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DMIC

review

OF RECENT DEVELOPMENTS

Refractory Metals (Cb, Ta, Mo, W)

J. Van Orsdel, D. J. Maykuth, and V. D. Barth • April 12, 1968

COLUMBIUM

The objective of a program at Westinghouse Astronuclear Laboratory was to develop a columbium-base alloy suitable for application as a gas-turbine-bucket material.⁽¹⁾ Mechanical-property target values selected were a 100-hour, 2400-F stress-rupture strength/density parameter of 70,000 inches, with a minimum of 5 percent tensile elongation over the entire operating temperature range. Other requirements were (1) that the alloy be capable of being forged into a complex airfoil shape, (2) that it be resistant to thermal shock and thermal cyclic loading, (3) that it possess good fatigue characteristics and (4) that it display good costability and tolerance for minor coating defects. The alloy developed on this program, XB-88 (Cb-28W-2Hf-0.067C) was reported to offer a 600 to 800-F temperature advantage over the best available superalloy and a 300 to 400-F advantage over the current generation of columbium-base alloys. The best combination of low-temperature ductility and high-temperature creep strength was exhibited by material annealed 1 hour at 1700 C (3090 F) following extrusion and warm swaging. The 100-hour rupture strength at 2400 F was 25,500 psi (strength/density parameter of 68,500 inches). Room-temperature elongation was 11 percent. Rupture-life comparisons of this new alloy with other gas-turbine-bucket materials are shown in Figure 1.

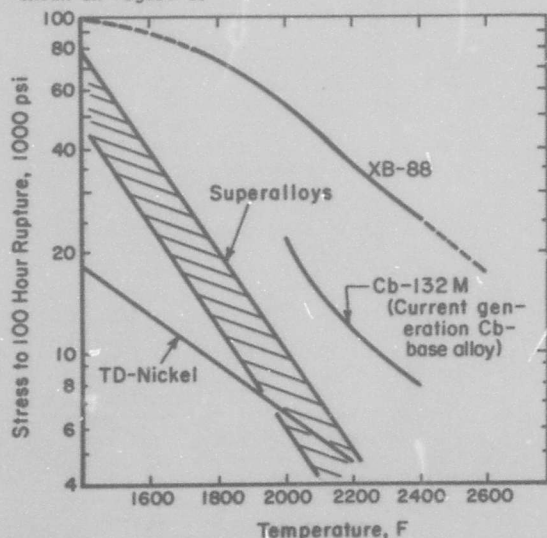


FIGURE 1. 100-HOUR RUPTURE STRENGTH VERSUS TEMPERATURE FOR XB-88 AND OTHER GAS-TURBINE BUCKET MATERIALS⁽¹⁾

The Astronuclear Laboratory of Westinghouse also recently concluded a comparative evaluation of the mechanical properties of the B-88 (Cb-28W-2Hf-0.067C) and Cb-1 (Cb-30W-12Zr-0.06C-0.032N) alloys.⁽²⁾ The creep resistance of Cb-1 alloy was not significantly different from B-88 alloy at 1315 C (2400 F). The ductile-to-brittle transition temperature of Cb-1 was 80 to 100-F higher than was that of B-88. The nitrogen-containing Cb-1 alloy experienced a potent 1-hour aging peak at 1200 C (2190 F). According to the investigators, this aging reaction apparently does not affect the creep resistance of Cb-1 at 1315 C, but would be expected to make a positive contribution to creep resistance at lower temperatures. Moreover, the phase relationships of the nitrogen-containing Cb-1 alloy were very similar to B-88, but the precipitates were much finer in the solution-plus aged conditions.

COLUMBIUM AND TANTALUM

Investigators at the RAI Research Corporation completed a study exploring the usefulness of easily deformable metals in diffusion-bonding columbium and tantalum to themselves and to each other.⁽³⁾ Filler-metal foils of aluminum, zinc, indium, tin, lead, and titanium were evaluated in various bonding experiments conducted in an argon atmosphere, using pressures to 2000 psi and temperatures to 500 C (930 F). Of the filler metals evaluated, best results were achieved with aluminum foil. Thus, at 2000 psi and 419 C (785 F), all three combinations of tantalum and columbium produced lap-bonds in 1/2 hour with aluminum foil between the faying surfaces. Under the same conditions when no foil was used, no bonds were obtained in 2 hours. Butt-bonded joints made under the same conditions exhibited tensile strengths of 12,000 psi for columbium and 19,850 psi for tantalum, even though, after rupture, poor joint efficiency was noted by the small fraction of contacts on the overall area.

