

10  
-19 C

OK  
DR-1949

LA-4752

MASTER

# High-Energy-Rate Forming of Refractory Metals

THIS DOCUMENT CONFIRMED AS  
UNCLASSIFIED  
DIVISION OF CLASSIFICATION  
BY J.H. Kohn / J.H. Lamb  
DATE 10/22/71



**Los Alamos**  
**scientific laboratory**  
of the University of California  
LOS ALAMOS, NEW MEXICO 87544



DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

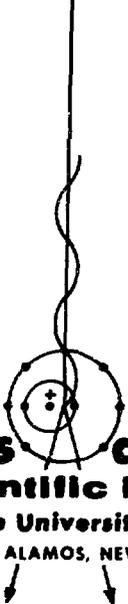
This report expresses the opinions of the author or authors and does not necessarily reflect the opinions or views of the Los Alamos Scientific Laboratory.

Printed in the United States of America. Available from  
National Technical Information Service  
U. S. Department of Commerce  
5285 Port Royal Road  
Springfield, Virginia 22151  
Price: Printed Copy \$3.00; Microfiche \$0.95

LA-4752

UC-25

ISSUED: September 1971

The logo features a vertical line with a wavy pattern that ends in a circular emblem. The emblem contains a stylized atomic symbol with a central nucleus and three orbiting electrons. Below the emblem, the text "Los Alamos scientific laboratory" is written in a bold, sans-serif font, with "Los Alamos" on the top line and "scientific laboratory" on the bottom line. Underneath this, in a smaller font, is "of the University of California". At the very bottom, "LOS ALAMOS, NEW MEXICO 87544" is printed. Two small arrows point downwards from the bottom corners of the text block.

**Los Alamos**  
scientific laboratory  
of the University of California  
LOS ALAMOS, NEW MEXICO 87544

# High-Energy-Rate Forming of Refractory Metals

by

Donald J. Sandstrom  
Dale S. Fisher

## NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

A handwritten signature or initials are present in the bottom right corner of the page, overlapping the "DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED" text.

# HIGH-ENERGY-RATE FORMING OF REFRACTORY METALS

by

Donald J. Sandstrom and Dale S. Fisher

## ABSTRACT

A variety of techniques for forming refractory metals, high-temperature alloys, and cermets are being investigated. Pneumatic mechanical forming, especially extrusion, has been used to produce a variety of shapes from refractory metal alloys and cermets. Using a conventional filled billet extrusion technique, sound, high-density W + UO<sub>2</sub> and Mo + U<sub>2</sub>O tubular products have been produced. Considerable attention has been given to the design of tools used in high-temperature extrusion processes and operational experience has been employed in the redesign of tools to increase their life. Attempts have been made, with limited success because of the difficulty, to instrument the extrusion press.

The application of magnetic fields to metal forming is receiving considerable attention. The magnetic field produced in a coaxial conductor carrying high, pulsed currents can be used to compact metallic powders into tubular shapes; to form tubular shapes not amenable to other joining processes; or as a technique for cladding regular or irregular shapes with suitable cladding materials. Refractory metal alloys, cermets, and refractory oxides have been formed using the theta magnetic field generated in a coaxial conductor. Comparisons of densities of materials compacted using this technique indicate an improvement over the conventional hydrostatic method. Leak-tight cladding of metals with other metals has also been accomplished.

## I. Introduction

The first atomic reactor that achieved criticality was designated CP-1, for Chicago Pile, and was fueled by a heterogeneous mixture of natural uranium and uranium dioxide in a matrix of graphite.

Modern nuclear reactors for rocket propulsion incorporate the same basic ideas with several refinements. The fuels have been upgraded and the quality of graphite has been significantly improved. However, the high core temperatures and conditions of both thermal and mechanical stress limit the effectiveness of graphite fuel elements.

Since increased power output requires, among other things, increased core temperature, the fabrication of fuel elements with a refractory metal alloy matrix holds considerable interest.

The processes that can be used to fabricate fuel elements must be capable of producing shapes with high L/D ratios. Applicable methods include conventional

extrusion, high-energy-rate extrusion, powder metallurgy extrusion from a slurry, rod rolling, or other powder metallurgy primary consolidation processes accompanied by conventional hot working. During the course of investigation, a new technique utilizing fast-rising magnetic fields for forming tubular shapes, both from solid and powdered metals, has been evolved.

## II. Pneumatic-Mechanical Extrusion

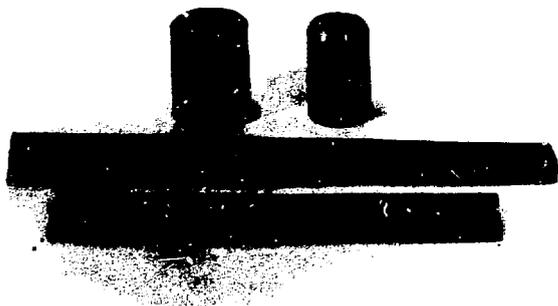
Many of the Laboratory programs require unusually shaped parts which must be capable of service at elevated temperature. The materials of greatest interest for nuclear fuel elements are tungsten, tungsten alloys, and composites. Cermet systems based upon molybdenum are also useful in several applications.

A technique frequently used for forming has been the pneumatic-mechanical process herein called Dynapak

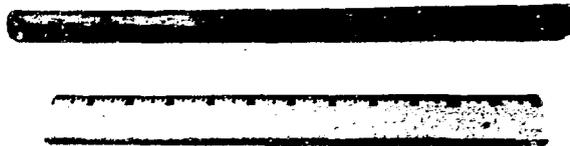
(trade name of the General Dynamics Corporation). Dynapak has been used primarily for extrusion studies, as opposed to forging operations, of refractory metals. The process implies advantages but also presents limitations that can be illustrated using tungsten. If extrusion ratios in excess of approximately 20:1 are used, the problem of inertial separation becomes quite critical. At press closure speeds in the range of 800 in./sec, the exit speed of the part being extruded at a 20:1 ratio is 16,000 in./sec, corresponding to an exit speed of 1300 ft/sec or Mach 1.3. At these speeds, and at elevated temperatures, the tensile load applied to the extruded piece exceeds the tensile strength of the material and failure occurs.

**A. Tungsten Extrusion.** Tungsten is considered a desirable structural material in several high-temperature nuclear environments in spite of its comparatively large neutron absorption cross section.

