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Investigation of a High-Density Tungsten-Base Alloy

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INVESTIGATION OF A HIGH-DENSITY TUNGSTEN-BASE ALLOY

by

H. Sheinberg, J. T. Frakes, and R. E. Riley

ABSTRACT

The effect of sintering time, temperature and atmosphere, post-sintering heat treatments, tungsten particle size, and compositional variations upon the mechanical properties and structure of a 95 wt% W-3.5 wt% Ni-1.5 wt% Fe base alloy were investigated. Improvements were observed with postsintering heat treatments, fine tungsten powders, and small rhenium or chromium additions.

I. INTRODUCTION

The 95 wt% W-3.5 wt% Ni-1.5 wt% Fe alloy investigated in this program comprised grains of tungsten distributed in a matrix phase of W-Ni-Fe formed when the initial binder phase, Ni-Fe, takes tungsten into solution from the dispersed tungsten grains. We attempted to understand the effects of initial tungsten powder size, sintering and heat-treatment schedules, atmosphere, and binder additives upon the tensile properties and ductility of this hydrostatically pressed and sintered material, and to attempt to correlate these effects with observed microstructures.

Variations in initial particle size and in sintering and heat-treatment schedules were anticipated to exhibit an effect upon the density, grain size, and dissolution of tungsten into the binder phase. The differences in atmospheres were expected to reveal the effect of hydrogen embrittlement in the alloy. Finally, the use of various additives was expected to affect the distribution and strength of the binder phase and, possibly, the ductility of the tungsten grains.

II. RAW MATERIALS

Four lots of tungsten powders, three lots of nickel, and one lot of iron powder were used in this investigation. The chemical analyses of these powders are recorded in Table I and the general powder characteristics are shown in Table II. A

brief explanation of powder characterization is given in Sec. IV. Lots W-44, 155, and 164 are hydrogen reduced tungsten powders. The commercial nickel powders were all prepared by decomposition of $\text{Ni}(\text{CO})_4$ and the iron was also a carbonyl type powder.

The microstructure of representative tungsten, iron, and nickel powders is shown in Figs. 1 and 2, and the particle-size distributions are presented in Figs. 3 and 4.

III. SAMPLE PREPARATION

The powders described previously were used in the as-received condition. For the three principal blends of the standard composition employing coarse, intermediate, and fine tungsten powders, the tungsten was weighed to ± 1 g and the nickel and iron to ± 0.1 g. The weights of these blends were 59.0, 18.2, and 12.0 kg, respectively; the largest was blended in a N_2 atmosphere in a 1-ft³ capacity Patterson-Kelley twin shell blender for 14 h. The other two lots were similarly blended in an 8-qt Patterson-Kelley twin shell blender. When small (1500-g) charges were employed to study compositional variations, the tungsten was weighed to ± 0.01 g. These charges were blended in inert atmosphere in rotating pint bottles equipped with aluminum agitator wires. The bottles revolved at 60 rpm for 14 h. In preparing rods to be machined into tensile specimens, blended powders were poured into 7/8-in. i.d.

TABLE I
CHEMICAL ANALYSES OF POWDERS^a

Powder Lot Element	Ni-21 ppm	Ni-23 ppm	Ni-24 ppm	Fe-31 ppm	W-44 ppm	W-155 ppm	W-164 ppm
Li	< 4	< 4	< 4	< 10		< 1	< 3
Be	< 4	< 4	< 4	< 10	< 10	< 1	< 1
B	< 10	< 10	< 10	< 100	< 10	< 3	< 3
Na	< 4	< 4	< 4	< 30	N.D.	< 3	< 3
Mg	< 1	< 1	< 1	< 3	30	< 1	< 1
Al	< 8	< 8	< 8	< 10	< 10	< 10	< 10
Si	< 8	< 8	< 8	< 3	100	< 10	< 10
K	< 20	< 20	< 20	< 30		< 10	100
Ca	< 1	< 1	< 1	< 10	N.D.	< 3	10
Ti	< 8	< 8	< 8	< 10	< 100	< 30	< 30
V	< 10	< 10	< 10	< 30	< 100	< 10	< 10
Cr	< 4	< 4	< 4	10	10	< 10	< 10
Mn	< 4	< 4	< 4	< 3	10	< 3	< 3
Fe	10	10	10		100	< 3	10
Co	< 40	< 40	< 40	< 10	< 100	< 10	< 10
Ni				100	300	< 10	< 10
Cu	< 4	< 4	4	< 10	< 10	< 3	< 1
Zn	< 100	< 100	< 100	< 100	< 100	< 300	< 300
Sr	< 8	< 8	< 8	< 10		< 10	< 10
Nb					< 300	< 300	< 300
Mo	< 20	< 20	< 20	< 100	100	< 100	< 100
Ag	< 1	< 1	< 1		< 10	< 3	3
Cd	< 80	< 80	< 80	< 10			
Sn	< 20	< 20	< 20	< 30	< 10	< 10	< 10
Ba	< 8	< 8	< 8	< 10		< 3	< 3
Pb	< 8	< 8	< 8	< 30	< 10	< 10	< 10
Bi	< 8	< 8	< 8	< 30	< 10	< 10	< 10
Ta					< 300		
Zr					< 100	< 30	< 30