The Bureau of Mines has issued another report⁽⁴⁾ in a series of three* which detail the results of a broad experimental program on evaluating the properties of columbium-hafnium- and tantalum-hafnium-based alloys. On this latest investigation over 30 alloys were prepared as button-type ingots and fabricated to sheet. The properties evaluated included hardness, electrical resistivity, elevated-temperature tensile properties, weldability, and oxidation behavior at 1000 C (1830 F) and 1400 C

* US Bureau of Mines Report of Investigation 6988 and 6777 were previously summarized in the Reviews of Recent Developments dated November 3, 1968, and July 28, 1967, respectively.

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(2550 F. In general, superior high-temperature mechanical properties were exhibited by alloys Cb-33Hf-10W-10Ti, Cb-20Hf-5Mo, and Cb-30Hf-5Mo (compositions in atomic percent).

TANTALUM

Standard Pressed Steel Company is in the early stages of a program to evaluate advanced tantalum alloys and tantalum coatings for use in mechanical fasteners.(5) The program consists of a screening phase and a fastener-evaluation phase. The purpose of the screening phase is to survey available alloys and coatings with respect to oxidation life up to 3500 F and to determine the effect of the coating on the ductile-brittle transition temperature of the substrates. The fastener phase will consist of the manufacture of fasteners and joints of the optimum alloy-coating combination and the evaluation of these at temperatures up to 3500 F.

MOLYBDENUM

Several interesting papers and reports relating to the diffusion, solubility, and/or effects of interstitial impurities in molybdenum have recently become available.

Two of these involved determinations of diffusion-coefficient values which are summarized as follows:(6,7)

Element in Molybdenum	Diffusion Coefficient cm ² /sec	Reference
Carbon	0.034 e ^{-41,000/RT}	6
Oxygen	0.0278 e ^{-25,200/RT}	7
Nitrogen	0.0615 e ^{-28,500/RT}	7

Rudman's work also included a determination of the solubility of carbon in molybdenum.(6) This was observed to follow the relationship

ln c_s = 16.78 - 2.29 x 10⁴/T

where c_s is the solubility in ppm by weight and T is measured in degrees Kelvin.

Stein examined the effect of impurities and orientation on the mechanical properties of molybdenum single crystals.(8) Most crystals were prepared by zone melting in 2 or 3 passes under vacuum (pressures from 2 x 10⁻⁸ to 4 x 10⁻⁷ torr). Such zone melting increased the resistivity ratios (298K/4.8K) from a value of 53 to values between 510 and 10,300. To explain these variations, correlations were sought with zone-melting pressure as well as with analyses of (1) gases in the vacuum before and after melting and (2) of the molybdenum crystals by standard- and mass-spectrometric techniques. No firm correlations were found, and the resistivity ratios were concluded to offer a "rather poor way of indicating the purity" of the molybdenum crystals.

ZrH₂ treatment of the single crystals reduced their carbon content from 21 ppm (maximum) to less than 10 ppm, while their oxygen and nitrogen content (<1 to 9 ppm) was not significantly changed.

Tensile and compressive properties of these crystals were determined over the range from 20 to 298 K.

The critical resolved shear stress (CRSS) and temperature dependence of CRSS were found to decrease with increasing purity. At all test temperatures, the CRSS law (i.e., Schmid's Law) fails. CRSS also was found to reach a minimum in the (100) tension axis orientation and a maximum in the tensile axis along the (110)(111) line of the stereographic triangle. Ductility, measured by reduction in area, was not improved by the ZrH₂ purification. However, in some orientations reductions in area as great as 100 percent at 78 K and 9 percent at 20 K were obtained.

Several English investigators have reported an extensive compilation of tensile creep data for unalloyed molybdenum at moderately low temperatures, i.e., 600 to 750 C (1110 to 1380 F).(9) Two types of molybdenum sheet (made by powder-metallurgical methods and arc melting, respectively) were evaluated in both the transverse and longitudinal test directions in the as-rolled, stress-relieved, and recrystallized conditions. Stress levels were varied from 2 to 20 tons/in², and deformation was measured over intervals up to 2000 hours. Selected data from this study are given in Table 1. In brief, both types of molybdenum were shown to withstand substantial stresses at 650 C. Creep resistance for longitudinal specimens was of the same order as that for transverse specimens and was greater for stress-relieved material than for recrystallized. Also, the powder-processed material had higher creep resistance at 650 C than did the arc-melted material.