A variety of fabrication methods may be used to form tungsten and tungsten alloys into the desired configurations. Figure 1 shows four representative extrusions fabricated from either arc cast or compacted powder-sintered billets. Figure 1A shows a flat, bar extrusion made at a 12.1 extrusion ratio and 1850°C using arc cast starting material. Figure 1B is a rod made from arc cast material at a 16:1 extrusion ratio and 1850°C. Figure 1C is a ribbed sheet bar made from a high-density powder



A



B



C



D

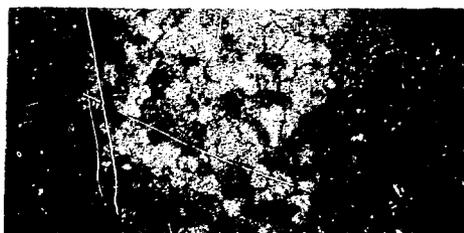
- A ARC CAST W BAR 1.375" x .187" EXTRUSION RATIO 12:1.
- B ARC CAST W ROD .750" DIA. EXTRUSION RATIO 16:1.
- C RIBBED W BAR EXTRUDED FROM A POWDER METALLURGY BILLET. EXTRUSION RATIO ~ 8.25:1.
- D SEAMLESS W TUBING 1" O.D. x .750" I.D. EXTRUDED FROM ARC CAST W. EXTRUSION RATIO 8:1.

*Fig. 1.*

*Representative extrusions of tungsten and tungsten alloys produced by the HER (Dynapak) process.*

compact. The severe edge cracking in Fig. 1C was attributed to the design of the shear die used. Presently all dies have an entry angle of  $120^\circ$  as determined from observations of the metal flow occurring in extrusions of ductile material through shear dies. Figure 1D shows extruded seamless tubing produced using conventional mandrel techniques. A solid billet was reduced from 2.5 to 2 in. diam to improve grain structure before piercing and extruding the billet. The initial extrusion of the billet to improve structure is no longer a standard procedure since extrusion ratios of at least 4:1 are necessary to cause recrystallization of arc-cast tungsten.

In general, finer grain size is achieved in material extruded from consolidated powder. Figure 2 represents typical microstructures developed in extruded high-purity tungsten produced from material consolidated by powder metallurgy techniques and by arc casting. The powder billet was isostatically cold pressed from powder that had an average particle size of 0.7 to  $0.8\mu\text{m}$ . The pressed billet was double-sintered at  $1700^\circ\text{C}$  in  $\text{H}_2$  for 3 h and at  $2200^\circ\text{C}$  for 3 h in vacuum to a density of 94% theoretical. The grain size of the powder-metallurgy material extruded at a 7:1 ratio and  $1850^\circ\text{C}$  was 0.06 mm; the hardness was found to be 365 DPH, typical of annealed material; and the extruded density was 100% of theoretical. The arc



EXTRUDED, PRESSED, AND SINTERED TUNGSTEN.  
EXTRUSION RATIO 7:1.  
GRAIN SIZE 0.06 mm.  
EXTRUSION TEMPERATURE  $1850^\circ\text{C}$ . 100X  
HARDNESS 365 DPH.



EXTRUDED ARC-CAST TUNGSTEN.  
EXTRUSION RATIO 11:1.  
GRAIN SIZE 0.10 mm.  
EXTRUSION TEMPERATURE  $1850^\circ\text{C}$ . 100X  
HARDNESS 365 DPH.

Fig. 2.

*Typical microstructures developed in extruded tungsten which utilized either powder metallurgy or arc-cast starting materials.*

cast material was extruded at the same temperature at a ratio of 12:1. The as-extruded grain size was 0.08 to 0.10mm and the hardness was 365 DPH. A highly worked structure was obtained in the extruded arc-cast material by rolling at  $850^\circ\text{C}$ . A total reduction of 65% was obtained with no evidence of failure.

The experience gained from the extrusion of tungsten is applicable to the extrusion of composites and cermets of ultimate interest for nuclear fuel elements.

**B. Filled Billet Extrusion Studies.** The extrusion of refractory metals provided valuable information applicable to the extrusion of cermets containing refractory metal alloy matrices. Factors of importance in this phase of the work included developing:

- A system containing sufficient loading of nuclear fuel, a minimum weight of the element, and adequate strength and ductility at operating temperatures.
- Suitable cladding techniques that would form a cladding impervious to the fissionable material and its reaction products.
- A procedure that would give sufficiently long fuel elements.

The coextrusion process offers a technique that can perform the dual functions of forming and cladding and can yield a complete metallurgical bond with no evidence of discontinuity.

Two cermet systems have been examined at Los Alamos. Dickinson and co-workers studied a composition comprised of a tungsten matrix filled with 45 vol.%  $\text{UO}_2$  and clad with molybdenum.<sup>1</sup> The system studied by the authors was based upon a molybdenum matrix loaded with 40 vol.%  $\text{UO}_2$  and clad with tungsten.

In both cases, the cermet was made by hydrostatically pressing and sintering the blended powders. The powder used in the extrusion studies of the Mo- $\text{UO}_2$  system averaged  $4.5\mu$  Fisher APS and the as-canned density was 75 to 77% of theoretical. Figure 3 shows a sketch of the billet design chosen for filled billet extrusion. The structure of the extruded Mo + 40 vol.%  $\text{UO}_2$  shows that  $\text{UO}_2$  has significant ductility at temperatures as low as  $1450^\circ\text{C}$ . The extruded density is 10.47 gm/cc, essentially 100% of theoretical.

This technique is not completely satisfactory for all reactor applications because of the possible loss of fuel from the molybdenum matrix. However, this situation can be improved by using a multifilled billet design with tungsten cladding as shown in Fig. 4. This billet design has been used and extrusion studies performed. The data indicate that the extrusion must be performed at a minimum temperature of  $1650^\circ\text{C}$  to get uniform reductions throughout the filled extrusion. Figure 5 shows the type of bond developed between the Mo + 40 vol.%  $\text{UO}_2$  and tungsten cladding and between the tungsten and molybdenum. This bond appears to be metallurgical with no



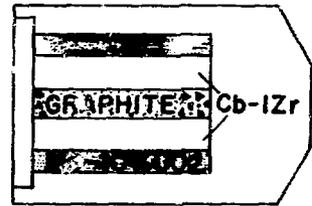
LONGITUDINAL MICROGRAPH OF  
Mo-UO<sub>2</sub> EXTRUDED AT 1450°C  
250x —UNETCHED



TRANSVERSE MICROGRAPH OF  
Mo-UO<sub>2</sub> EXTRUDED AT 1450°C  
250x —UNETCHED



MACRO OF EXTRUDED TUBE .435" O.D.  
.050" WALL.

