why?

____

____

____

Please list references to pertinent publications and Government reports. Where possible, attach copies of photographs and tabular data.

____

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Completed by ______________________

Title ______________________

Company ______________________________ Date ______________

SECTION IV. BILLET PREPARATION - ARC-CAST TECHNIQUES

A. Raw Materials

(1) Have you melted tungsten ingots? Yes ______ No ______

Maximum ingot size: Diameter ________ Weight ____________

(2) Are you a supplier of tungsten ingot? Yes ______ No ______

Maximum ingot size: Diameter ________ Weight ____________

(3) Do you have a purity specification for extrusion billet?

Yes ______ (see below) No ______

Specified Impurity Levels in Melted Extrusion Billet

C ____________________
H ____________________
N ____________________
O ____________________
Cb ____________________
Fe ____________________
Ni ____________________
Si ____________________
Ti ____________________
W ____________________
Others ___ ____________________
___ ____________________
___ ____________________
___ ____________________

(4) What alloying additions have you investigated in extrusions?

Element	Amount Added, weight per cent	Form of Addition	Purpose of Addition

(Continued)

SECTION IV. (Continued)

B. Melting Practice

(1) General methods used:

	Dimensions of Largest Ingot Made		
Method	Diameter, inches	Length, inches	Weight, pounds
Consumable electrode			
Electron beam			
Others			

(2) What type of electrode materials are used?

	Electrode Materials Used	
Method of Melting	Sintered	Others
Consumable electrode		
Electron beam		
Others		

(3) What minimum electrode density is permissible for satisfactory consumable-electrode melting?

(Continued)

SECTION IV. (Continued)

(4) Does purification occur in melting?

Yes ______ (see below)   No ______

	Impurity Level. per cent			
	Melt Method ______		Melt Method ______	
	Ingot Diameter ______		Ingot Diameter ______	
Impurity Elements	Before Melting	After Melting	Before Melting	After Melting
C				
H				
N				
O				
Others ____				
____				
____				
____				

(5) What inspection method do you use for arc-cast billets?

(a) Penetrant methods	Yes ______	No ______
(b) Magnetic particle	Yes ______	No ______
(c) Ultrasonic	Yes ______	No ______
(d) Magnetic susceptibility	Yes ______	No ______

(e) Other ______________________________

(6) Which inspection method is best and why?

____

____

____

Please list references to pertinent publications and Government reports. Where possible, attach copies of photographs and tabular data.

____

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Completed by ______________________

Title ______________________

Company ______________________________ Date ____________

SECTION V. EXTRUSION

A. Extrusion Equipment

(1) What type of extrusion equipment do you have?

	Press 1	Press 2	Press 3
Type (Horizontal or Vertical)			
Max. Tonnage			
Max. Ram Speed, in./min			
Max. Billet Size (diam. x length)			

(2) What is the size of the press tooling?
- a. Container liner ______________________________
- b. Stem ______________________________
- c. Mandrel ______________________________
- d. Dies (circumscribing-circle diameter) ______________________________

(3) What types of tool steels or other materials are used for tooling?
- a. Container liner ______________________________
- b. Stem ______________________________
- c. Mandrel ______________________________
- d. Dies ______________________________

(4) What preheat temperatures are used for extrusion?
- a. Container liner ______________________________
- b. Stem ______________________________
- c. Mandrel ______________________________
- d. Dies ______________________________

(5) Under the conditions indicated for stems, what do you consider maximum permissible stress during extrusion? ______________________________ psi

B. Billet Preparation

(1) What types of W billets have you extruded?
- a. Sintered Yes ____ No ____
- b. Arc-cast Yes ____ No ____
- c. Other ______________________________

(2) If sintered, what density variations have you observed in the billets?

Billet Diameter, inches	Density Variation, Surface to Center
<3	
3-4	
4-5	
5-6	
6-8	
>8	

(3) What inspection methods do you use for sintered W billets?
- a. Penetrant methods Yes ____ No ____
- b. Magnetic particle Yes ____ No ____
- c. Ultrasonics Yes ____ No ____
- d. Magnetic susceptibility Yes ____ No ____
- e. Other ______________________________

(Continued)

SECTION V. (Continued)

(4) Which inspection method is best and why? ____________________

____

____

(5) What is the minimum W billet density acceptable for extrusion? ____________

(6) What billet configuration do you use for extruding W?
a. Size ____________
b. Nose Shape. Flat ____ Conical ____
c. Front corner radiused? Yes ____ No ____; if yes, how much ____________

(7) What surface finish is required on W billets? ____________ How does surface finish of billet affect quality of the extrusion? ____________

____

C. Billet Heating

(1) How do you heat W billets for extrusion?