TUNGSTEN-MOLYBDENUM-RHENIUM ALLOYS

Interest and support continues in the development and evaluation of tungsten-molybdenum-rhenium alloys, largely on the basis of their potential as nuclear construction materials having good low-temperature ductility in combination with high strength at elevated temperature. Two recent publications have described some property data for such alloys.

One of these was concerned with a determination of the tensile properties on an as-sintered W-30Mo-30Re (at percent) composition at temperatures from -100 through 1000 C (-148 through 1832 F).(10) Tensile strengths varying from 165,000 psi at room temperature to 92,000 psi at 1000 C, with corresponding elongation values of 10 and 17 percent, respectively, were obtained on samples that averaged 95.5 percent of theoretical density. Small-diameter tubing of this material also was cold- and/or warm-drawn through reductions in area up to 68 percent.

Flagella and Tarr have reported the results of bend and creep-rupture testing on 34 wrought ternary alloys in the tungsten-molybdenum-rhenium system.(11) Compositions of these, in atomic percent, ranged from 15 to 75 percent tungsten, 0 to 65 percent molybdenum, and 15 to 30 percent rhenium. No single alloy was outstanding for all of the properties evaluated, although several important trends were observed. While many alloys retained some ductility after high-temperature exposure, two compositions (W-25Re-60Mo and W-30Re-55Mo) were exceptional in this regard, as these both retained their initial room-temperature ductility (4T bend through 90 degrees) after 100-hour exposures at temperatures from 1200 C (2190 F) through 2400 C (4350 F).

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TABLE 1. SELECTED RESULTS OF TENSILE CREEP TESTING^(a) UNALLOYED MOLYBDENUM IN VACUUM⁽⁹⁾

Temperature,		Stress, tons/in ²	Elastic Modulus, 10 ⁶ psi	Plastic Strain, percent, at time given (hours)					
C	F			0	10	100	500	1000	2000
<u>Arc-melted; stress-relieved 1 hr/950 C</u>									
600	1112	20	35	0.104	0.190	0.520	-	-	-
650	1202	14	39	0.048	0.080	0.10	-	-	-
650	1202	20	28	0.040	0.300	1.23	-	-	-
750	1382	14	29	0.018	0.144	0.556	-	-	-
<u>Arc-melted; recrystallized 2 hr/1350 C</u>									
650	1202	2	--	0	0.006	0.014	0.032	0.044	0.052
650	1202	4	35	0.168	0.350	0.792	0.938	0.980	1.05
750	1382	4	24	0.063	0.260	0.414	0.662	0.814	1.00
<u>Sintered; stress-relieved 1 hr/950 C</u>									
600	1112	20	45	0	0.023	0.058	0.102	0.134	-
650	1202	20	37	0.018	0.078	0.148	0.268	0.360	-
750	1382	14	33	0	0.060	0.210	0.438	0.606	-
750	1382	20	58	0	0.200	0.88	-	-	-
<u>Sintered; recrystallized 1 hr/1250 C</u>									
650	1202	6	36	0.282	0.300	0.383	0.460	0.528	-
650	1202	8	42	0.423	0.460	0.554	0.722	0.858	-
750	1382	8	24	0.473	0.72	1.22	2.28	3.10	-

(a) All data given on samples cut transversely from rolled sheet of 0.040-inch thickness.

In 1600 C (2910 F) creep-rupture testing, alloys containing 10 to 30 percent molybdenum exhibited a transition in creep resistance in the region of 23 to 25 percent rhenium, apparently as a result of the single-phase boundary in this region. Thus, single-phase alloys (i.e., those of this group containing less than about 23 percent rhenium) showed a trend toward higher creep strengths. A similar and more pronounced effect was observed at 2200 C (3990 F). Selective creep-rupture data for a few of the alloys tested are given in Table 2. These include the W-25Re-30Mo and W-30Re-30Mo alloys, which are presently being considered for high-temperature applications.