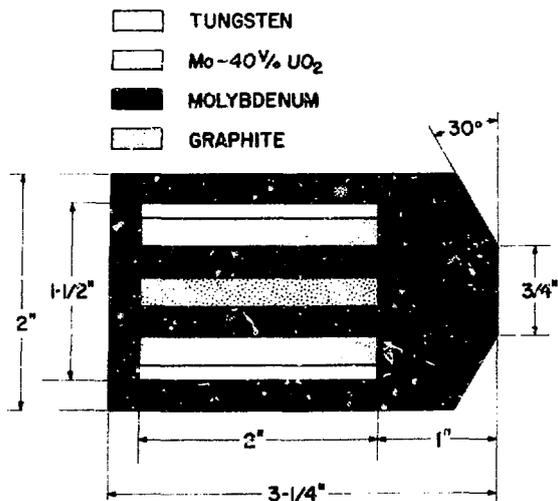


FILLED BILLET

Mo-40<sup>v</sup>% UO<sub>2</sub> EXTRUDED USING THE FILLED BILLET TECHNIQUE  
EXTRUSION WAS PERFORMED AT 1450°C USING AN EXTRUSION  
RATIO OF 8:1. FILLER MATERIAL - Cb-1<sup>w</sup>% Zr.

*Fig. 3.*

*Typical Microstructure developed in Mo + 40 vol% UO<sub>2</sub> extruded by the filled-billet techniques.*



FILLED BILLET FOR EXTRUSION OF W CLAD Mo-40% UO<sub>2</sub> TUBING

Fig. 4.

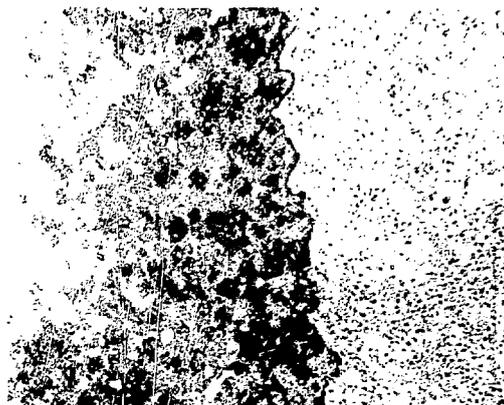
Filled billet used for extruding a clad Mo + 40 vol% UO<sub>2</sub> tube.

evidence of discontinuities for the full length of the extrusion.

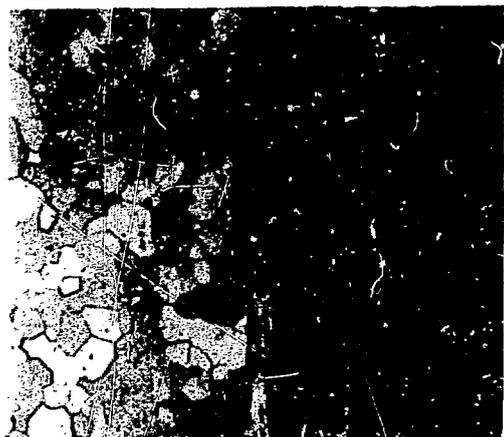
The pronounced directionality of the fuel matrix structure is not considered ideal from a nuclear viewpoint. Such directionality can result in localized hot spots and decidedly lamellar condition in the longitudinal direction. A second disadvantage lies in the obvious expense associated with the processing of such highly complex parts.

**C. A Comparison of High-Energy-Rate and Conventional Press Extrusion.** Several investigators have compared high-energy-rate extrusion of tungsten with conventional press extrusion of the material.<sup>2</sup> Some theoretical presentations of energy delivered by Dynapak have also been presented.<sup>3, 4</sup> Theoretical treatments to date neglect the restraint imposed by shock absorber mechanisms and assume that moving parts move on frictionless bearings. In all cases an impulse momentum analysis is made which pre-supposes knowledge of velocity or displacement of one or both active members.

An analysis of this motion has been undertaken using high-speed photography as a means of defining press motion during extrusion. Figure 6A is a plot of ram and bolster displacement versus time for the extrusion of annealed aluminum using a 4:1 extrusion ratio at room



INTERFACE LAYER SHOWING DIFFUSION BETWEEN CO-EXTRUDED MOLYBDENUM AND TUNGSTEN. TUNGSTEN AT LEFT OF PHOTOGRAPH.



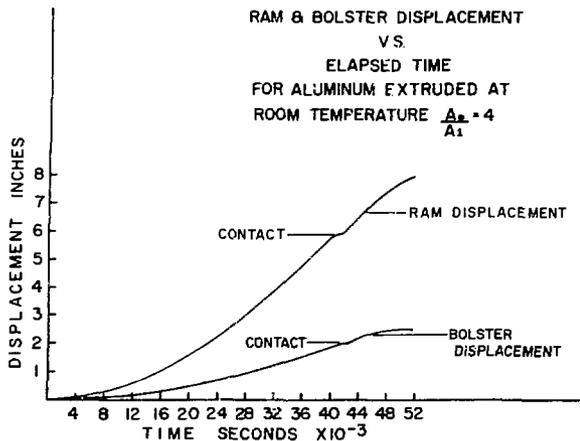
INTERFACE LAYER BETWEEN TUNGSTEN AND Mo + 40 vol% UO<sub>2</sub> SHOWING BONDING. (Mo + 40 vol% UO<sub>2</sub> on right) 250X

Fig. 5.

Typical microstructures of W-Mo and W-Mo + 40 vol% UO<sub>2</sub> coextrusions.

temperature. The photography was done at 6000 frames/sec. Figure 6B is a plot of velocities of the ram and bolster as determined by graphically differentiating the displacement-time curve. The results of such an analysis are significant in that they give actual ram and bolster displacement and velocity at the instant of impact.

The maximum extrusion pressure should be encountered at the instant just before the billet begins to exit through the die. At this point all upsetting should have occurred and the billet length and corresponding container wall friction should be maximum. Since the energy delivered to the billet is the sum of the kinetic energies of the individual members, the maximum energy occurs at the instant of impact when the velocity is a maximum.



RAM & BOLSTER DISPLACEMENT  
VS.  
ELAPSED TIME  
FOR ALUMINUM EXTRUDED AT  
ROOM TEMPERATURE  $\frac{A_2}{A_1} = 4$

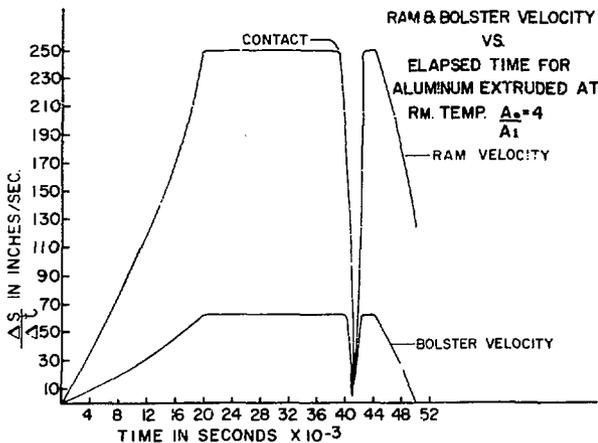


Fig. 6.