^aWhere values are not reported, no determinations were made.

TABLE II
POWDER CHARACTERISTICS

	Ni-21	Ni-23	Ni-24	Fe-31	W-44	W-155	W-164
Bulk density (ρ_B) g/cm ³	1.07	2.43	2.28	2.41	4.54	4.01	7.58
Tap density (ρ_T) g/cm ³	1.59	3.43	3.68	3.66	6.59	5.84	10.12
ρ_T/ρ_B	1.48	1.41	1.61	1.52	1.45	1.45	1.33
Surface area m ² /g, BET	0.85	0.94	0.50	-	0.62	0.78	0.12
FSS av particle size, μ m	1.7	3.9	2.5	4.4	3.8	1.7	9.1
Median particle size, Sedibal, μ m	10.3	6.8	9.2	7.3	4.5	2.8	10.0

plastisol (polyvinyl chloride) sacks and jolted lightly to a powder height of ~5-1/2 in. A plastisol lid was inserted on top of the powder and sealed to the sack with a liquid solvent consisting of plastisol dissolved in a 50-50 mixture of acetone and methylethyl ketone. The same procedure was used to load and seal 2-in. i.d. sacks to press cylinders for machining flexure specimens. Sacks were placed in tight fitting perforated micarta sleeves (to

ensure relatively straight pressings) and then were placed in a perforated metal basket and hydrostatically pressed at 50,000 psi in a 12-in. i.d. chamber. Pressing time was approximately 20 min. Pressed cylinders were removed from the sacks and scribed to maintain identity during subsequent sintering.

A 0.03-in.-thick walled tungsten can (2.3 in. o.d. by 7.0 in. long), with a perforated bottom to

250X

W-155

100X

W-164

500X

W-155

250X

W-164

Fig. 1. Microstructure of tungsten powders.

100X

Fe-31

100X

Ni-21

250X

Fe-31

250X

Ni-21

Fig. 2. Microstructure of iron and nickel powders.

Fig. 3. Particle-size distributions of tungsten powders.

allow for gas flow, was used for sintering the alloy pressings. A porous Al_2O_3 disk, 3/8-in. thick, covered the perforated bottom and supported the pressings that were surrounded by and covered with -60 mesh Al_2O_3 . A calibrated W-(W-Re) thermocouple was inserted into the Al_2O_3 adjacent to the pressings to monitor temperature. The can was centered vertically in the 3.0 in. i.d. by 20.0 in. long resistance heated tungsten tube of a furnace manufactured by General Electric.

The standard sintering cycle consisted of heating in hydrogen to 1300°C in 120 min, holding at 1300°C for 120 min, heating to 1520°C (or other designated temperature) in 30 min, and holding at 1520°C for 60 min. The furnace was cooled to 1200°C in 20 min and held at that temperature for 20 min while the atmosphere was changed to argon. Finally, the power was turned off and the furnace tube was cooled in an argon atmosphere.

After unloading, immersion densities were calculated on the pieces to check the degree of sintering. These numbers were for information only and are not submitted as data.

Fig. 4. Particle-size distribution of iron and nickel powders.

The pressings were machined to right cylinders, cut to length, and center holes were drilled in both ends of each specimen. The cylinders were then turned to 0.435 in. diam with a Kennametal grade 907 carbide tool; 0.02-in. cuts were taken on the radius. A proprietary liquid, Tap Magic, diluted with conventional cutting oil, was used to facilitate machining. The specimens were then machined (using 0.02-in. cuts) with a tracer template arrangement to within ~0.020 in. of the finished diametric dimensions of a standard 1/4-in. tensile specimen with 1.0-in. gauge length (ASTM E8-66, p. 207, Fig. 8). The final tracer cuts were only ~0.005 in. Unified 7/16-14 threads were ground on each end.

