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AN EXPERIMENTAL ATTEMPT TO ACHIEVE MICROSTRUCTURE VARIATIONS
THROUGH TEMPERATURE/TIME PERMUTATIONS FOR A NONWROUGHT
POWDER METALLURGY URANIUM-6 NIOBIUM ALLOY

G. M. Alexander-Morrison

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ABSTRACT

The powder metallurgical processing of uranium-6 niobium (U-6Nb) alloy has undergone 3 years of characterization at the Oak Ridge Y-12 Plant. The current structure of the powder metallurgy (P/M) alloy has been described as duplex: a mixture of two or more distinct grain sizes. An experiment was conducted to determine a gamma stabilizing heat treatment (time and temperature combination) to produce a structure consisting of one gamma grain size. Two heat treatments were found to produce an irregular ASTM 4 grain size. Additional P/M U-6Nb alloy material was hot pressed and characterized using the two new heat treatments; however, the resulting microstructures were size ASTM 8. Mechanical properties of the fine-grained P/M material were characterized. Continued evaluation of these results did not determine the cause of the grain refinement.

PREFACE

Powder metallurgical hot-pressing techniques have been successfully used to produce full-density U-6Nb alloy. The most successful P/M procedures convert a hydride alloy powder, using an in situ consolidation/dehydriding technique, into a full-density, two-phase material which has several near-net-shape applications. In the two-phase condition, the P/M material - like the regular wrought material - displays poor corrosion and ductility properties. A gamma stabilizing heat treatment is used to improve the properties of the wrought material. However, when the P/M alloy is heat treated using this gamma stabilizing heat treatment, the resulting structure consists of one phase; but several grain sizes are present. This project was initiated to determine a method of eliminating this "duplex" structure and to achieve an equiaxed structure without the incorporation of deformation to the P/M material.

SUMMARY

To simulate the normal processing of near-net-shape P/M parts, hydrided U-6Nb alloy powder was vacuum hot pressed to produce a full-density, cylindrical as-hot-pressed U-6Nb alloy blank. After a slow cool to room temperature, the P/M blank was removed and sampled. No hot or cold deformation was added in order to complete simulation of a near-net-shape part processing. This blank was sectioned into 20 heat treatment samples. Each of the 20 specimens was tested with a temperature/time combination in an attempt to produce a nonduplex, equiaxed microstructure. Two vacuum heat treatments were found to minimize the duplex characteristic: 750°C, 1.5 h hold; and 850°C, 1.0 h hold.

Two additional P/M blanks were hot pressed and sectioned for characterization of the new heat treatments. Both blanks were prepared in a similar manner; however, the powder for the second blank was rod milled for 30 min prior to screening. The two heat treatments (750°C, 1.5 h hold; and 850°C, 1.0 h hold) and the standard production (800°C, 1 h hold) vacuum heat treatment were tested. The resulting microstructures for all three heat treatments and for both blanks of material were characterized as a uniform nonduplex ASTM 8. The altered heat treatments did affect the mechanical properties, but not because the duplex structure was removed. The properties, yield strength, and reduction-in-area increased with the fine grain size. No improvement was achieved for notched Charpy impact energy. No significant compositional difference was found between these two additional blanks and the initial heat treatment test blank; however, the second blank in the second set of as-hot-pressed blanks contained a higher impurity content. After reviewing all available data, the cause of the grain size refinement was not known.

INTRODUCTION

Several deficiencies exist with the current processing procedures of arc-melted U-6Nb alloy. These problems are primarily chemical inhomogeneity and a very low material utilization. In an attempt to eliminate these problems, powder metallurgy processing techniques are being applied to this alloy. The most successful technique to date is the hydride/dehydride, hot-pressing method. Three years of characterization and processing experimentation have shown that this P/M alloy has uniaxial tensile properties equivalent to standard production alloy. However, several properties are not at this time equitable. Of these less than optimal properties, two (the stress corrosion resistance and the notch toughness) have been selected for improvement.

The literature cites several physical attributes that influence corrosion properties and toughness.^{1 2} For P/M, the most applicable attributes are chemical homogeneity, chemistry, and the microstructure of the material. (Other factors such as surface finish and number of inclusions will not be considered at this time.)

