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BY Ed Guehena / ambDATE 3/9/73

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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-5111

UC-25

ISSUED: January 1973

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FABRICATION AND PROPERTIES OF TUNGSTEN-60 VOL% LEAD HOLLOW CYLINDERS

by

H. Sheinberg and T. I. Jones

ABSTRACT

This report documents the development of an inexpensive high-density tungsten-lead composite. Details of fabrication and testing of long, thin-walled cylinders of W-60 vol% Pb are described. It was demonstrated that a hydrostatically pressed mixture of powders yielded a product that met the high-density, low-cost, ease of fabrication, and mechanical-property goals.

I. INTRODUCTION

In the Los Alamos Scientific Laboratory's continuing technology in heavy metals, interest has been expressed for an inexpensively fabricated shape having a minimum density of 13.5 g/cm^3 . Arbitrary dimensions chosen at the beginning of the program were 7.5 in. o.d. by 7.0 in. i.d. by 6.0 in. long. Additional desired properties were that the material should support its own weight, and remain crack free and dimensionally stable for extended periods of time. A tungsten-lead composite was selected as the candidate material because it appeared to meet density, cost, and ease of fabrication requirements.

The fabrication of these cylinders was accomplished with existing tooling and we feel that scale-up to larger sizes will be no problem.

II. RAW MATERIALS

Four available tungsten powders and two lead powders were employed to make small scale ~ 0.8 -in.-diam by 1.0-in.-long pressings of tungsten-lead with the lead content varying from 40 to 70 vol%.

The powder characterization data and chemical analysis for the tungsten and lead powders are presented in

Tables I and II, respectively. The particle size distribution of the two powders, as determined by sedimentation, are shown in Figs. 1 and 2, respectively. The structure and size of these powders are shown in Figs. 3 and 4.

The cost of typical tungsten powder is \$5.00/lb in 100-lb lots and the cost of lead is \$0.60/lb in 100-lb lots.

III. FABRICATION PROCEDURE

All powders were screened through 100 mesh, United States Standard, to remove large agglomerates before weighing and blending. For the small-scale pressings, powders were weighed to $\pm 0.01 \text{ g}$ and for the large hollow cylinders, powders were weighed to $\pm 0.1 \text{ g}$. Powders were blended 8 h at $\sim 60 \text{ rpm}$ in bottles equipped with aluminum agitator wires. The powder charge was loaded into plastisol (polyvinyl chloride) sacks and hydrostatically pressed at 50,000 psi. Pressings were machined to right cylinders and densities determined by mensuration.

The 7.4-in.-o.d. by 6.9-in.-i.d. by 6.0-in.-long hollow cylinders were pressed at 50,000 psi around an aluminum mandrel tapered ~ 0.002 -in./in. to facilitate pressing removal. The outside diameter of the tungsten-lead pressing was machined before removal from the mandrel. Figure 5 shows a pressed and machined cylinder.

IV. TESTING PROCEDURES

The following test specimens were machined from each of the two large hollow cylinders.

Four flexure specimens, 0.190-in. by 0.190-in. by 1.00-in.-long.

Four compression specimens, 0.200-in.-diam by 0.600-in.-long.

Four creep specimens, 0.200-in.-diam by 0.700-in.-long.

Four tensile specimens, 0.180-in.-thick, dogbone-type as per Dwg. No. 26Y-75832.

The compressive creep specimens were positioned in a fixture (Fig. 6) that accommodated six specimens. The guide-supported weights applied a stress of 24 psi $\pm$ 0.5%. The fixture was placed near one end of a vibration-dampened, heavy-granite surface plate, 20-in.-wide by 78-in.-long. A cathetometer was placed near the other end of the plate for measuring creep in the specimens. The smallest division on the cathetometer scale was 0.05 mm (0.002 in.) and this may be used as the least detectable movement, which would amount to 0.28% on these specimens. A Bristol humidigraph recorded the temperature and humidity throughout the duration of the test. Cathetometer, temperature, and humidity readings were made daily for the five-day work week.

The temperature varied between extremes of 69 and 76°F for the 10-month period, with an average reading of 70.3°F for the first 6 months and 72.9°F for the last 4 months. The relative humidity had extremes of 14 to 62%, with an average reading of 51% for the first 6 months and 27.5% for the last 4 months.

V. DATA AND DISCUSSION

Figure 7 is a plot of calculated theoretical density of tungsten-lead mixtures and cost per pound as a function of volume percent lead. These data indicate that a 70 vol% Pb composite would meet the 13.5 g/cm³ minimum density requirement; however, this composition would have to be pressed to 98.6% of theoretical density to achieve the 13.5 g/cm³ value. This is a very high percent of theoretical density for a pressing operation, considering that the tungsten particles do not deform at the maximum available pressure of 50,000 psi.