Grinding to the required dimensions and specified 16-rms finish in the reduced section was accomplished with an 80-grit silicon carbide wheel turning at 3400 rpm according to the following schedule:

First pass:	0.001-in. maximum
Second pass:	0.001-in. maximum
Third pass:	0.0005-in. maximum to spark.

Simcool spray diluted about 40 to 1 was used as the grinding coolant. The specimens were inspected for complete compliance to ASTM and finish specifications. Close control over machining eliminated, as much as possible, variations in this operation that conceivably could mask effects of intentionally introduced variables.

IV. TESTING PROCEDURES

A. Powder Characterization

The average particle size of powders was determined in accordance with ASTM Designation B 330-58, "Average Size of Refractory Metals and Compounds by Fisher Sub-Sieve Sizer (FSS)." The method is designed to measure the specific surface area of a powder by determination of resistance to fluid flow through a packed column of powder and subsequent calculation of powder size from these values. The equipment includes an air pump, pressure regulator, precision manometer, precision calibrated sample tube, and a calculator chart for direct observation of average particle size.

Measurement of the bulk or apparent density (ρ_B) was made according to ASTM designation B-329-61, "Apparent Density of Refractory Metals and Compounds by the Scott Volumeter." A brass funnel and metal screen directs powder into a glass baffle box with two wooden sides and a funnel at the base of the box directs powder into an accurately sized (1.00 in.³) and tared density cup.

Tap density was determined by filling an accurately tared 10 cm³ graduate with powder and tapping the graduate with a plastic rod until no further reduction in volume was observed with continued tapping. The quotient, powder weight/minimum volume, is the tap density.

Particle-size distribution was derived from sedimentation data obtained with a continuously recording sedimentation balance. The powder specimen is dispersed in a 50-50 ethylene glycol-distilled water solution and allowed to settle; a time-cumulative weight record allows application of Stokes Law for calculation of cumulative weight percent vs particle size.

The surface area of the powders was determined by using a Numec Model AFA-2 adsorption flow apparatus to measure the volume of N₂ gas required to form a monomolecular layer on the specimen surface at liquid N₂ temperature.

B. Sample Testing

Mechanical testing was performed on an Instron testing machine utilizing a crosshead speed of 0.005 in./min. The tensile strength was the maximum load carried during the tension test divided by the original cross-sectional area of the specimen. The yield strength was based on a 0.2% offset from the approximate modulus. This approximate modulus was based on the best straight line drawn tangent at the origin of a curve traced from signals from a 1-in. gauge length strain gauge extensometer and load values. Because the extensometer was not an averaging type, the values were not too accurate, and were used only as a base for yield strength calculations. Elongation was determined by measuring the gauge length after fracture and by comparing this value to the original gauge length, expressed as a percent increase in length (ASTM E-8-66).

The procedure for estimating grain size was essentially the mensuration procedure described as one of the ASTM standard methods for estimating the average grain size of metals (ASTM E 112-63). Photomicrographs of a representative field of the test specimen were compared to standard grain size charts and the value of grain size most closely resembling the test specimen was chosen.

The hardness determination method is described in ASTM E 92-67, and is a Vickers hardness obtained by using a diamond indenter and evaluation of its impression after forcing this indenter under a predetermined load into the surface of the material being tested.

Density values were computed by applying immersion techniques to machined tensile and flexure specimens.

V. DISCUSSION OF RESULTS

The process of densification believed to occur in these tungsten alloys was described by Krock;¹ it proceeds first in the solid state and later as liquid-phase sintering when the temperature reaches a sufficient level. In the standard sintering schedule used in this investigation (Table III, footnote c), the solid-state sintering occurred primarily during the 2-h hold at 1300°C. This solid-state sintering comprised initial solution of tungsten into the Ni-Fe matrix phase followed by tungsten diffusion through the W-Ni-Fe boundary or through the Ni-Fe phase. This transport is