From past investigations, the P/M U-6Nb alloy has been demonstrated to be more homogeneous than the typical production alloy.³ The 3 years of characterization have shown that the P/M alloy has the same basic alloy composition but a higher percentage of impurities. These impurities are primarily oxygen (≈ 100 wppm in P/M versus 30 wppm in regular wrought material) and nitrogen (≈ 60 wppm in P/M versus 30 wppm in typical wrought material). Therefore, this difference in impurity levels could be the cause of the corrosion and toughness problems.

Another significant difference between the production alloy and the P/M alloy is found when comparing structures. The current P/M alloy (Fig. 1) is characterized with a "duplex" grain structure: an inconsistent mixture of at least two distinct grain sizes. The larger grains, ASTM 3-4, usually have more than six sides. These large gamma phase grains are not typical of regular wrought U-6Nb alloy material. Their grain boundaries are jagged and irregular, and the grain boundary curvatures are not consistently convex or concave. The smaller grains, ASTM 7-8, are also irregular, jagged, and contain six or less sides. The smaller grains tend to cluster giving the appearance of subgrains but fail to be absorbed by neighboring large grains during grain growth. To date, the duplex structure has not been definitely linked to the low toughness or poor corrosion properties. But based upon the normal appearance of U-6Nb gamma grains, the grain irregularity is considered a most likely contributor to these detrimental properties.

Four factors are known to affect the grain size of metallic materials: deformation, impurity level, annealing temperature, and annealing time. First consider the relationship between deformation and P/M parts. Generally, P/M parts are fabricated from powders into their final size and shape. Near-net-shape processing is one advantage gained from P/M processing. After pressing and sintering, all subsequent operations are incorporated so that the shape of

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Fig. 1. Typical structure of P/M U-6Nb alloy after a vacuum heat treatment of 800°C, 1 h hold, and water quench (specimen 72579) (100X).

the P/M part is not altered. (Powder forging preforms are an exception to this rule.) Therefore, no additional deformation, cold or hot, can be utilized to refine the grain size.

The second factor, impurity level, will also aid in grain size refinement; however, impurities - especially inclusions - tend to decrease corrosion resistance. Knowing that the corrosion resistance is already inferior to the regular wrought alloy's resistance, no attempt should be made to increase the inclusion content of the P/M alloy. Two remaining factors - annealing temperature and time at temperature - are the only processing variables that can be used to alter the structure of the P/M alloy.

As previously stated, the current structure of the P/M alloy contains both large and small grains. For most wrought metal structures, inferences can be made as to which grains formed from which grains based upon the curvature of the grain boundaries.⁴ However, for the P/M alloy the grain curvatures are inconsistent as if the material may be partially undergoing recrystallization and/or grain growth. If recrystallization is assumed, then the smaller grains - strain free - are formed at the high energy intersections of the large grains. With increasing time, these new grains coalesce to form the large grains. This explanation follows with the knowledge that the P/M structure contains the large grains surrounded by a cluster of small grains. However, if recrystallization is assumed to occur immediately after the phase change occurs, then the small grains are undergoing subgrain coalescence and the larger grains are their products. In either case, the final product should be the larger grains with few of the smaller grains in the structure.

OBJECTIVE OF THIS INVESTIGATION

The current production U-6Nb alloy material displays superior stress-corrosion resistance and toughness properties when compared to the P/M alloy. Of the possible causes of these inferior properties, only one can be experimentally investigated: the duplex microstructure. The chemistry and the homogeneity depend on the processing environment for the powder and should not be considered controllable variables. Therefore, the microstructure of P/M alloy will be investigated as the possible cause of the inferior properties. The first objective of this investigation is to determine how to transform the P/M microstructure into a nonduplex structure composed mostly of ASTM size 4. After determining how this transformation can be achieved, the second objective is to determine if these new features improve the corrosion resistance and/or toughness but have no detrimental effects on other properties.

EXPERIMENTAL PROCEDURE

BLANK PREPARATION

Powder. The preparation of P/M U-6Nb alloy powder has been documented in an experimental procedure.⁵ Massive U-6Nb alloy scrap - from the current alloy

production program - is abrasively and chemically cleaned of major surface contaminants. The cleaned scrap is placed in a stainless steel reactor, and the vessel is evacuated. The hydriding reaction consists of purging the vessel with argon, heating the vessel and contents to $\approx 100^{\circ}\text{C}$, and backfilling the vessel with hydrogen. The heat source is removed, and hydrogen is continuously fed to the vessel. The outlet gas flow is monitored; and when hydrogen is no longer being consumed, the reaction is considered complete. After cooling to room temperature, the vessel is transported to an argon glove box where the powder is screened and stored in a "clean" environment: oxygen, water, and nitrogen levels are monitored and filtered continuously.