A series of small pressings, ~0.8-in.-diam by 1.0-in.-long, were made at 50,000 psi to determine the effect of

volume concentration of lead on percent of theoretical density. Figure 8 shows the results of this series, which utilized four different tungsten powders and two lead powders. Clearly the use of Pb-17 improved the pressed density, but the vendor supplying the powder was unable to reproduce powder with the same characteristics. The density vs lead content curves indicate the advantage of using a very coarse 21.0- μ m tungsten powder, Lot W-48, for low-lead-content high-density composites. Use of a relatively fine 4.45- μ m tungsten, Lot W-3A, is contraindicated for the lead concentration range studied if maximum density was the only criterion. However, because the finer tungsten might improve strength or reduce creep, one large hollow cylinder, designated H-1, was made with this 4.45- μ m tungsten powder; and one, designated H-3, was made with 7.35- μ m powder, Lot W-162. The outside diameter of the pressed cylinders was fairly uniform and only ~0.15 in. was removed from this surface to yield a uniform wall thickness. Density of both cylinders was 14.1 g/cm³ or 97.5% of theoretical. The structure of the two pressings is shown in the photomicrographs in Fig. 9. It is likely that the relatively large voids (black areas) resulted from the hard tungsten particles being pulled out from the lead matrix during polishing.

Radiographically, the density of the pressings was uniform; there were, however, two slightly low-density areas about 0.05 in. by 0.06 in. in the cylinder (H-1) made with 4.45- μ m tungsten and thirty ~0.03- by 0.04-in. low-density areas in the one (H-3) made with the coarser powder. Maximum allowable defect had been arbitrarily set as a 0.06- by 0.06- by 0.06-in. equivalent volume. These low-density areas are clearly larger than the 0.006-in. opening in the screen through which the powders were screened, indicating either agglomeration of lead during blending or nonuniform packing of the powder in the plastisol sack before pressing. The two defects in cylinder H-1 were examined metallographically and are shown in Fig. 10. One appeared to be a large lump of extraneous material surrounded by a void and the other an agglomerate of lead surrounded by a void. Screening after the blending operation would perhaps reduce the size of, or eliminate these defects.

Care was taken to machine test specimens from locations on the cylinders that contained no low-density areas. Mechanical properties of W-60 vol% Pb are shown in Table III. The data indicate that these lead-tungsten composites have considerable strength in the as-pressed condition.

There were no dimensional changes (creep) in the first 6 months. Apparent shortening of 0.05 mm, 0.28%, occurred in 6 months and 21 days for creep specimens H1C and H3A, and in 7 months for specimen H3C. These

specimens then stayed constant throughout the remaining 10 months. The other three specimens, H1A, H1B, and H3B, had no measurable change in the 10-month test. Posttest measurement of the specimens revealed, as expected, only very slight increases in the diameters and very slight shortening of the heights in all specimens, except for specimens H1A and H1B. These two specimens lengthened (swelled ?) 0.001 in. in length, which was an amount considered above measuring accuracy or measuring errors. No explanation is offered for this lengthening.

After considering the 0.28% maximum apparent creep observed on three specimens with the cathetometer measurements, it is concluded that these W-60 vol% Pt specimens had essentially no creep in the 10 months tested under compressive loads of 24 psi and the variation in ambient temperature and humidity stated above.

Scale-up in size of a hollow cylinder beyond ~11-in. o.d. would require that the powders be pressed in a larger hydrostatic press with a maximum operable pressure of only 20,000 psi. Therefore, a series of small tungsten-lead pressings was made at 20,000 psi pressure to determine the effect of lead concentration on pressed density. Results shown in Fig. 11 indicate that a density of 13.5 g/cm³ can be attained at 60 vol% Pb with the coarse powder. Use of the coarser W-162 powder yielded considerably higher density, but these pressings exhibited cracks 10 to 40 days after fabrication. The lower density pressings made with the 4.45- μ m tungsten, Lot W-3A, were discarded shortly after fabrication. Additional pressings were made with the same W-162 and Pb-13 powders; dimensions and weights were monitored with time. Dimensions after cracks appear are not considered to be as accurate. One of the 70 vol% Pb pressings was sintered at 120°C for 2 h in argon after machining; sintering did not

affect dimensions or weight. Results are shown in Table IV. The sintered pressing exhibited no cracks, although its dimensions and weight changed slightly with time. It is possible that the heating merely relieved the residual stresses that promoted cracking in the piece. The more porous pressing apparently oxidized at a higher rate.