Displacement-time and velocity-time curves for the Dynapak machine.

The displacement-time curve indicates that at the instant of impact the ram has moved six in. and the bolster has moved approximately two inches. At this point the relative velocities of the ram and bolster are 310 in./sec.

Figure 7 is a chart comparing the manufacturers observed data with the experimental data obtained by high speed photography. Measuring the static distance between the ram and the point where billet contact is made yields a close approximation to the actual total ram travel. However, the amount of pressure at the instant of breakthrough will appear to be slightly higher than what is actually measured by high speed photography. This in itself yields interesting information in regard to extrusion

DYNAPAK ENERGY VELOCITY RELATIONSHIPS  
ENERGY-VELOCITY DATA BASED UPON MANUFACTURERS  
THEORETICAL DIAGRAM.

#### MACHINE PARAMETERS

RAM TRAVEL	TOTAL ENERGY (THEORETICAL)	RELATIVE VELOCITY
8" ( $X_r$ ) <sup>ⓐ</sup>	715,000 IN.-LBS.	320 IN./SEC.
10" ( $X_r + X_b$ ) <sup>ⓑ</sup>	875,000 IN.-LBS.	370 IN./SEC.

ENERGY-VELOCITY DATA BASED UPON HIGH SPEED  
PHOTOGRAPHY.

RAM VELOCITY AT CONTACT	=	250 IN./SEC.
BOLSTER VELOCITY AT CONTACT	=	63 IN./SEC.
RELATIVE VELOCITY AT CONTACT	=	313 IN./SEC.

$$\text{ENERGY AT CONTACT} = \frac{1}{2} M_r V_r^2 + \frac{1}{2} M_b V_b^2$$

$$E_T = 653,000 \text{ IN.-LBS.}$$

ACTUAL RAM TRAVEL  $X_r$  6

ⓐ  $X_r$  = RAM TRAVEL

ⓑ  $X_r + X_b$  = RAM + BOLSTER TRAVEL

Fig. 7.

Comparison of theoretical data vs data developed by high speed photography for Dynapak extrusions.

constants for tungsten.

Figure 8 is a comparison of extrusion constants for tungsten extrusion using conventional high-speed extrusion and high-energy-rate techniques. The extrusion constant for the high-energy-rate extruded stock was calculated using the formula developed by Rabenold and co-workers.<sup>2</sup> This formula is

$$\frac{E}{L(A)} = K \ln \frac{A}{A_1}$$

where E = the kinetic energy,  
L = billet length before extrusion,  
A = starting cross-sectional area,  
 $\frac{A}{A_1}$  = extrusion ratio,

and K = extrusion constant in psi.

In the calculation of the extrusion constant using the Dynapak, the ram travel was measured as the *free-fly*

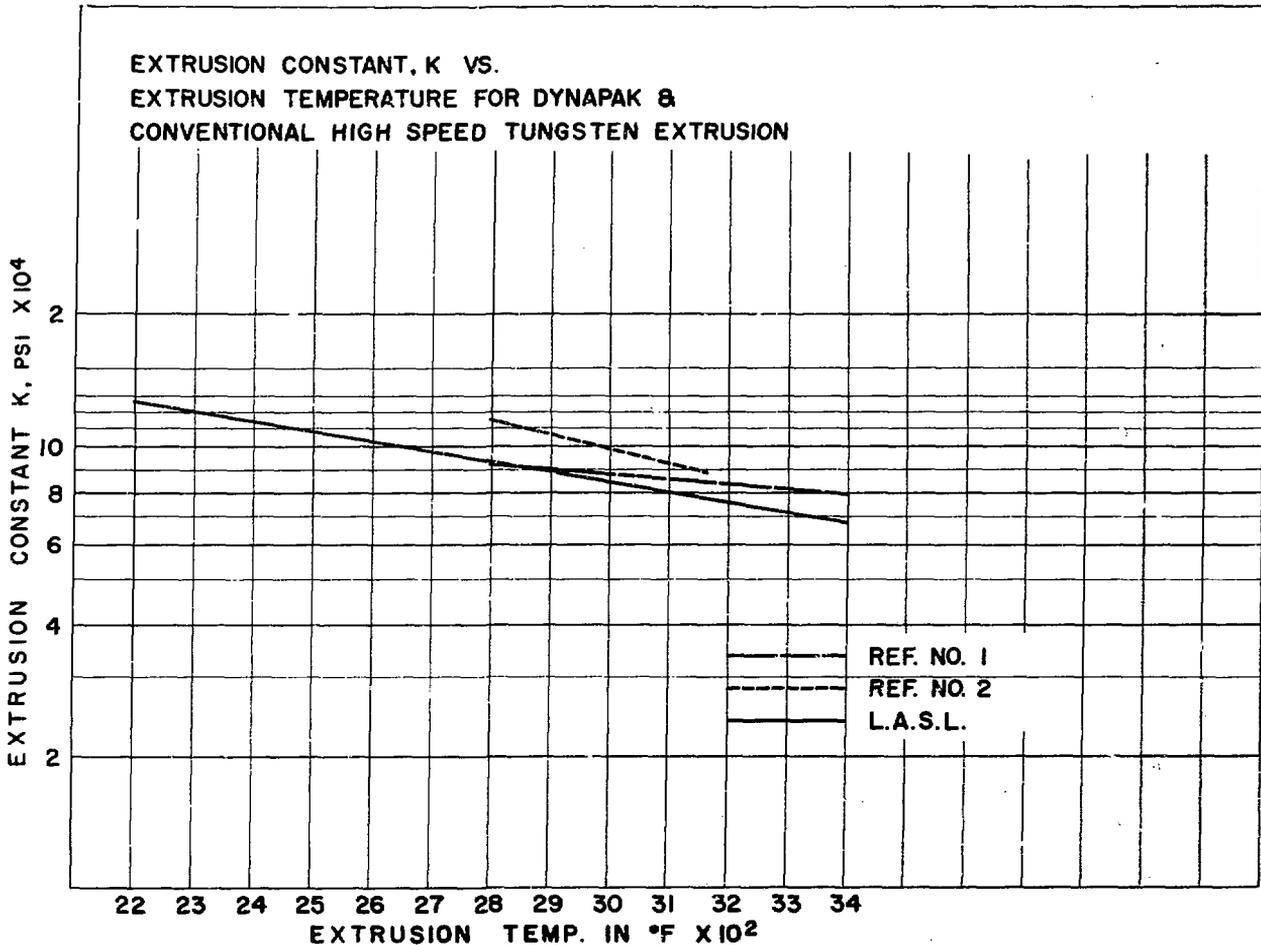


Fig. 8.