P/M Blank. In preparation for the hot-pressing process, a vacuum out-gassed graphite die is cleaned, coated with yttria oxide wash (wherever powder will contact the die), and partially assembled. The die is transported to the argon glove box where the required alloy hydride powder charge is loaded into the die. The die assembly is completed and all die openings sealed with a colloidal graphite solution. The die is transported to a vacuum hot press.

Hot Pressing. The hot-pressing parameters are currently under investigation. For the hot pressings in this project, the maximum temperature and load were 1100°C and 14 MPa (2030 psi). The hot-pressing run cycle is shown in Fig. 2. The initial portion of the cycle is slow in order to allow for the complete dehydriding of the powder. Load is increased during this period to ensure densification during this chemical decomposition. The final stages of the hot-pressing cycle are completed when ram travel ceases (some back expansion of the hot-pressing system may occur). At this point the powder is removed and the contents slow cooled under vacuum to 600°C . At this time, argon is purged through the vessel and the die cooled to room temperature. The as-hot-pressed blank is then removed and ready for further processing.

HEAT TREATMENT STUDY

As previously described, an as-hot-pressed blank of U-6Nb alloy was obtained for the initial heat treatment study. The blank (identified as Blank B10) was hot pressed to full density (17.66 g/cc). A sampling plan was designed (Fig. 3) to maximize the number of samples in the blank. Twenty heat treatment samples (1.27 cm x 1.27 cm x 2.54 cm), two tensile samples, and a chemical sample were removed from the blank. The tensile samples were vacuum heat treated using the regular production heat treatment procedure: 800°C , 1 h hold, and water quench.

Each vacuum heat treatment sample was placed in a tantalum wire holding fixture and heat treated according to the experimental procedure. After holding at temperature for the desired time, the samples were water quenched. Each sample was cut into two pieces and the center cross section of the sample mounted for metallographic inspection. ASTM grain size measurements were measured for each sample. Square grid quantitative analysis was incorporated to determine the approximate percentage of each individual grain size.

Fig. 2. Hot-pressing run cycle for producing U-6Nb alloy material from a hydrided alloy powder charge.

Blank

Diameter: 4.70 in.

Thickness: 0.97 in.

Run Order

Time Temp. °C.	0 m.	10 m.	30 m.	60 m.	90 m.
700	10	11	7	4	8
750	12	13	9	5	16
800	14	15	3	1	2
850	17	18	6	20	19

Fig. 3. Sampling plan for heat treatment study.

At the conclusion of the vacuum heat treatment tests, two temperature/time combinations were selected for further testing: 750°C for 1.5 h and 850°C for 1.0 h. The standard 800°C for 1 h hold vacuum heat treatment was also selected for control purposes.

The second objective of this heat treatment study was to determine if the stress-corrosion resistance or toughness properties were improved by the heat treatment change. Prior to the corrosion testing, the mechanical properties of the two treatments were evaluated. Two P/M blanks were hot pressed and sectioned for characterization of the new heat treatments. The first blank, C8, was made from powder screened through a 170-mesh sieve (60% yield) immediately after powder production. The second blank, C9, was made from powder that was rod milled prior to screening.* The rod milling produced a powder (~100% yield) with a particle size distribution equivalent to powder that had been screened through a 170-mesh screen.

Each blank, in the as-hot-pressed condition, was sectioned into three pieces (Fig. 4). Each section was heat treated using one of the specified time/temperature combinations and water quenched. Two (T6) tensile specimens (2.54 cm gage, 0.65 cm diam) and two notched Charpy specimens (only taken from two of the three sections - I1 specimens: 5.72 cm length, 1.27 cm width, 1.27 cm depth) were removed from each section along with metallography, grain size, density, and chemical analysis samples.

RESULTS

The results of the chemical analysis and tensile tests, for Blank B10, are shown in Tables 1 and 2.

Table 1. Chemical analysis of heat treatment Blank B10

Condition of material	Uranium (g/g)	Niobium (g/g)	Carbon (wppm)	Oxygen (wppm)	Nitrogen (wppm)
As pressed	0.9362	0.0599	80	103	41
Heat treated				105	45

The results of the heat treatment study are tabulated in Table 3. Grain size estimates were mixtures of ASTM 2-10. Based upon these measurements, two heat treatments were selected: 750°C for 1.5 h and 850°C for 1.0. The associated microstructures for the heat treatments are shown in Figs. 5a-c. The effect of the temperature and time at temperature on the structure is demonstrated by the series of photographs in Figs. 6 and 7.

