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## **TECHNICAL REPORT ARLCB-TR-85017**

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# NEW POWDER TECHNOLOGIES FOR MOLYBDENUM ALLOY GUN BARREL LINERS

J. M. BARRANCO

SAUL ISSEROW

**JUNE 1985** 



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US ARMY ARMAMENT RESEARCH AND DEVELOPMENT CENTER LARGE CALIBER WEAPON SYSTEMS LABORATORY BENET WEAPONS LABORATORY WATERVLIET N.Y. 12189



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7. AUTHORS (CONT'D)

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20. ABSTRACT (CONT'D)

utilization of the benefits of molybdenum for this and related applications. Work will be reported on alloy powders prepared by various methods with emphasis on rapid solidification, either by rotating electrode (REP and PREP) or by plasma melting (PMRS, plasma melted rapidly solidified). To date, consolidation has been primarily by hot isostatic pressing (HIP).

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Compression stress vs. strain for Mo-0.1% Co, HIP densified:
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#### BACKGROUND

Erosion of gun barrels is still a problem for current weapons and for projected systems with sustained rapid fire requirements. Microscopic changes in the barrel surface frequently provic evidence of the following: melting of the surface layer, white layer embrittlement in gun steel, and often, heat checking and embrittlement. Such changes ultimately lead to macroscopic changes that degrade gun performance and accuracy. These changes are collectively referred to as erosion.

The erosion of a gun barrel is not uniform along its length, but is localized near the origin-of-rifling (0.R.), being less severe in the muzzle region. The use of a short liner can provide a suitable bore surface at or near the 0.R. to overcome the most severe erosion. The ideal liner should be fabricated from a material with good thermal, chemical, and mechanical properties. A thermal resistant material would need a high melting point with high specific heat and good thermal conductivity. Resistance to thermal and chemical shock is necessary under conditions of rapid heating and cooling in the presence of chemically active propellant gases. Suitable mechanical properties are needed to prevent deformation of lands or grooves, with little wear and with resistance to cracking under firing conditions.

Molybdenum appears to be the ideal erosion-resistant liner material. Some objections might be raised because of differences in coefficient of expansion and modulus of elasticity. In addition, compared to steel, arccast molybdenum is soft and lacks ductility. These latter shortcomings can be reduced greatly by alloying and proper mechanical working, while innovative liner design may alleviate the others. The alloy TZM contains nominally 0.5

percent titanium, 0.08 percent zirconium, and 0.02 percent carbon (by wt.) with the balance molybdenum. TZM attains an unworked or annealed hardness of 20 Rc, while working to a 50 percent reduction-in-area increases hardness to about 30 Rc with favorable ductility. Molybdenum with 0.1 p:rcent cobalt (by wt.) behaves similarly.

Attempts were made during World War II to fabricate molybdenum liners from tubes bored from swaged rods (refs 1,2). Such liners failed after only a few rounds; transverse and longitudinal cracks were observed. A helical twisted 0.50 cal liner was also introduced (ref 3). This liner consisted of a two-stave component, starting with forged flat stock joined by a hot twisting operation as shown in Figure 1. This design was an attempt to improve the normally weak transverse strength associated with large grained uniaxially worked material. Unfortunately, the seams opened up during firing, allowing hot gases to penetrate, causing spalling to occur along the edges. The World War II work is summarized in Reference 4. The follow-on post-war work is covered in References 5 and 6.