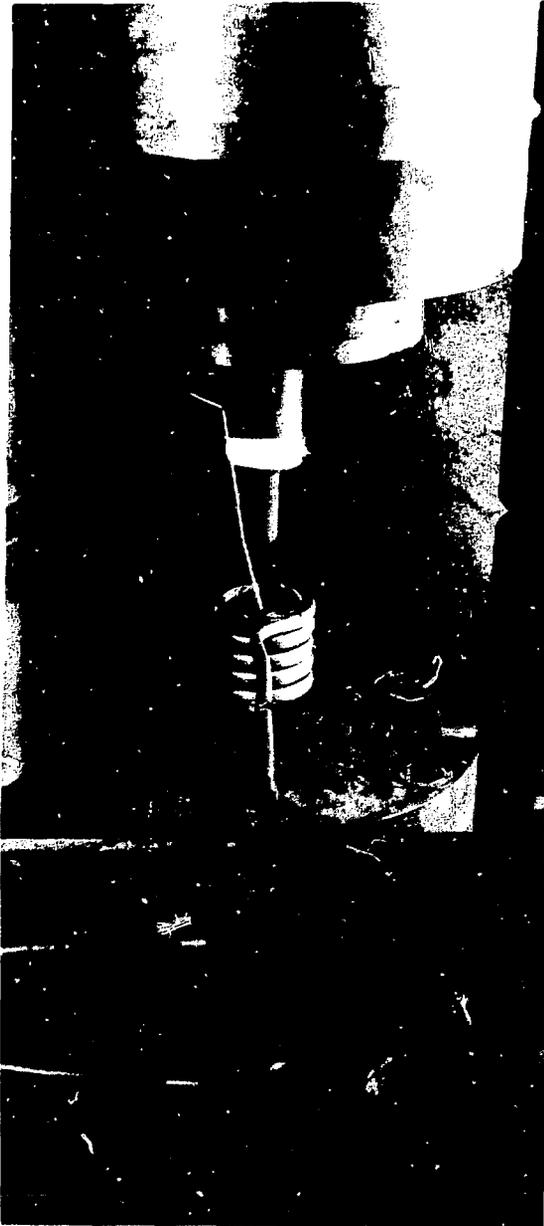
*Extrusion constant as a function of extrusion temperature for HER (Dynapak) and conventional high speed extrusion of arc-cast tungsten.*

travel up to billet contact. The energy at this point amounts to that required to produce breakthrough and is equal to the peak pressure encountered in extrusion. Data developed in this manner do not differ greatly from the extrusion constants developed for conventional high-speed extrusion of tungsten.<sup>5</sup>

The effect of high strain rates on the increase of the dynamic yield strength suggest that high-energy-rate extruded material should have a higher extrusion constant. The fact that this is not observed for the data herein developed may be attributable to the low heat loss associated with Dynapak extrusion.

Figure 9 is a photograph of a typical billet heating and tooling arrangement for refractory metal extrusion. The material is heated by using a 50kW, 10kHz motor-generator set and suspending the material in a water-cooled copper induction coil directly in front of the billet chamber. Insulation between the billet and coil is achieved by supporting the billet on a quarter section ceramic boat.

Thermocouples (Pt + 6 wt% Rh vs Pt + 30 wt % Rh) are imbedded in the billet to monitor the temperature. The machine is fired at the time the billet achieves the desired extrusion temperature and the total



*Fig. 9.*

*Tooling set up used for extrusion of tungsten tubing by the Hooker process.*

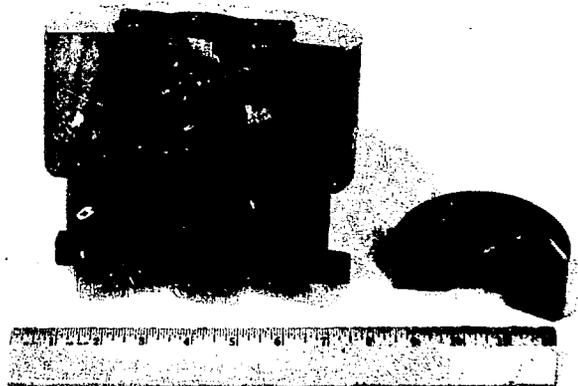
time in contact with the cold tools is 20 milliseconds or less. As a result of the minimal transfer time, the heat loss to cold tools is negligible and the heat of deformation probably elevates the extrusion temperature significantly over the last reading observed before firing the machine.

This increased temperature, coupled with a loss in temperature by conventional processes, suggests a reason for the excellent agreement between the extrusion constants for the two processes when a high constant would more reasonably be expected by Dynapak extrusion. There is some evidence to support the theory that yield strength may be more temperature sensitive than strain-rate sensitive.

**D. Tooling for High Energy Rate Extrusion.** The tooling used for refractory and cermet extrusion is comparatively standard. The punches are normally fabricated from type S-2 steels heat treated to 56 to 58 Rockwell C and are always triple drawn to minimize the amount of retained austenite in the structure. The triple-drawing operation is extremely critical and tool life can be increased significantly by using this step.

The billet chambers used are fabricated from H-13 tool steel, heat treated to a hardness of 48 to 50 Rockwell C. This hardness level yields a material of acceptable toughness and strength for all refractory metal extrusion performed to date. At hardness levels greater than this there is a tendency to split the massive billet chambers because of the excessive hoop stresses developed on impact. Figure 10 is a photograph of a burst die and billet chamber which were used for tungsten sheet bar extrusion. In this case the billet chamber was hardened to 55 to 56 Rockwell C. The punch was softer than acceptable and barreled upon impact.

The typical die design used for refractory metal and cermet extrusion is shown in Fig. 11. This die is composed of a hardened 4340 case into which a WC + 6 wt.% Co insert is shrunk in place; the shrink fit employed is



*Fig. 10.*

*Catastrophic tooling failure associated with improper heat treatment and design of tools.*

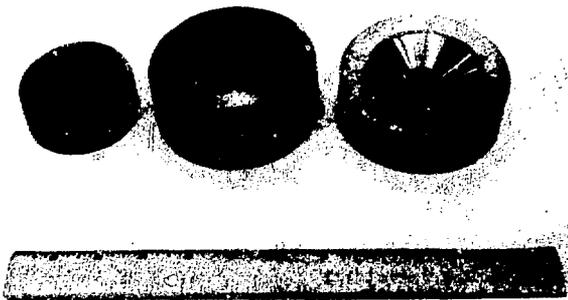


Fig. 11.  
Composite dies of WC + 6 wt% Co which are used for all high temperature extrusion works.