A large hollow cylinder pressed at 50,000 psi was monitored for dimensional stability; results are recorded in Table V.

VI. CONCLUSIONS

The feasibility of fabricating an inexpensive, dimensionally stable, dense-hollow cylinder, 7.5-in.-o.d. by 7.0-in.-i.d. by 6.0-in.-long, by hydrostatically pressing a mixture of tungsten and lead powders at 50,000 psi was demonstrated. It was further demonstrated that the W-60 vol% Pb cylinder would support its own weight and be crack free for extended times and would possess adequate mechanical properties. The as-pressed composite exhibited no creep at pressure equivalent to approximately eight times that exerted by its own weight. It is believed that scale-up in size would present no problems.

ACKNOWLEDGMENTS

We wish to acknowledge the assistance of J. E. Magnuson for characterization of the powders, C. Javorsky for metallographic examination, LASL Group M-1 for the radiography, and Group CMB-1 for chemical analysis. We also appreciate the technical supervision of R. E. Riley and J. M. Taub, and valuable discussions with W. S. Clouser.

TABLE I

POWDER CHARACTERISTICS OF TUNGSTEN AND LEAD POWDERS

Characteristic	Powder Lot					
	W-3A	W-48	W-161	W-162	Pb-13	Pb-17
Average particle size, Fisher, μm	4.45	21.0	8.30	7.35	5.8	5.7
Surface area, Blaine, calculated, m^2/g	0.070	0.015	0.037	0.042	0.092	0.093
Bulk density, g/cm^3	4.06	10.07	7.32	7.15	4.8	
Tap density, g/cm^3	5.97	11.94	10.6	10.5	6.19	
Surface area, Numec BET, m^2/g	0.423		0.078	0.287		
Pycnometer density, g/cm^3	19.25	18.53	19.07	19.02	11.54

Fig. 1.
Particle size distribution of tungsten powders.

Fig. 2.
Particle size distribution of lead powders.

TABLE II
COMPOSITION OF TUNGSTEN AND LEAD POWDERS

<u>Element</u>	<u>Powder Lot</u> <u>(Concentration, ppm)</u>					
	<u>W-3A</u>	<u>W-48</u>	<u>W-161</u>	<u>W-162</u>	<u>Pb-13</u>	<u>Pb-17</u>
Li	<3		<3	<3	<2	<10
Be	<1	<10	<1	<1	<2	<10
B	<3	<10	<3	<3	<2	<10
Na	<3		<3	<3	<5	<10
Mg	<1	<30	<1	<1	2	<10
Al	<10	<10	<10	<10	5	<10
Si	10	200	10	10	2	<10
K	50		<10	<10	<50	<10
Ca	<3	<10	<3	<3	<3	<10
Ti	<30	<30	<30	<30	<3	<30
V	<10	<30	<10	<10	<2	<10
Cr	10	10	<10	<10	<2	<10
Mn	<3	10	<3	<3	<2	<10
Fe	30	100	30	30	<2	10-100
Co	<10	100	<10	<10	<2	<10
Ni	50	1000	<10	<10	<2	<10
Cu	20	<10	10	10	15	10-100
Zn	<300	<100	<100	<100	<150	<100
Sr	<10		<10	<10	<1	<10
Zr	<30	<100	<30	<30	<150	<30
Nb	<300	<300	<300	<300	<150	<300
Mo	<100	1000	200	200	<2	<100
Ag	<1	<10	<3	<3	30	<10
Sn	<10	<10	<10	<10	<2	10-100
Ba	<3		<3	<3	15	<10
Pb	<10	<10	<10	<10		<10
Bi	<10		<10	<10	300	10-100
Cd				2	<2	
W					<150	
U					<300

W-161

250X

W-162

250X

W-3A

250X

W-48

100X

Fig. 3.
Photomicrographs of tungsten powders.

Pb-13

Pb-17

Fig. 4.
Photomicrographs of lead powders, 250X.

Fig. 5.
Hollow cylinder of tungsten-60 vol% lead.

Fig. 6.
Creep test fixture.

Fig. 7.
Theoretical density and cost per pound vs volume concentration of lead.

Fig. 8.
Theoretical density of pressed cylinders vs concentration of lead.

Cylinder H-3 made with 7.35- μ m tungsten powder.

Cylinder H-1 made with 4.45- μ m tungsten powder.

Fig. 9.
Photomicrographs of structure of tungsten-60 vol%
lead hollow cylinders, 500X.