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- <sup>1</sup>"Investigation of Gun Erosion at the Geophysical Laboratory," Vol. I, July 1941 to July 1943, OSRD 3448, Report No. A-203, Geophysical Lab., CIW, March 1944.
- <sup>2</sup>"Investigation of Gun Erosion at the Geophysical Laboratory," Vol. II, July 1943 to December 1943, OSRD 3449, Report No. A-204, Geophysical Lab., CIW, March 1944.
- <sup>3</sup>J. W. Marden, "Fabrication of Molybdenum as a Gun Liner Material," OSRD 6494, Final Report No. A-423, October 31, 1945, Westinghouse Electric and Manufacturing Co., Inc.
- <sup>4</sup>F. Palmer, "Molybdenum," Hypervelocity Guns and the Control of Gun Erosion, Summary Technical Report of Division 1, NRDC, AD 221585, Washington, D.C., 1946, p. 370.
- <sup>5</sup>G. Cohn, "Barrels for Automatic Weapons," Summary of Research and Development 1946 to June 1955, F-A 2251, AD 314698, Franklin Institute, December 1959. <sup>6</sup>G. Cohn, "Barrels for Automatic Weapons," Summary of Research and Development
- 1955 to 1960, F-A 2461, AD 325116, Franklin Institute, May 1961.

A recent Benet Weapons Laboratory program unsuccessfully attempted to hot roll an arc-cast blank 22 inches long, with a 6-inch 0.D. and 4-inch I.D., to fabricate a 105 mm liner. Circumferential and longitudinal cracking occurred as shown in Figure 2. Maintaining the temperature and the reductions per pass of the roll extrusion process were critical. Extremely coarse grains were observed as shown in Figure 3. Cracking along the grain boundaries was also common. The excessive grain size and low transverse strength resulting from the ingot metallurgy approach have led to emphasis on powder metallurgy to achieve fine-grained isotropic molybdenum components.

#### APPLICATION OF POWDER METALLURGY

Powder metallurgy merits investigation for gun barrel requirements now that new technologies are available for preparation and consolidation of powders. These technologies offer the possibilities of improving standard compositions or making new compositions practical. Pre-alloyed powders can now be prepared by methods that lead to greater compositional uniformity and finer grain size through rapid solidification of molten globules. In addition, a rather new processing method, hot isostatic pressing (HIP), is now well established; it offers a path to substantial, even complete consolidation of the powders with retention of the benefits of fine grain size and isotropy.

This report describes the results of a study on rapidly solidified TZM powders prepared by two processes, rotating electrode and plasma melting. Also included is molybdenum with 0.1 percent cobalt (Mo-0.1% Co) obtained by co-reduction. Consolidation was sought by HIP under various conditions. Evaluation was based on microstructural examination and determination of mechanical properties.

#### POWDER PREPARATION

Two versions of the rotating electrode process, the basic REP and the modified version (plasma rotating electrode process, PREP) were used by Nuclear Metals, Inc., Concord, MA (refs 7,8). For the REP, a water-cooled tungsten-tipped cathode is used to strike an electric arc to the rapidly rotating TZM alloy anode from which molten droplets are centrifugally flung into an inert gas chamber as shown in Figure 4. The solidification rate is moderate, being 10<sup>3°</sup>C/sec or slightly higher. This process requires anode feed stock of high mechanical integrity and a smooth finish.

TZM powder was also prepared by PREP, shown schematically in Figure 5. In this version of the process, a helium plasma arc is the heat source instead of the tungsten-tipped cathode, which has been found to introduce tungsten contamination. Both versions of the process have essentially the same constraint on the lower limit of particle size. The process is inherently limited to relatively coarse particles (ref 9).

Much finer powder was prepared by GTE Sylvania, Towanda, PA using its proprietary plasma melting rapid solidification process (PMRS). This relatively new process (ref 10) uses a powder blend with a liquid binder. The PMRS process for TZM starts with the alloying elements titanium and zirconium

<sup>&</sup>lt;sup>7</sup>P. R. Roberts, "Commercial Atomization by the Rotating Electrode Process," Seminar Preprint, <u>Atomization Processes:</u> Current and Future, Toronto, P/M .84, June 19, 1984.

<sup>&</sup>lt;sup>8</sup>P. R. Roberts, "Rotating Electrode Process," Metals Handbook, <u>Powder</u> <u>Metallurgy</u>, Vol. 7, Ninth Edition, 1984, p. 39.