TABLE III

Sample No. ^a	SAMPLE DESCRIPTIONS Variation (Standard Composition, ^b Heat Treatment, ^c and Atmosphere ^c Unless Indicated)	Theoretical Density (g/cm ³)
C-1480-1	Sinter 30 min at 1480°C	18.16
C-1500-2	Sinter at 1500°C	18.16
C-1520-1	Sinter 30 min	18.16
C-1520-2	Standard sample for coarse W powder	18.16
C-1520-4	4 h at 1400°C Ar anneal	18.16
C-1520-5	8 h at 1400°C Ar anneal	18.16
C-1520-10	Hold at 1400°C for 1 h on heat up instead of 1300°C for 2 h	18.16
C-1520-12	4 h at 1050°C postsinter treatment in vacuum	18.16
C-1520-13	No hold at 1300°C on heat up	18.16
C-1520-14	Complete cycle in H ₂	18.16
C-1520-15	Heat to 1300°C in H ₂ , remainder of cycle in Ar	18.16
C-1460-18	1.5 wt% of the total 3.5 wt% Ni plated on the W powder	18.16
C-1460-19	2.3 wt% of the total 3.5 wt% Ni plated on the W powder	18.16
C-1520-21	95.00 wt% W + 3.15 wt% Ni + 1.35 wt% Fe + 0.50 wt% Zr	18.10
C-1520-22	95.00 wt% W + 2.80 wt% Ni + 1.20 wt% Fe + 1.00 wt% Zr	18.03
C-1520-25	94.50 wt% W + 3.13 wt% Ni + 1.34 wt% Fe + 1.03 wt% Hf	18.20
C-1520-26	93.97 wt% W + 2.78 wt% Ni + 1.19 wt% Fe + 2.06 wt% Hf	18.24
C-1520-27	94.47 wt% W + 2.78 wt% Ni + 1.19 wt% Fe + 1.56 wt% ThO ₂	18.14
C-1520-28	94.73 wt% W + 3.14 wt% Ni + 1.35 wt% Fe + 0.78 wt% ThO ₂	18.15
C-1520-31	92.90 wt% W + 2.73 wt% Ni + 1.16 wt% Fe + 3.12 wt% Re	18.45
C-1520-32	95.00 wt% W + 3.50 wt% Ni + 1.00 wt% Fe + 0.50 wt% Zr	18.12
C-1520-34	95.00 wt% W + 5 wt% [15Cr-7Fe-78Ni alloy]	18.15
F-1480-1	Sinter 30 min at 1480°C	18.16
F-1500-2	Sinter at 1500°C	18.16
F-1510-1	Sinter 30 min at 1510°C	18.16
F-1510-2	Sinter 30 min at 1510°C, plus 4-h vacuum anneal at 1050°C	18.16
F-1520-1	Sinter 30 min at 1520°C	18.16
F-1520-2	Standard sample for fine W powder	18.16
F-1520-4	8 h at 1400°C Ar anneal	18.16
F-1520-5	4 h at 1050°C vacuum anneal	18.16
F-1520-6	No hold at 1300°C on heat up	18.16
F-1520-9	95.00 wt% W + 3.15 wt% Ni + 1.35 wt% Fe + 0.50 wt% TaC	18.24
I-1500-3	Sinter at 1500°C, 4 h at 1050°C vacuum anneal	18.16
I-1500-4	Sinter at 1500°C	18.16
I-1500-5	Sinter at 1500°C 95.0 wt% W + 2.8 wt% Ni + 1.2 wt% Fe + 1.0 wt% Cr	18.09
I-1500-6	Sinter at 1500°C 95.0 wt% W + 2.24 wt% Ni + 0.96 wt% Fe + 1.80 wt% Cr	18.03
I-1520-2	Standard sample for intermediate W powder	18.16

^a The letter prefix to the sample number indicates the size of tungsten powder used in the sample; C, F, and I denote coarse, fine, and intermediate powder, respectively. The four-digit number following the letter indicates the sintering temperature used. The final number of the three-part designation is merely a specimen designation number.

^b Standard composition was 95.0 wt% W + 3.5 wt% Ni + 1.5 wt% Fe.

^c Standard heat-treatment schedule and atmosphere was as follows: heat to 1300°C in H₂ in about 2 h; hold for 2 h; heat to 1520°C in 30 min; hold 1-h; cool to 1200°C in 20 min; hold for 20 min while changing to argon atmosphere; and finally, turn off power and cool in argon.

apparently an activated process wherein nickel accelerates the movement of tungsten by creating lower energy transport paths. Growth of the dispersed particles occurs by particle agglomeration preceding volume diffusion of tungsten. The higher melting point of the tungsten component is taken into solution in the liquid phase, is transported by volume diffusion, and is reprecipitated onto solid particles of the higher melting phase.