TUNGSTEN

Vapor deposition of tungsten continues to be of industrial interest as a way of preparing tungsten. In research at Oak Ridge National Laboratory, simple shapes such as tungsten tubing in lengths up to nearly 4 feet have been made easily and less expensively than equivalent tubing made by the conventional extrusion-drawing method.⁽¹²⁾ Such tubes can be made in various diameters with wall thickness variations not exceeding ± 0.001 in. in the as-deposited condition. A thin-wall tube shell also was made in this way and was conventionally drawn through five passes to yield tubing of 0.708-inch O.D. with a wall thickness of 0.010 inch.

Procedures have been developed for the production of tungsten wire of uniform and controlled quality in diameters less than 10 microns (i.e., less than 0.4 mil).⁽¹³⁾ Among the procedures found useful in development is a torsional deformation test which measures torsion modulus, yield point of torsional shear, wire plasticity, and recrystallization temperature. The replacement of aluminum in the conventional wire dope by gallium is reported to yield wire with improved properties including a higher recrystallization temperature.

According to a General Electric report, the severe forming of tungsten sheet is best accomplished at a red heat (about 1200 F).⁽¹⁴⁾ For optimum formability at these temperatures, the tungsten sheet initially should be stress-relieved at 1650 to 2000 F to produce structures containing from 1 to 5 percent recrystallization. These workers caution that sheet with this type of structure may not be well suited for less severe forming operations at room temperature.

In a preliminary study, W-3.8 vol % ThO₂ sheet has been produced by Westinghouse investigators.⁽¹⁵⁾ Provisional test results obtained at 2200 C (4000 F) yielded strength values of about four times those characteristic of pure tungsten. Elevated-temperature properties are expected to be further improved when process parameters are optimized.

TABLE 2. SELECTED CREEP-RUPTURE DATA^(a) FOR (11)
 TUNGSTEN-RHENIUM-MOLYBDENUM ALLOYS

Alloy Composition, at. %	6,000-psi Stress at 1600 C (2912 F)				800-psi Stress at 2200 C (3992 F)			
	Time to Indicated Strain, hours		Rupture Time, hours	Linear Creep Rate min ⁻¹	Time to Indicated Strain, hours		Rupture Time, hours	Linear Creep Rate min ⁻¹
	0.5%	10%			0.5%	10%		
W-15Re-55Mo	0.65	18.0	18.0	6.0×10^{-5}	3.25	-	48.1	1.6×10^{-5}
W-25Re-60Mo	0.07	--	1.34	--	0.33	4.18	4.34	2.1×10^{-4}
W-30Re-55Mo	0.26	1.67	1.73	--	0.32	3.46	3.94	2.6×10^{-4}
W-25Re-30Mo	1.5	13.9	14.8	6.1×10^{-5}	0.90	19.2	28.8	8.1×10^{-5}
W-30Re-30Mo	0.08	4.7	10.0	3.0×10^{-4}	0.43	9.7	22.0	1.6×10^{-4}
W-26Re	2.0	35.8	45.4	3.9×10^{-5}	0.61	13.8	30.6	1.1×10^{-4}

(a) All alloys tested as 0.020-in. sheet in a hydrogen atmosphere. Prior to testing, each alloy was annealed 2 hours at the test temperature used.

The extrusion of tungsten at relatively low temperatures has been the objective of a Soviet investigation.⁽¹⁶⁾ Successful extrusion (reductions of 1-inch-diameter rod stock of 77 to 80 percent in area) was carried out in the 500 to 600 C (930 to 1110 F) temperature range by the use of Armco iron sheaths. For smooth as-extruded tungsten surfaces, starting billets must not have coarse grains. Considerably better die life as compared with die life in the hot-extrusion procedure was reported.

Tungsten and tungsten-alloy tubing have been extruded successfully from arc-cast and powder-metallurgy billets by use of the floating mandrel technique, with naturally forming tungsten oxide as the lubricant.⁽¹⁷⁾ Figure 2 illustrates schematically the technique employed. Extrusion temperatures ranged from 1650 to 2250 C (3000 to 4100 F) at extrusion ratios in the range of 4 to 9. Satisfactory service was obtained from zirconia-protected dies and mandrels, but surface melting of the container sleeve began when billet temperatures exceeded 1850 C (3360 F). Rapid extrusion speeds were necessary to minimize heat loss and reduce contact time with the tooling. Thin-walled tungsten tubing (sizes not disclosed) has been produced with the floating-mandrel technique by reextruding a section of tube shell in a duplex billet.