 <sup>&</sup>lt;sup>9</sup>B. Champagne and R. Angus, "Fabrication of Powders by the Rotating Electrode Process," <u>International Journal of Powder Metallurgy</u>, Vol. 16, No. 4, 1980.
 <sup>10</sup>R. F. Cheney, Plasma Melted and Rapidly Solidified Powders," GTE Products Corporation, Presented at the Annual Meeting of the Materials Research Society, Bocton, MA, November 14-17, 1983.

added as carbides and/or hydrides. The powder, blended with the binder, is spray dried into droplets or agglomerates which are sintered, sized, and then passed through an inert plasma flame followed by quenching into inert gas, as shown in Figure 6. The result is a homogeneous spherical powder with each powder particle containing the alloying composition so desired. The solidification rate is faster than for the REP or PREP process with cooling rates at about  $10^{5\circ}$ C/sec. The mean particle size for this process has been typically in the 20 µm range, while for the rotating electrode process 220 µm is not an uncommon mean particle size. Process parameters for PMRS can be adjusted to achieve even finer powder.

The program also included the Mo-0.1% Co (wt.%) alloy produced by a co-reduction chemical process. This is not a rapid solidification process, but it presumably insures a homogeneous distribution of cobalt in the molybdenum. The parti is was less than 2 µm.

#### CONSOLIDATION

HIP consolidation of the canned powder was performed by Industrial Materials Technology, Inc., Andover, MA. The cans, typically made of titanium, were three-quarters-inch in diameter and up to eight inches long. After being filled with the molybdenum alloy powder, the cans were evacuated and sealed. HIP was in argon for three hours at 30 Ksi at temperatures from 1300 to 1600°C, as shown in Table II. After the HIP cycle, the cans were machined off to leave the bare compact for sectioning into specimens for metallography and mechanical testing.

#### MECHANICAL TESTS

Bend and compression values were determined from all HIP specimens. The bend bars (ASTM E-23) were machined with the same dimensions as a Charpy bar with a notch (N) or without a notch (S for smooth). The slow bend tests performed on these bars were done with an Instron Universal Testing machine at a cross-head speed of 0.02 in./min with a low voltage differential transformer (LVDT) gage to measure bend deflection. Tests conducted on these bend bars gave values for bend yield and bend rupture strength as well as flexural modulus (not compensated for machine compliance) and fracture energy. The compression specimens measured three-eighths inch in diameter and one inch long. This specimen allowed the yield strength and modulus (not compensated for machine compliance) to be measured in compression and also served for density determination from weight and volume.

Metallographic evaluation was done using the scanning electron microscope (SEM) and employing standard polishing and etching procedures.

#### RESULTS

#### Qualitative Observations

The powders used in this study are listed in Table I. Various photomicrographs are shown in Figures 7 to 13. As-produced powders are in Figures 7 and 10, respectively, for REP and PMRS. The other photomicrographs show HIP samples.

The REP and PREP powders had approximately the same particle size with a median of the order of 100-200 µm. Figures 7a and b, respectively, show the exterior surface and microstructure of REP powder. These coarse spherical

powders did not densify fully even at the higher HIP temperature 1600°C. The first attempt at 1500°C left voids as shown in Figure 8. For the next attempt, the temperature was raised to 1600°C. In addition, the powder was sieved to obtain the 200 mesh (74 microns) fraction for HIP. These changes reduced the porosity, but did not eliminate it (Figure 9). Voids and prior particle boundaries are still evident. Some improvement is indicated by the occurrence of transparticle fracture in the bend specimen (Figure 9a) compared to mostly interparticle fracture previously (Figure 8a).

The PMRS powder was much finer having a median diameter of 24.7 µm with very fine grains within each powder particle (Figure 10). HIP at 1600°C gave full densification (Figure 11) with only slight remnants of prior particle boundaries. These observations are consistent with the substantial increase in fracture energy compared with the PREP powder HIP at 1600°C (145.3 vs. 47.7 ft-1b).