0.003 to 0.005 in. The dies are expensive but the quality of extrusions produced and the die life easily offset the cost incurred. Dies of this type have been used for as many as thirty extrusions of tungsten at temperatures of 1800°C or greater. The only service the die required consisted of polishing to remove the *picked up* material. Inserts of ZrO<sub>2</sub> have also been used, but, in general, the WC type appears to be more satisfactory for refractory metal extrusion at high temperatures.

### III. Magnetic Forming

As previously mentioned, the extrusion process does not always lend itself to the development of an ideal structure because of the directionality developed. For this reason, investigation is being conducted in the area of developing more desirable processes for the fabrication of loaded fuel elements.

**A. Coaxial Pinch Magnetic Forming.** A technique has been devised for the consolidation of powdered metals and forming of conventional tubular products using fast-rising magnetic fields produced in a coaxial conductor configuration carrying high, pulsed currents. Refractory metal and cermet tubes have been fabricated from powders by using the crushing action of this magnetic field.

Figure 12 is an artist's conception of a coaxial coil device in place on a header assembly. The current passes up the outer conductor and down the inner conductor generating magnetic fields around the inner and outer conductors in accordance with the "Right Hand Rule". The opposing magnetic fields are confined within the annular gap between the conductors such that the magnetic field acts as if it is a compressed gas, tending to crush the inner conductor, or bulge the outer conductor depending upon the respective mechanical strengths of the conductors. This effect can be used for high-energy-rate metal forming by collapsing the inner conductor around a

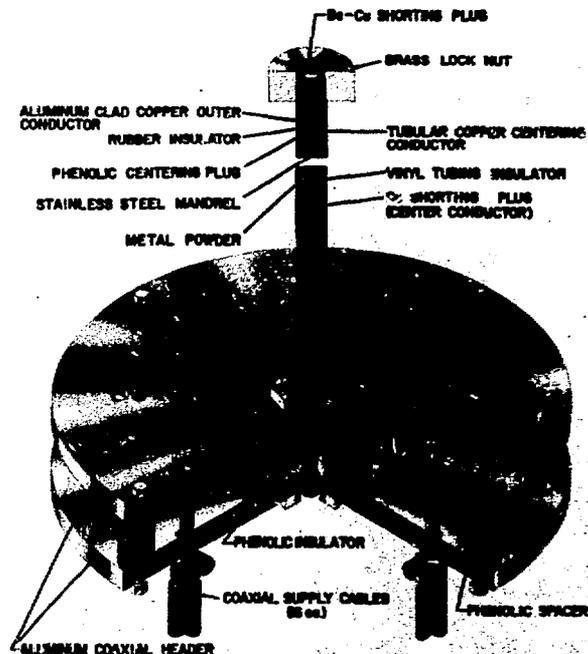


Fig. 12.  
Artist's schematic of the coaxial conductor in a configuration used for magnetically compacting metal powders into tubular shapes.

mandrel or other object that may require cladding.

Figure 13 is a photograph of clad shapes in which the inner copper conductor was clad around a mandrel. This technique of cladding has been used successfully in operations where a good electrical conductor has been reinforced with a mechanically strong core material. Sound joints which are vacuum leak tight at pressures less than 10<sup>-7</sup> Torr have consistently been produced by this technique.

**B. Limitations of the Process for Metal Forming.** A severe limitation to this process exists in that only materials that are good electrical conductors are readily formable. This then limits the effectiveness of the cladding process to copper, aluminum, or silver if relatively heavy clad thicknesses are to be formed. The difficulty in forming other than good electrical conductors is explained by

## MAGNETIC FORMED TUBES

Fig. 13.

*Some typical tubular shapes formed from copper using coaxial pinch.*

the fact that current is carried on the skin of the the conductor in question. The skin depth for current conduction is given by:<sup>6</sup>

$$\epsilon = K \frac{\rho}{f} \quad (1)$$

Where  $\epsilon$  = skin depth  
 $\rho$  = resistivity  
 and  $f$  = frequency.

Figure 14 is a log-log plot of the skin-depth variation for three selected materials vs frequency of the capacitor bank-coil system. This plot shows that at frequencies of 15 kHz the skin depth for conductivity in austenitic stainless steel is approximately 0.187 in. thick whereas in annealed copper it is approximately 0.011 in. Since one skin depth carries approximately 60% of the current it is clear that for maximum magnetic fields the conductor would be more than one skin-depth thick.

The pressure generated in a coaxial system can be calculated if the capacitance and period of the system are known. The equation used for this calculation is:<sup>7</sup>

$$P_{\text{load}} = \left( \frac{L_{\text{load}}}{2} \times I^2 \times 10^7 \right) \left( \frac{8}{\text{Vol}_{\text{load}}} \right) \left( \frac{1}{5000} \right)^2 \text{ ATM.} \quad (2)$$

where  $P$  = pressure (atmospheres)  
 $L$  = inductance  
 $I$  = current  
 and  $\text{Vol}$  = volume.

In a system where the current produced is on the order of 60 kA/kV the pressure developed is approximately 30,000 psi. This pressure is insufficient to form materials, such as austenitic stainless steel, which have yield strengths greater than this value.

The skin-depth limitation can be overcome if long lengths of thin-walled, poor conductors are to be formed by using a good conductor as a "pusher". Figure 15 is a composite photograph showing parts formed from 5 mil wall Inconel tubing over a finned aluminum mandrel. In

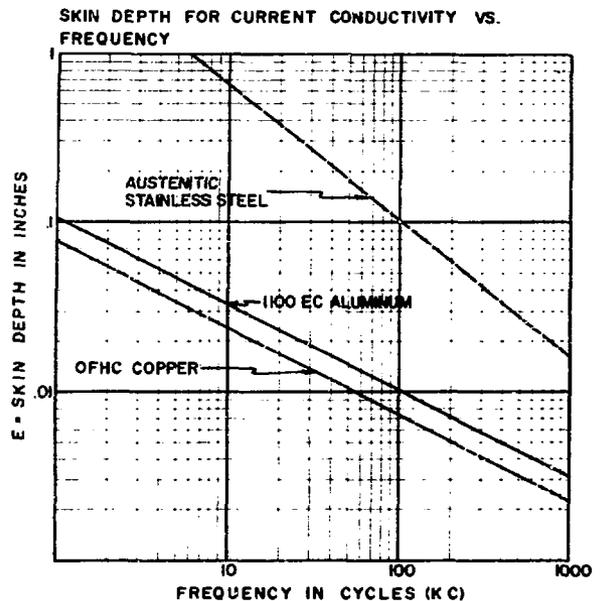


Fig. 14.