Descriptions of the various samples tested are given in Table III. These samples incorporated variations in tungsten particle size, sintering schedule, atmosphere, annealing, and compositional additives. General and specific observations were made with respect to these variables and are presented below. Table IV presents the properties determined for the various specimens, and in most cases the numbers presented are averages of three values. The

TABLE IV
PROPERTIES OF W-Ni-Fe BASE ALLOYS

Sample No.	Tensile Strength (psi x 1000)	Yield Strength (psi x 1000)	Elongation (%)	Modulus (psi x 10 ⁶)	Grain Size (Av Diam mm)	Hardness (dph)	Density (g/cm ³)	(% TD)
C-1480-1	(A)	(A)	(A)	41.8	0.011	150	16.94	93.28
C-1500-2	(A)	(A)	(A)	(A)	0.011	165	16.03	88.27
C-1520-1	94.0	83.1	2.3	43.0	0.025	280	17.83	98.18
C-1520-2	124.8	86.0	8.5	35.2	0.045	310	18.03	99.28
C-1520-4	124.2	84.0	11.5	54.0	0.045	320	17.91	98.62
C-1520-5	128.5	85.6	17.0	37.6	0.045	315	18.15	99.94
C-1520-7	124.6	81.0	18.0	36.7	0.038	295	18.14	99.89
C-1520-12	123.6	84.6	12.0	56.0	0.045	310	17.98	99.01
C-1520-13	100.0	85.4	3.0	42.4	0.032	315	18.04	99.34
C-1520-14	106.2	76.6	8.0	39.8	0.053	265	18.07	99.50
C-1520-15	121.0	78.4	17.3	37.5	0.038	275	17.68	97.36
C-1460-18	96.3	95.5	2.0	41.8	0.053	315	18.05	99.39
C-1460-19	96.3	95.7	2.0	39.1	0.053	310	18.09	99.61
C-1520-21	89.6	71.4	3.3	35.4	0.038	250	16.98	93.81
C-1520-22	83.7	70.3	2.0	34.3	0.035	275	16.80	93.18
C-1520-25	97.5	74.5	3.0	37.7	0.032	275	17.49	96.10
C-1520-26	80.0	69.9	1.3	69.2	0.032	240	17.12	93.86
C-1520-27	103.0	75.0	6.3	45.3	0.032	280	17.39	95.87
C-1520-28	77.9	78.5	< 1.0	49.6	0.038	275	17.50	96.42
C-1520-31	128.4	122.5	1.3	37.6	0.019	400	18.42	99.84
C-1520-32					0.053	325	17.66	97.46
C-1520-34	83.9	79.1	2.0	57.9	0.064	305	17.76	97.85
F-1480-1	(A)	(A)	(A)	38.2	0.027	315	17.79	97.96
F-1500-2	(A)	(A)	(A)	36.9	0.013	310	17.29	95.21
F-1510-1	103.4	87.6	3.0	36.0	0.032	310	18.10	99.67
F-1510-2	133.0	92.2	25.0	74.7	0.027	315	18.01	99.17
F-1520-1	123.5	87.7	11.3	53.2	0.032	320	17.95	98.84
F-1520-2	130.8	86.3	26.7	38.0	0.035	315	18.03	99.28
F-1520-4	123.2	84.2	16.7	41.5	0.045	320	18.16	100.00
F-1520-5	124.9	86.9	25.3	41.0	0.050	315	18.03	99.28
F-1520-6	116.1	88.4	7.0	37.3			18.03	99.28
F-1520-9	99.3	85.4	3.0	56.9	0.027	325	17.98	98.57
I-1500-3	97.4	81.1	2.0	43.5			18.03	99.28
I-1500-4	90.8	82.1	2.0	40.8	0.045	310	17.92	98.68
I-1500-5	107.8	78.6	4.3	65.6	0.032	320	17.65	97.57
I-1500-6	114.3	92.2	2.0	41.8	0.022	370	17.69	98.11
I-1520-2	94.4	83.0	4.0	42.0	0.053	310	18.13	99.83

(A) Failure occurred in threads of sample.

average deviation for these values was about $\pm 3\%$. Many sequential numbers are absent from both Table III and Table IV. Many more samples were made and scribed with numbers than were tested. Early results with sintering schedules and sintering atmosphere studies obviated the need for some previously anticipated investigations. Some samples numbered beforehand and assigned to specific investigations were not used and, therefore, were omitted from the tables. As mentioned previously, flexure test specimens were prepared. After a few tests, however, it became obvious that this material was too ductile to furnish meaningful values of flexure strength and this phase of testing was discontinued.