For the Mo-0.1% Co powder, the co-reduction method of preparation was tried after the lack of success with elemental blends. Such blends had shown segregation of these elements upon sintering. The co-reduced powder had a mean particle size below 1.7 µm. This powder, with a low apparent density of 2.36 g/cc, sometimes agglomerated during the canning process. This agglomeration was evident after HIP as shown in the microstructures in Figure 12. Packing density variations caused by agglomeration resulted in a grain growth phenomenon in a specific area with the larger grains surrounded by smaller grains. The result was a crack running through this area to the inside of the can. Increasing HIP temperature from 1300 to 1500°C resulted in excessive grain growth as shown in Figure 13. The fractograph of the broken

bend bars shows consistent intergranular fracture mode for all three HIP temperatures used.

#### Mechanical Properties of Compacts

The results of the mechanical tests summarized in Table II and plotted in Figures 14 through 17 reflect response to HIP parameters. Additional hardness values of TZM spherical powders are shown in Table III.

The HIP consolidated PREP powders exhibited low bend rupture strengths and fracture energy even for the smooth bar. The PMRS powder reached full theoretical density and achieved higher mechanical properties as seen in Figure 14. In constrast, the compression tests (Figure 15) showed higher strengths for compacts prepared from the PREP powder.

The co-reduced Mo-0.1% Co compacts showed lower strengths in both bend (Figure 16) and compression tests (Figure 17) as the HIP temperature was successively increased from 1300 to 1500°C. The inferiority of this material relative to both types of TZM powder is seen in the mechanical properties, most notably the fracture energy shown in Table II. The co-reduced Mo-0.1% Co powder was so fine as to have so low an apparent density (2.36 g/cc) and so strong an agglomeration tendency as to make it difficult to achieve a uniform fully densified compact.

In light of these results, effort is now concentrated c the PMRS process, initially TZM and subsequently richer alloys. In fact, the first go-around included a partially successful attempt to prepare a molybdenum alloy with high cobalt content of the order of four percent (by wt.). The continuing activity is first devoted to establishing conditions for producing TZM powder with uniform distributions of alloying elements and secondly, to

controlling oxygen and carbon contents.

#### DISCUSSION

TZM powders produced by either version of the rotating electrode process did not densify adequately even under the most severe HIP conditions. The bend tests showed low fracture energies associated with voids and fracture at prior particle boundaries. The inadequate consolidation is attributed to the inherent coarseness of powders prepared by this process. Consolidation was not appreciably helped by prior screening of the powder to recover a finer fraction (-200 mesh) for HIP consolidation at 1600°C.

The plasma melting rapid solidification process is capable of producing much finer powder. The benefit of the finer powder size was demonstrated by the HIP densification of the powder and by the consequently enhanced mechanical properties. Compacts were also shown to retain a fine grain size even after exposure to temperatures as high as 1600°C during HIP. The retained fine grain size should be beneficial for any subsequent working to a required shape for service applications such as needed in a gun liner.

It should be noted that the first lot of TZM powder supplied by the PMRS process is not necessarily representative of the capabilities of the process. Control of the process parameters is necessary for achieving optimum composition and particle size. The lack of process refinement was present in this lot which was found to be compositionally unsatisfactory in at least two respects: segregation of the titanium and excessive oxygen content was present. Work is now proceeding to overcome these deficiencies. Meanwhile this route appears most promising for relatively inexpensive preparation of a

standard molybdenum alloy such as TZM and also new compositions that will exploit the benefits of rapid solidification.

SUMMARY AND CONCLUSIONS

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1. Rapidly solidified powders of the molybdenum alloy TZM were prepared by two methods, rotating electrode and plasma melted rapidly solidified method. These powders were consolidated by hot isostatic pressing and mechanical properties were determined for the compacts.

2. Rotating electrode powder is inherently coarse and therefore difficult to consolidate in the absence of significant deformation. Full densification was not achieved with the TZM powders even under the most drestic HIP conditions using temperatures to 1600°C and pressures as high as 30 Ksi (207 MPa) up to three hours duration.

3. Plasma melting of sized agglomerates from the PMRS method results in much finer powder. Encouraging results were obtained with this powder consolidated by HIP. Process improvements are needed to control uniformity of composition and the presence of interstitials, but this method is considered most promising for TZM and other molybdenum alloy compositions.