*Skin depth for current conductivity as a function of frequency for various conductors of interest in coaxial magnetic pinch forming.*

this instance, annealed aluminum has been used as the inner conductor-pusher. The aluminum mandrel and pusher are subsequently dissolved from the formed part in an alkaline solution.

**C. Consolidation of Refractory Metal Powders and Cermets.** A second application for the coaxial magnetic forming technique lies in the area of powdered-metal



TECHNIQUE FOR FORMING LOW CONDUCTIVITY THIN WALLED TUBING OVER A MANDREL BY THE PUSHER TECHNIQUE



INCONEL RIBBED TUBING MAGNETICALLY FORMED USING THE HIGH CONDUCTIVITY PUSHER TECHNIQUE

*Fig. 15.*

*Technique used for forming poor conductors using the "pusher" technique and a typical shape formed from thin walled Inconel 718.*

consolidation. Since good conductors can be used for pusher materials, high, instantaneous compaction pressures can be achieved using the coaxial conductor technique. Figure 12 is an artist's conception of the coaxial device in a configuration used for compacting powder into a tubular shape. A metallic mandrel is used for producing the bore of the tube. The metal powders and mandrel are confined in a cellulose acetate sack to protect the compacted powder from acid attack during the dissolution of the copper inner-conductor pusher material. The acetate sack is sealed on the ends and placed inside the copper inner-conductor pusher before forming. Tapered end plugs are driven into the inner tube to yield good electrical contact with the copper tube. The top plug also acts as a "dead short" across the annular gap between the inner and outer conductors thereby completing the current path. The annular gap between the

outer and inner conductor should be minimized to develop maximum pressure according to Eq (2). However, the insulation between the inner and outer conductors must be sufficient to eliminate arcing between conductors since high voltage breakdown between conductors drastically reduces the efficiency of the process.

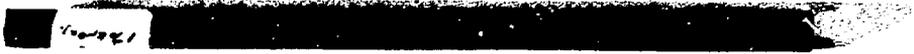
Tubular shapes have been magnetically compacted from tungsten, molybdenum,  $W + UO_2$  and  $Mo + UO_2$  powders. All these systems have been examined with and without binders, lubricants, and with different mandrel materials. In general, it can be said that lubricants such as cetyl alcohol, stearic acid, and paraffin are desirable for the compaction of fine particle size tungsten tubing. These lubricants do not, however, impart any strength to the formed tubes in the green state. As a result, mandrel extraction becomes extremely difficult and the handling problems minimize the possibility of producing long lengths of tubing. The use of binders has been investigated, but suitable binders have yet to be developed. Epoxies have been used and extremely rugged, easy to handle green tubes have been manufactured. Figure 16 shows three lengths of tungsten tubing formed with epoxy as a binder. These tubes were extremely tough and could be machined in the green state. However, in a typical tungsten sample having a green density of 75% of theoretical, the percent of closed pores will be sufficient to eliminate the possibility of complete epoxy burn-out on sintering. This condition results in a heterogeneous mixture of tungsten metal and WC in the final product. Continued studies into the development of suitable binders and lubricants are now being conducted.

As was previously mentioned, the effect of mandrel material on the sintered and green density of formed tubes has also been studied. In the work performed thus far, a pre-mixed alloy of 70 wt% W and 30 wt% Mo has been used for evaluation. The mandrel materials selected for investigation are austenitic stainless steel and high density, centerless ground tungsten. The tests were performed using bank voltages varying from 13.5 kV to 19.5 kV; this corresponds to a variation in pressure from 23,600 psi at 13 kV to 56,000 psi at 20 kV. Figure 17 is a graph showing the variation in green and sintered density of compacted tubes using the mandrels noted. A maximum in the sintered density does not occur at the maximum green density value but actually decreases. This is believed to be attributable to the Kirkendall effect for alloys. It is apparent from these studies that no real advantage is to be gained by using a higher-modulus mandrel for compaction.

The materials most readily consolidated by this technique are those which possess some ductility. For example, Molybdenum is easier to compact than pure tungsten. Similarly,  $W + Mo$  powder mixtures are more readily compactable than pre-alloyed powders which would have the same composition. Figure 18 is a photograph of magnetically compacted and sintered molybdenum tubes using a starting material that had an average Fisher particle size of  $4.5\mu$ . No binders were used. The mandrel material was stainless steel. The compaction



TUNGSTEN - .5w% EPOXY BINDER



TUNGSTEN - 1 w% EPOXY BINDER

FORMED TUNGSTEN

# MAGNETIC FORMED TUBES

Fig. 16.

*Tungsten tubing produced by magnetically compacting powders to which various weight percentages of epoxy have been added as a binder.*

pressure used on the samples was approximately 40,000 psi and the duration of the pulse was about  $45\mu$  sec. The sintered density of these tubes was 95.21% of theoretical as determined by mercury porosimeter techniques.

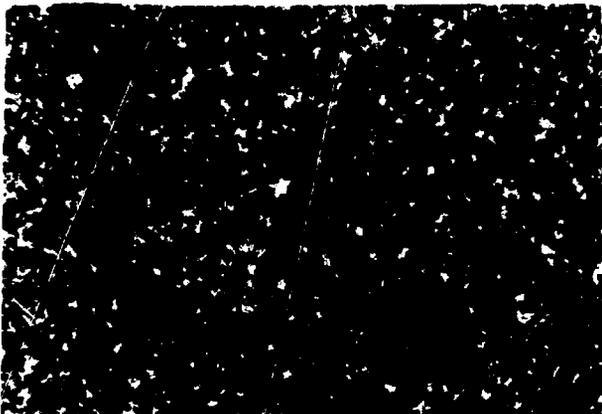
Figure 19 is a photograph of the "as-pressed" structure of this material and a typical macrosection of the pressed and sintered tubing.

The composite of greatest interest for fabrication by this process is a cermet composed of W + Mo loaded with  $UO_2$ . This material is of interest in reactor design because it yields a homogeneous fuel element with little or no directionality. The work performed on a system of Mo + 40 Vol%  $UO_2$  has shown the most promise thus far. Figure 21 is representative of the microstructure of longitudinal and transverse slices from a compacted mixture of powders sintered to 92% of theoretical density. The molybdenum has an average particle size of  $4.5\mu$ . The  $UO_2$  particles are defined as a coarse agglomerate powder. This powder was chosen so the shrinkage between the  $UO_2$  and molybdenum could be closely matched.