Ceramic Particle

Agglomerated Lead Powder

Fig. 10.
Defects in tungsten-60 vol% lead cylinder (H-1)
made with 4.45- μ m tungsten powder.

TABLE III
MECHANICAL PROPERTIES OF TUNGSTEN-60 VOL% LEAD

Sample	Size W Powder (μm)	Tensile Strength (psi)	Elongation 1 in. (%)	Approx^a Tensile Modulus	Modulus of Rupture	Compressive Strength (psi)	Compressive Yield Strength (psi)	Approx^b Compressive Modulus
H-1	4.45	5460	1.0	6.2×10^6	9440	10,200 ^c	8400 ^c	2.6×10^6
H-1	4.45	5480	0.5	8.7×10^6	10,570	11,300	10,100	2.7×10^6
H-1	4.45	4250	0.5	6.8×10^6	9710	11,000	9700	3.0×10^6
H-1	4.45	3060	0.5	7.0×10^6	9740	11,000	9800	2.8×10^6
Average		4560		7.2×10^6	9870	11,000	9800	2.8×10^6
H-3	7.35	3960	0.5	8.2×10^6	9490	10,500	9600	2.9×10^6
H-3	7.35	5400	0.5	6.9×10^6	9340	10,400	9500	2.8×10^6
H-3	7.35	3500	0.5	5.1×10^6	9670	10,500	9610	2.9×10^6
H-3	7.35	4420	0.5	5.3×10^6	10,300	10,400	9300	3.2×10^6
H-3					10,650			
H-3					8900			
Average		4320		6.4×10^6	9730	10,400	9500	2.9×10^6

^aBased on the best straight line portion nearest the origin of a curve traced by signals from a 1-in. strain gauge extensometer and load values.

^bSimilar to "a" but crosshead motion vs load values determined the curve.

^cA ball joint load applier was used for this specimen but proved not good, values obtained were omitted from average; all others were tested using the fixed crossheads.

Note:

Tungsten powder Lot W-3A was used for cylinder H-1, W-162 used for H-3; Pb-13 was used for both cylinders.

TABLE IV
CHANGE IN DIMENSIONS AND WEIGHT OF
TUNGSTEN-LEAD PRESSED AT 20,000 PSI

<u>Time Days</u>	<u>Diameter (in.)</u>	<u>Length (in.)</u>	<u>Weight (g)</u>	<u>Remarks</u>
<u>Piece No. 3B^a</u>				
0	0.826	1.110	131.5	←cracks observed, probably occurred before observation
74	0.838	1.126	132.4	
82	0.839	1.126	132.4	
88	0.840	1.130	132.5	
95	0.840	1.130	132.5	
109	0.840	1.130	132.5	
126	0.840	1.130	132.5	
150	0.840	1.131	132.5	
250	0.840	1.131	132.5	
286	0.840	1.131	132.5	
<u>Piece No. 6B^b</u>				
0	0.808	1.150	126.2	←started to crack
35	0.810	1.153	126.3	
40	0.813	1.159	126.6	
64			127.0	
74			127.1	
211	0.827	1.193	127.2	
<u>Piece No. 6B-1^c</u>				
0	0.850	1.155	140.5	← no cracks
3	0.850	1.155	140.5	
11	0.850	1.155	140.5	
17	0.851	1.157	140.5	
26	0.851	1.158	140.55	
40	0.851	1.158	140.6	
57	0.851	1.158	140.6	
79	0.851	1.158	140.65	
188	0.852	1.159	140.65	
224	0.852	1.159	140.65	

^a60 vol% Pb pressed to 92.8% of theoretical density.

^b70 vol% Pb pressed to 94.6% of theoretical density.

^c70 vol% Pb pressed to 94.6% of theoretical density and sintered 2 h at 120°C in argon.

Fig. 11.

Theoretical density vs volume concentration of lead.

TABLE V
 CHANGES IN DIMENSIONS
 AND WEIGHT OF HOLLOW CYLINDER

Time Days	O. Diam (in.)	I. Diam (in.)	Length (in.)	Weight (g)
0	7.415	6.892	5.960	8145
34	7.416	6.893	5.960	8145
88	7.416	6.892	5.961	8140
201	7.418	6.885	5.962	8140
735	7.419	6.886	5.962	8139

Notes:

- (1) Scale accuracy ± 3 g.
- (2) Length measurements to ± 0.001 in.
- (3) Diametrical measurements were made at three unmarked vertical positions and at two positions 180 deg apart at each vertical position. Values listed are average values considered accurate to ± 0.003 .
- (4) Pb-13 and W-48 (21- μ m) powders used.