As seen from the powder characteristics discussed in Sec. II, three general size ranges of tungsten powder were used in this study. There was fine (1.7 μ m), intermediate (3.8 μ m), and coarse tungsten (9.1 μ m). The nickel powders used have also been described previously. Three lots of nickel powder were used with the tungsten powders due to limitations of available quantities. An effort was made to choose these nickel powders so that they would be as similar as possible. No property variations were attributed to variations in the nickel powders. The green or as-pressed strength was determined for samples of both the fine and coarse tungsten batches of standard 95 wt% W: 3.5 wt% Ni:1.5 wt% Fe composition. These values,

as determined by flexure testing, were 1220 and 750 psi, respectively. Metallography of the coarse and fine standards in the green state is shown in Fig. 5.

Tungsten alloys, prepared by liquid phase sintering, generally exhibit a characteristic microstructure such as sample C-1520-2 (Fig. 6), which consists of rounded particles of tungsten dispersed in a W-Ni-Fe matrix. This microstructure is significantly different from that of sample C-1480-1 (Fig. 6), which is of the same composition sintered under an identical schedule except that it had a sintering temperature of 1480°C rather than 1520°C. This temperature was too low to effect significant liquid phase sintering. The metallography of standard samples prepared with coarse, fine, and intermediate tungsten powders are presented as sample C-1520-2 (Fig. 6), F-1520-2 (Fig. 7), and I-1500-4 (Fig. 8), respectively. In general, with specimens differing only in the initial particle size of the tungsten powder, samples fabricated with the finer tungsten showed better strength and elongation. Near-theoretical density was attainable at shorter times and lower temperatures than required for more coarse powders (sample F-1510-1, Fig. 9).

In analyzing the effects of variations in sintering conditions, the following conclusions were drawn. (a) At a given sintering time, increases in temperature up to an optimum for densification led to increases in ultimate tensile strength, elongation, and grain size for all initial tungsten powder sizes utilized. (b) At a given sintering temperature, increases in sintering time yielded increased ultimate tensile strength, elongation, and grain size up to the point where excessive grain growth became counterproductive. Insufficient sintering times and temperature produced materials of unsuitable structures (sample C-1480-1, Fig. 6, and sample F-1500-2, Fig. 9). These specimens, with densities far below theoretical values, failed in the threads of the tensile test specimens.

Utilizing hydrogen for the complete cycle resulted in significantly lower strength values, large grains, and low hardness values. Switching from hydrogen to argon at 1300°C during the early part of the standard cycle resulted in increased ductility, but lower yield strength.

Elimination of the 2-h hold at 1300°C during heat up yielded a decrease in grain size and significant decreases in ultimate tensile strength and elongation. Eliminating this pause precluded much of the solid-state sintering in the material before liquid phase transport. A 1-h substitution at 1400°C for the standard 2-h at 1300°C decreased the yield strength slightly, but resulted in a significant increase in elongation and reduction in grain size.

With specimens made with the coarse tungsten powder, anneals in argon or vacuum appeared to decrease yield strengths slightly, but increased the ductility with no apparent effect on grain size. With the fine tungsten samples, however, no improvement was noted on the already excellent ductility of the standard fine tungsten sample. Increases in grain size were noted. The 8-h argon treatment did seem to increase the tungsten particle-to-particle joining. With the fine tungsten samples sintered at 1510°C for 30 min, the 4-h vacuum anneal helped properties markedly, manifesting significant increases in strength and elongation. Vacuum annealing the samples made with intermediate tungsten powder had no effect on ductility, but did result in an increased ultimate tensile strength.