*Rod milling was included here from a related experiment looking at fine powder yield improvements.

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Sampling Plan for Blanks C8 and C9

Fig. 4. Sampling plan for mechanical property tests for Blanks C8 and C9.

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(a) 750°C, 1.5 h (100X).

MS-83-1556-4

(b) 850°C, 1.0 h (100X).

MS-83-1556-1

(c) (control) 800°C, 1.0 h (100X).

Fig. 5. Best resulting structures from the vacuum heat treatment study.

MS-83-1125-2

MS-83-1161-4

(a) 700°C, "no defined grain" (100X).

(b) 750°C, ASTM 8/4 (100X).

MS-83-1074-3

(c) 800°C, ASTM 8/3 (100X).

Fig. 6. Effect of temperature on P/M U-6Nb alloy with a constant heat time of 30 min (vacuum and water quench for all).

MS-83-1297-12

(a) No hold (100X).

MS-83-1297-13

(b) 10 min (100X).

MS-83-1161-3

(c) 30 min (100X).

MS-83-1083-2

(d) 60 min (100X).

MS-83-1632-2

(e) 90 min (100X).

Fig. 7. Effect of time on P/M U-6Nb alloy with a constant temperature 750°C (vacuum and water quench for all).

Table 2. Mechanical properties of heat treatment test
Blank B10 with 800°C, 1 h hold, vacuum heat treatment

Sample	Tensile strength (ksi)	Yield strength (ksi)	Elongation (%)	Reduction in area (%)	Young's modulus (10 ³ ksi)
1	119.5	20.7	28.5	31.9	10.0
2	120.1	20.0	25.0	29.3	10.0

Table 3. Grain size measurements
for variable time/temperature heat treatments

ASTM grain size/percentage

Time (min) --> Temperature (°C)	0	10	30	60	90
700	not defined	not defined	10-7/ 100%	8/ 80% 5/ 20%	8/ 60% 4/ 40%
750	10/100%	9/ 75% 5/ 25%	8/ 50% 4/ 50%	8/ 25% 4/ 75%	8/ 12% 3-4/ 88%
800	9/ 80% 6/ 20%	8/ 70% 6/ 30%	8/ 30% 4/ 70%	8/ 20% 4/ 80%	8/ 20% 3/ 80%
850	8/ 80% 3/ 20%	8/ 70% 3/ 30%	8/ 50% 3/ 50%	7/ 10% 2/ 90%	7/ 10% 2/ 90%

The characterization results for physical and mechanical properties of both Blank C8 and C9 are tabulated in Tables 4, 5, and 6. The microstructures of each section, resulting from the different heat treatments, are shown in Figs. 8 and 9.

Table 4. Physical property results
for Blanks C8 and C9 at specified heat treatments

Blank	Heat treatment	Density (g/cc)	Grain size	U (g/g)	Nb (g/g)	O (wppm)	N (wppm)
C8	750°C, 1.5 h	17.447	ASTM8	0.9423	0.0585	104	46
C8	800°C, 1.0 h	17.444	ASTM7			96	44
C8	850°C, 1.0 h	17.453	ASTM7			108	45
C9	750°C, 1.5 h	17.459	ASTM8.5	0.9402	0.0575	102	72
C9	800°C, 1.0 h	17.461	ASTM8			121	73
C9	850°C, 1.0 h	17.445	ASTM8			109	70

Table 5. Notched Charpy impact test
results for Blanks C8 and C9
at the two test heat treatments

Blank	Heat treatment	Impact energy (ft-lb)	
		Specimen 1	Specimen 2
C8	750°C 1.5h	26.5	26.0
	850°C 1.0h	23.5	22.0
C9	750°C 1.5h	20.0	19.0
	850°C 1.0h	19.0	19.0

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(a) 750°C, 1.5 h (100X).

MS-84-1138-6

(b) 850°C, 1.0 h (100X).

MS-84-1177-3

(c) 800°C, 10. h (100X).

Fig. 8. Microstructure results for Blank C8.

MS-84-1138-4

(a) 750°C, 1.5 h (100X).

MS-84-1177-1

(b) 850°C, 1.0 h (100X).

MS-84-1177-5

(c) 800°C, 1.0 h (100X).

Fig. 9. Structure of Blank C9 after test heat treatments.