	Maximum Temperature	Billet-Size Capability	Heat-Up Time
a. Salt bath	____	____	____
b. Muffle	____	____	____
c. Induction	____	____	____
d. Inert atmosphere	____	____	____

e. Other ____________________

____

(2) What protective coatings are used during heating W billets? ____________

____

____

(3) How much billet contamination occurs during heating?

Depth Below Surface, inches	Hardness, BHN
0	____
0.005	____
0.010	____
0.015	____
0.020	____
0.025	____
____	____
____	____

D. Billet Accessories

(1) Do you use the following items in the extrusion of W?
a. Dummy block Yes ____ No ____
b. Spacer (between dummy and billet) Yes ____ No ____
c. Lead-in cone (between billet and die) Yes ____ No ____

(Continued)

SECTION V. (Continued)

(2) If yes to any of the above, please supply the following details?

	Dummy Block	Spacer	Lead-In Cone
Size and shape			
Material			
Heat treatment			
Hardness			
Preheat temperature			

E. Lubricants

(1) What type of lubricant do you use for extrusion of W?

a. Grease Yes ____ No ____ Details ____________

b. Glass Yes ____ No ____ Details ____________

c. Canning Yes ____ No ____ Details ____________

d. Other ____________

(2) How is the lubricant applied? ____________

____________ To which parts? ____________

(3) Do the lubricants do a satisfactory job? ____________

(4) What is the typical surface finish obtained? ____________

F. Die Design

(1) What die designs have you used for extruding W?

Dimension	Die 1	Die 2	Die 3
Entrant angle			
Bearing radius			
Land length			
Relief angle			

(2) What has been the die life for the various die materials and die shapes used? ____________

(3) Will you please attach any drawings or sketches which might be available that would show the die designs used?

(Continued)

SECTION V. (Continued)

G. Extrusion Studies

(1) Will you indicate the results of your extrusion work on W?

Condition	Trial 1	Trial 2	Trial 3	Trial 4
Material				
Shape				
Billet temperature				
Extrusion ratio				
Ram speed				
Max. extrusion pressure				
Min. extrusion pressure				
Quality of extrusion				
Surface finish				

(2) What have been your major problems in the extrusion of W? (Die wear, surface, cracking, etc.)

(3) How have you altered extrusion variables to overcome extrusion defects?

(4) What do you think is the maximum extrusion ratio possible in extruding W?

What is the most practical minimum section thickness?

(5) What areas in the extrusion process do you think need the most emphasis in order to realize commercial production of W extrusions?

(Continued)

SECTION V. (Continued)

H. Post Extrusion

(1) Have you done any of the following operations on W extrusions?

a. Surface conditioning Yes ____ No ____
b. Straightening Yes ____ No ____
c. Heat treatment Yes ____ No ____
d. Drawing Yes ____ No ____
e. Swaging Yes ____ No ____
f. Rolling Yes ____ No ____

(2) If yes to any of the above, please describe below.

Completed by ______________________

Title ______________________

Company ________________________ Date ____________

SECTION VI. ALLOY DATA SHEET

Source ____________

Designation ________ Composition ________ Experimental ________; pilot plant ________;
commercial ________
Consolidation and fabrication ________________________

____
____
____
____

Tensile Data

Sample Condition ____________ ____________ ____________
Test Condition ____________ ____________ ____________

	1000 psi				1000 psi				1000 psi			
Test Temp, F	Y.S.	T.S.	% El.	% RA	Y.S.	T.S.	% El.	% Ra	Y.S.	T.S.	% El.	% RA

Stress-Rupture Data

Condition	Temp, F	Atmosphere	Stress, 1000 psi	Life, hours	% El.	% RA	Comments

Impact Data

Oxidation Data

Temperature, F	Time	Atmosphere	Weight Gain	Penetration, mils/side	Metal Loss, mils/side

(Continued)

SECTION VI. (Continued)

Comments (include pertinent references to publications or Government reports)

Completed by ____________________

Title ____________________