## IV. Summary

It is possible to form high L/D ratio tubes from refractory metals and cermets using a variety of conventional techniques. The high-energy-rate extrusion process can be used, but there appears to be a practical limit to the lengths produced by this process.

The coaxial pinch forming technique offers a highly satisfactory method for forming long lengths of small to medium diameter tubing and appears to be feasible for consolidating refractory metals and alloys into tubular geometries. Additional research into the effect of pressure pulse form and time are now being conducted to determine the optimum parameters for consistent consolidation of metal powders. These data must be developed if the process is to achieve industrial importance.



TYPICAL MICROSTRUCTURE OF "AS PRESSED" 4.5 MICRON MOLYBDENUM POWDER. 100X



PHOTOMICROGRAPH OF A TYPICAL CROSS SECTION OF SINTERED MOLYBDENUM TUBING. 5X

Fig. 17.  
Green and sintered density of a W + 30 wt% Mo alloy.

DENSITY MEASUREMENTS AT VARIOUS SINTER TEMPERATURES

- STEEL MANDREL GREEN DENSITY
- STEEL MANDREL 1750° C - H<sub>2</sub> SINTER
- STEEL MANDREL 2200° C - VACUUM SINTER
- - -■- - - W MANDREL GREEN DENSITY
- - -■- - - W MANDREL 1750° C - H<sub>2</sub> SINTER
- - -●- - - W MANDREL 2200° C - VACUUM SINTER

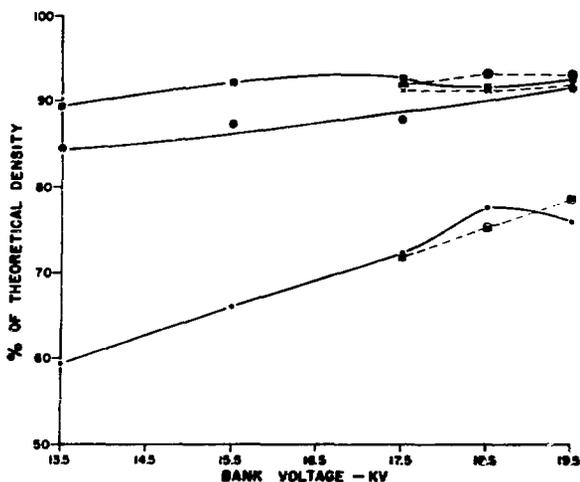
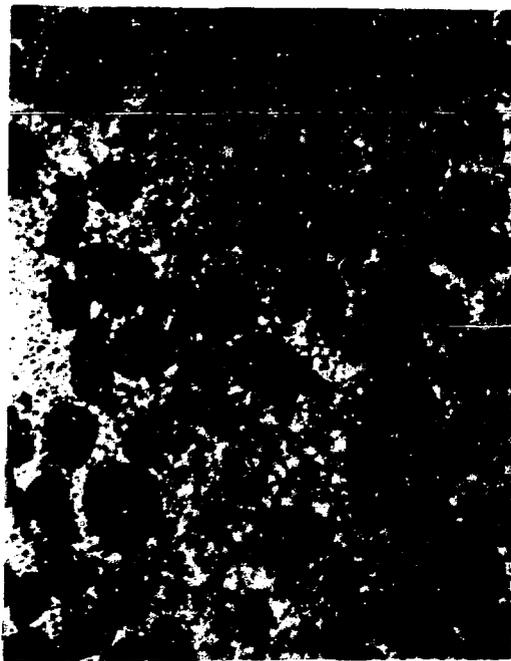


Fig. 19.  
Typical microstructure of green compacted Mo and the resultant macrostructure of a sintered tube.

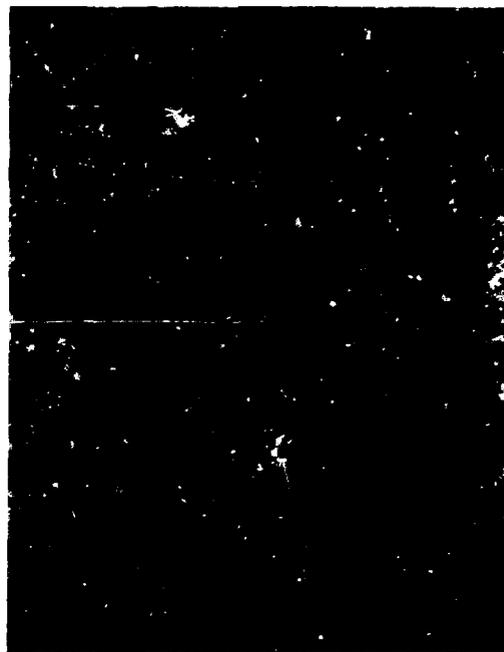


Fig. 18.  
Magnetically compacted and sintered tungsten tubing produced from 4.5μ APS powder.



TRANS.

100X



LONG.

100X

**PHOTOMICROGRAPH OF Mo-40% UO<sub>2</sub> TUBING PRODUCED BY MAGNETIC COMPACTION AND SINTERED AT 1700°C IN DRY H<sub>2</sub> FOR 15 HOURS.**

*Fig. 20.*

*Microstructure of Mo + Mo vol% UO<sub>2</sub> tubing produced by magnetic compaction and sintering at 1700°C in Dry Hydrogen for 15 hours.*

**Acknowledgments**

The authors gratefully acknowledge the following individuals for their assistance in carrying out these programs.

James M. Taub, Group Leader, CMB-6 and Gale S. Hanks, Fabrication Section Leader for technical direction and supervision of the program; Charles L. Terrell, Victor Vigil and Thomas I. Jones for technical assistance and George F. Erickson and Edwin L. Kemp without whose help this would not be possible.

**References**

1. Feasibility Study of the Coextrusion of Refractory Metal Clad Reactor Fuel Elements: LAMS 3065, May 4, 1964, J. M. Dickinson and T. J. Ready.
2. Comparison of High Energy Rate (Dunapak) and Conventional Estrusion of Refractory Metals. ASD-TDR-62-506, September, 1962, D. G. Rabenold et al.
3. High Energy Rate Extrusion-Interim Technical Progress Report No. 1. ASC-TR-7-882 (I) by G. A. Reimann.
4. Tooling Manual-General Dynamics Corporation.
5. Radio Engineers Handbook-Frederick Terman, 1943, pp 28-35.
6. Private Communication with E. L. Kemp, Los Alamos Scientific Laboratory.