Table 6. Uniaxial tensile property results
for Blanks C8 and C9 at specified heat treatments

Heat treatment	Sample	Tensile strength (ksi)	Yield strength (ksi)	Elongation (%)	Reduction in area (%)	Elastic modulus (10^3 ksi)
<u>Blank C8</u>						
750°C 1.5 h	1	127.1	27.3	28.0	37.8	10.1
	2	123.8	25.1	29.0	39.4	10.1
800°C 1.0 h	1	123.3	24.2	28.5	38.6	10.7
	2	124.8	25.3	30.0	43.0	10.6
850°C 1.0 h	1	123.8	26.1	28.0	42.0	10.0
	2	123.5	25.3	27.0	34.6	10.0
<u>Blank C9</u>						
750°C 1.5 h	1	124.8	26.3	25.0	22.1	10.1
	2	124.6	31.1	24.0	27.7	7.5
800°C 1.0 h	1	129.3	26.3	27.0	30.1	10.1
	2	128.3	26.0	27.0	30.6	10.6
850°C 1.0 h	1	124.3	26.5	27.0	33.3	10.0
	2	124.1	23.8	27.0	34.6	10.0

DISCUSSION

The results of the preliminary temperature/time tests demonstrated the sensitivity of the P/M alloy to heat treatment variations. The resulting microstructures were coarse and still contained the duplex characteristic (when viewed at 100X magnification). The temperature/time variations did successfully determine that an equiaxed structure can be produced from the P/M material when the "blank" is constant. The difficulty with this experiment only occurred when the as-hot-pressed material blank varied. The heat treatments produced two radically different microstructures based on whether the material was Blank B10 or C8 or C9.

Overall, the refinement in grain size found with Blanks C8 and C9 is very surprising. The structures were viewed at a depth very similar to the location in the heat treat samples (roughly 0.25 to 0.50 in. below the surface). To test that the structure did not vary from one edge of a hot-pressed blank to the middle, one-half of a broken tensile sample, for C8 - 850°C, was mounted for metallography. The structure was fine grained, and no gradient in size was apparent from the middle of the specimen to its end. Several samples from the heat treatment tests were also reexamined to ensure that the grain sizes were mostly ASTM size 3-4. The original grain size measurements were reconfirmed. The material mass in the furnace was then suspected. For review, the two tensiles from Blank B10 were sectioned and compared to the tensile in Blank C8. The two B10 tensiles were machined from a heat treated piece of material of 4 x 0.5 x 1.0 in. The structure of the tensile - from end to end - was similar to Fig. 5c rather than to Fig 9c (fine grained). Therefore, the size of the material in the furnace was eliminated as the cause of the fine grain material. All information on the three blanks of material was reviewed. The only difference in their processing was the date - in time - that the blanks were prepared.

The second part of the test, the mechanical property testing, showed that the heat treatment change had a large effect on properties. This effect is seen dramatically in the yield strength and reduction-in-area increase for the second two blanks. The only explanation for the increase is that the fine grained material has more grain boundary area to hinder deformation but at the same time allowed for more grain rotation - biaxial ductility. Blank B10 - characterized with the standard 800°C/1.0 h vacuum heat treatment - displayed results similar to past P/M tensile results: a yield of 20 ksi and reduction-in-area of 30%. Blanks C8 and C9 had values of 24.8 ksi and 26.2 ksi for yield strength and 40.8% and 30.3% for reduction-in-area. The notched Charpy results for C8/C9 were unexpected. The fine grained material should have displayed higher impact energies than the typical P/M U-6Nb alloy. The premise that the notch sensitivity of a material will decrease with increased grain boundary area and decreased grain size does not appear applicable in this situation.

As a secondary experiment, the yield improvement by rod milling demonstrated that the milling might have been detrimental to the chemical purity of the powder. Before such a statement is made, a similar experiment will be repeated.

CONCLUSIONS

The objective of this study was to produce P/M material with a grain size that was considered uniform, i.e., contained one grain size. This objective was achieved, but the reason for the achievement will require further study. This series of experiments did allow for the study of properties of two different grain size P/M materials. Originally, the belief was that the notched impact strength would be improved with a nonduplex P/M structure. This was disproven and there is now evidence that the reduction-in-area can be improved if the grain size is refined. As to whether the corrosion properties would improve with the nonduplex structure, additional material - with the fine grain size - will have to be made and tested. Further work will concentrate on producing the fine grain material and testing its corrosion properties.

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