4. Powders of Mo-0.1% Co (by wt.) were prepared by co-reduction. These powders were very fine and tended to agglomerate. They did not respond well to HIP.

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Material	Manufacturers	Type Powder	Particle Size Average (µm)	Apparent Density (g/cc)
TZM(REP)	Nuclear Metals	spherical	220.0	6.22
TZM (PREP)	Nuclear Metals	spherical	74.0	6.33
TZM (PMRS)	Sylvania-GTE	spherical	24.7	5.32
Mo-0.1% Co	AMAX	irregular/ co-reduced	1.7	2.36

## TABLE I. POWDERS USED FOR HIP EVALUATION

1.15

TABLE II. R.T. PROPERTIES OF HIP SPECIMENS\*

						Three-Poi	nt Slow Be	nd Test		Comp	ression
Material	Code	HIP Tenp °C	Hardness Rockwell Rc	Density kg/m <sup>3</sup> 10 <sup>3</sup>	Yield Ksi (MPa)	Rupture Str Ksi (MPa)	Flex Mod psi 10 <sup>6</sup> (MPa) 10 <sup>3</sup>	Max Defl mils (µm)	Fracture Energy inlb (Joule)	YS .2% Ksi (MPa)	Modulus psi 10 <sup>6</sup> (MPa) 10 <sup>3</sup>
TZM (PREP)	11(N)	1500	8.6	9.6	73.6	73.6	22.9 (157.9)	4.3 (109.2)	2.6 (0.3)	61.5 (424.0)	10.5 (72.4)
	12(N)	1600	15.3	10.0	73.1	73.1 [	29.8 (205.5)	3.2   (81.3)	1.9 (0.2)	65.0 (448.2)	17.0   (117.2)
	8(S)	1600	15.8	6.6	53.6 (369.6)	111.5   (768.8)	13.2 (91.0)	22.1   (561.3)	47.7 (5.4)	65.0 (448.2)	15.3 (105.5)
TZM (PMRS)	15(N)	1500	20.0	10.1	85.8 (591.6)	85.8 (591.6)	21.8 (150.3)	5.2 (132.1)	3.6 (0.4)	59.9 (413.0)	9.7 (66.9)
	16(S)	1500	20.0	10.1	69.3 (477.8)	127.0   (875.6)	9.8 (67.6)	35.2 (894.1)	87.4 (9.9)	59.9 (413.0)	9.7 (66.9)
	17(N)	1600	18.0	10.1	79.7	79.7	21.7   (149.6)	4.9   (124.5)	3.2 (0.4)	53.4 (368.2)	13.0 (89.6)
	18(S)	1600	18.2	10.1	61.3 (422.6)	126.0 (868.7)	9.7 (66.6)	57.1 (1450.3)	145.3 (16.4)	53.4 (368.2)	13.0 (173.7)
Mo-0.1% Co	16(S)	1300	15.7	10.1	73.2	73.2   (504.7)	14.9 (102.7)	5.2 (132.1)	4.9 (0.6)	88.4 (609.5)	25.2 (173.7)
	21(S)	1400	13.6	10.1	65.3 (450.2)	65.3 (450.2)	14.1 (97.2)	4.9   (124.5)	4.1 (0.5)	94.0	40.5 (279.2)

TABLE II. R.T. PROPERTIES OF HIP SPECIMENS\* (CONT'D)

Modulus ps1 10<sup>6</sup> (MPa) 10<sup>3</sup> 10.4 (71.7) 20.3 (140.0) 11.6 (80.0) Compression Test 73.0 | (503.3) 58.7 | (404.7) 57.0 | (393.0)| (MPa) YS •2% Ksi .2 (0.02) (10.) Fracture Energy in.-lb (Joule) 4.6 (0.5) ۲. 1.3 | (33.0)| .8 (20.3) 5.5 | (139.7) Test afls (m) Max Defl Three-Point Slow Bend |Flex Mod | | psf 10<sup>6</sup> | |(MPa) 10<sup>3</sup>| 19.3 | (133.1) 12.7 (87.6) 17.5 (120.7) 57.7 | (397.8) 18.3 | (126.2)| 10.01 (68.9) Rupture (MPa) Str Ksi 42.3 | (291.6) | 18.3 | (126.2)| 10.0 | (68.9) Ksi (MPa) Yield | Density | |kg/m<sup>3</sup> 10<sup>3</sup>| 10.1 10.1 10.1 Hardness Rockwell 14.0 13.6 13.6 Rc HIP Temp °C 1400 1500 1500 32(N) 54(S) 43(N) Code Mo-0.1% Co Material