A novel process for making tungsten shapes has been patented recently.⁽¹⁸⁾ In the process, metal powder is fluidized with a meltable binder (e.g., paraffin plus oleic acid), and then cast at low pressure into a shape. After removal of the fluidizing medium, by vaporization for example, sintering is carried out to densities of at least 18.0 g/cm³. Surface finishes in the 30 to 60 μ in., rms range are produced. The process is said to be adaptable at low cost to the production of complex shapes. Among the applications reported are preforms for machine finishing or forging, molds, processing crucibles, and missile hardware.

The tungsten-hafnium-carbon alloys, previously shown to possess high strength, have been further characterized and shown to be heat treatable.⁽¹⁹⁾

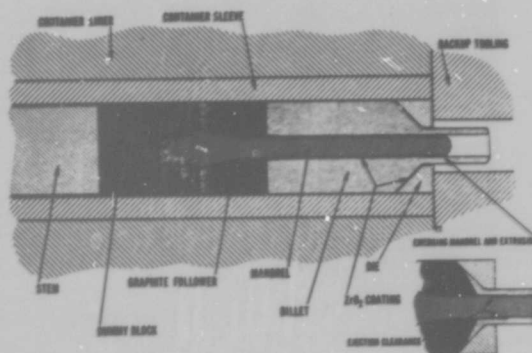


FIGURE 2. FLOATING-MANDREL TECHNIQUE USED TO PRODUCE TUNGSTEN TUBING AT OAK RIDGE NATIONAL LABORATORY⁽¹⁷⁾

The minimum solution temperature of hafnium carbide in tungsten is approximately 4600 F, and 1 hour at 2500 F is an effective aging temperature. A tensile specimen heat treated in this way displayed a strength at 3500 F of 69,000 psi, i.e., approximately a sevenfold improvement over unalloyed tungsten. One solution treated and aged specimen at 3500 F showed a minimum creep rate of 10^{-6} second⁻¹ under a stress of 23,000 psi. For high-strength applications at 3500 F, compositions containing equiatomic amounts of carbon and hafnium, approximately 0.3 to 0.4 percent, are recommended.

Further determinations of elastic moduli in tungsten from room temperature to 1480 C (2700 F), have been made by an ultrasonic pulse-echo method, and compared with recent data from other investigations.⁽²⁰⁾

Radial heat-flow measurements of the thermal conductivity of W-26 wt % Re have been made over the temperature range 300 to 2200 C (570 to 3990 F).⁽²¹⁾ Data indicate a small positive temperature coefficient on conductivity, with conductivity values ranging from about 56.0 Wm⁻¹ deg⁻¹ at 300 C to about 69.0

$\text{Wm}^{-1} \text{ deg}^{-1}$ 2200 C. Electrical resistivity measurements are also reported for both W-26 wt % Re and for unalloyed tungsten.

Tungsten oxidation equilibria have been measured by a solid-electrolyte galvanic-cell technique.⁽²²⁾ Estimates of ΔG_f° for tungsten-oxygen reactions in the 700-1000 C (1290-1830 F) temperature range were as follows:

Reaction	ΔG_f° , cal./g-atom oxygen
$1/2 \text{ W} + 1/2 \text{ O}_2 = 1/2 \text{ WO}_2$	$-68,660 + 20.31 T_K$
$1/2.72 \text{ W} + 1/2 \text{ O}_2 = 1/2.72 \text{ WO}_{2.72}$	$-66,272 + 18.90 T_K$
$1/2.90 \text{ W} + 1/2 \text{ O}_2 = 1/2.90 \text{ W}_{2.90}$	$-66,371 + 19.23 T_K$
$1/3 \text{ W} + 1/2 \text{ O}_2 = 1/3 \text{ WO}_3$	$-66,384 + 19.48 T_K$

According to Bureau of Mines work, tungsten and tungsten-cobalt alloys containing up to 9.7 wt % cobalt oxidize according to the linear-rate law at 1000 and 1100 K (1340 and 1520 F).⁽²³⁾ At 9.7 wt % cobalt, threshold reduction in oxidation rate at 1000 K occurs. At 29.8 wt % cobalt, a specimen of the alloy exposed for over 2 hours at 1100 K was about 17 times more resistant to oxidation than that of the 9.7 wt % cobalt alloy. The principal components of the protective scale were Co_3O_4 at the outer surface, a CoWO_4 intermediate zone, and a $\text{W}_{18}\text{O}_{49}$ inner zone.

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