The experimental variations that most affected the microstructure and properties were additives to the binder phase. Additions of Zr, Hf, ThO₂, and TaC were deleterious to properties generally, although this might be misleading because the material produced did not approach theoretical density. Microstructures of the resultant materials are shown as follows: TaC additions as sample F-1520-9 (Fig. 7), hafnium additions as sample C-1520-26 (Fig. 10), and ThO₂ additions as sample C-1520-27 (Fig. 10). The microstructure of the material with zirconium additions is not shown because it appeared similar to the standard material. We hoped that the zirconium or hafnium additions might strengthen the binder phase through an alloying or compound forming process. The ThO₂ and TaC additions were anticipated to provide a dispersion strengthening effect. Either these improvements were not manifested or the effect was masked by the low densities obtained in the materials. Further investigations

250X

Coarse Standard

250X

Fine Standard

500X

Coarse Standard

500X

Fine Standard

Fig. 5. Microstructure of standard composition of W-Ni-Fe pressings in green state utilizing coarse and fine tungsten.

100X

C-1480-1

100X

C-1520-2

500X

C-1480-1

500X

C-1520-2

Fig. 6. Microstructure of W-Ni-Fe alloy of standard composition.

100X

F-1520-2

100X

F-1520-9

500X

F-1520-2

500X

F-1520-9

Fig. 7. Microstructure of W-Ni-Fe alloys of standard composition (left) and with TaC additions (right).

100X

I-1500-4

100X

I-1500-6

500X

I-1500-4

500X

I-1500-6

Fig. 8. Microstructure of W-Ni-Fe alloys of standard composition (left) and with chromium additions (right).

100X

F-1510-1

100X

F-1500-2

500X

F-1510-1

500X

F-1500-2

Fig. 9. Microstructures of W-Ni-Fe alloys of standard composition.

100X

C-1520-26

100X

C-1520-27

500X

C-1520-26

500X

C-1520-27

Fig. 10. Microstructure of W-Ni-Fe alloys with hafnium additions (left) or ThO_2 additions (right).

of sintering schedules would be necessary to determine the correct combination for obtaining dense material.

Additions of chromium yielded some success in strengthening the material. The smaller additions increased the ultimate tensile strength and elongation, and kept the grain size small, but were detrimental to the yield strength. Higher chromium additions, however, led to an increase in the ultimate tensile strength, yield strength, and hardness by again keeping the grain size small. Microstructure of the material with the higher chromium additions is shown in sample I-1500-6 of Fig. 8.

The rhenium addition showed the most marked effect of any additive. Hopefully, the rhenium addition would strengthen the matrix phase and toughen the tungsten particles. As seen in Fig. 11, the microstructure obtained was entirely different from that of the standard structure. The material with the rhenium addition exhibited the highest strengths, greatest hardness, and smallest grain size of any material tested, but had less ductility.

A Cr-Ni-Fe alloy was used as a binder material in specimen C-1520-34. The photomicrograph in Fig. 11 shows that some of the tungsten grains had a fuzzy texture, the cause of which is unknown. Properties were poorer than those of the standard, but again the density was not near theoretical. This specimen was sintered for 1 h at 1520, 1540, and 1560°C in an effort to improve the density. Some improvement was effected, but we felt that further attempts at densification were not warranted.

In samples C-1460-18 and C-1460-19, the coarse tungsten powder was plated with nickel in an attempt to ensure good distribution and to provide a coherent diffusion path for the tungsten. Although the ultimate tensile strength and elongation were diminished, the yield strength was significantly higher than that of the standard.

It appears that with these alloys the DPH hardness number is a good indicator of relative value of the yield strength.

VI. SUGGESTIONS FOR FUTURE WORK

From information gathered in this investigation it appears that further study of the W-Ni-Fe alloy system, incorporating into the system some or all of the beneficial variations observed here, would be useful. Some promising variations are

(a) substituting a 1-h hold at 1400°C for the previously used 2-h hold at 1300°C, (b) utilizing fine tungsten powder, (c) vacuum annealing, (d) small additions of rhenium or chromium, and (e) nickel plating the tungsten powders.

An interesting facet for future investigations would be to utilize the electron microprobe to determine the chemistry of the binder phase, and the compositional gradients existing between the tungsten grain-matrix interface and the bulk of the matrix material.

Still another area for investigation would be possible attempts to toughen the tungsten grain as well as to strengthen the matrix.

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100X

C-1520-31

100X

C-1520-34

500X

C-1520-31

500X

C-1520-34

Fig. 11. Microstructure of W-Ni-Fe alloys with rhenium additions (left) and Cr-Ni-Fe alloy additions (right).