0.394 in Charpy bar (N) notched, (S) smooth 0.357 diameter compression specimen by one-inch long \*30 Ks1 - Three hours

## TABLE III. MICROHARDNESS OF TZM MOLYBDENUM ALLOY POWDERS

# KNOOP $(k_g/mm^2)$

Size Range   (Mesh) 	Test Load (Grams)	PREP Lot 4562-1	REP Lot 4562-2	PMRS SX-207
BWL				
-60 + 80	50	339.0	254.0	-
AMMRC (82)		:		
-35 + 45	100	244.2	272.5	-
<del>-</del> 35 + 45	50	256.8	241.8	-
-170 + 230	50	380.7	353.2	-
AMMRC (83)				, ,
-170 + 230	50	442.0	444.0	-
-170 + 230	25	486.0	463.0	-
-250	25	-	-	623.0



Figure 1. Steps in processing a two-stave liner of molybdenum (ref. 3).



Figure 2. TZM cylinder arc cast (AMAX) and roll formed (Rollmet). Reduced from 6" 0.D./4" I.D. to 5" 0.D./4" I.D. (55 percent reduction-in-area).



(b)

Figure 3. (a) Longitudinal section of the cylinder shown in Figure 2 (50%). (b) Transverse section of the same (50%).















(a)



(b)

Figure 7. TZM spherical powder produced by the rotating electrode process (REP) - (a) powder, SEM (300X), and (b) polished and etched microstructure (200X).



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(a)

**(**b)

Figure 8. TZM spherical unsieved powder (mean particle diameter 220 µm) produced by the rotating electrode process (REP) and HIP densified at 1500°C at 30 Ksi for three hours indicating point contact sintering - (a) tensile bar fracture surface SEM (125X) and (b) polished and etched microstructure (100X).



(a)



(b)

Figure 9. TZM spherical powder produced by the plasma electrode rotating process (PREP) and HIP densified at 1600°C at 30 Ksi for four hours - (a) three-point bend fractograph SEM (300X) and (b) polished and etched microstructure (200X).





Figure 10. TZM spherical powder SX207 (average particle diameter 24.7 µm) produced by the plasma melting and rapid solidification process (PMRS) - (a) powder, SEM (300X) and (b) polished and etched microstructure (1000X).



<u>1</u>



(b)

Figure 11. TZM spherical powder, SX207 (average particle diameter 24.7 µm) produced by the plasma melting and rapid solidification process (PMRS) and HIP densified at 1600°C at 30 Ksi for three hours -(a) three-point bend fractograph, SEM (1500X) and (b) polished and etched microstructure (500X).



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Figure 12. Mo-0.1% Co co-reduced powder of small particle size (1.7 µm) and low apparent density (2.36 g/cc) showing powder packing segregation with cracking occurring during the HIP densification cycle at 1400°C - (a) transverse view across the can diameter showing macro segregation (20X), (b) variation in grain size across interface with crack (100X), and (c) powder surfaces SEM (4000X).



Polished and etched microstructures (500X).



Intergranular fracture surfaces produced by three-point bending of bars (0.394 in. sq.) SEM (1500X).

Figure 13. Mo-0.1% Co co-reduced powder densified by the HIP process at 30 Ksi for three hours at the temperatures indicated (a) 1300°C, (b) 1400°C, and (c) 1500°C.







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Corrression stress vs. strain for Mo-0.1% Co. HIP densified: 30 Ksi, three hours at 1300°C, 1400°C, 1500°C. Figure 17.

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