Development Study of Cartridge/Crucible Tube Materials

A Final Report to NASA-MSFC

Contract No. NAS8-97213

Submitted by:

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ABSTRACT

The limitations of traditional alloys and the desire for improved performance for components is driving the increased utilization of refractory metals in the space industry. From advanced propulsion systems to high temperature furnace components for microgravity processing, refractory metals are being used for their high melting temperatures and inherent chemical stability. Techniques have been developed to produce near net shape refractory metal components utilizing vacuum plasma spraying. Material utilization is very high, and laborious machining can be avoided. As-spray formed components have been tested and found to perform adequately. However, increased mechanical and thermal properties are needed. To improve these properties, post processing thermal treatments such as hydrogen sintering and vacuum annealing have been performed. Components formed from alloys of tungsten, rhenium, tantalum, niobium and molybdenum are discussed and a metallurgical analyses detailing the results are presented. A qualitative comparison of mechanical properties is also included.

INTRODUCTION

Traditional nickel based superalloys have been used extensively in the aerospace and space industries in high temperature applications. Advancements in component fabrication such as directional solidification of single crystal turbine blades and ceramic based thermal barrier coatings have only slightly increased the operating temperatures of these alloys.[1-2] If significant advancements in high temperature operations such as propulsion systems and high temperature furnace components are to be made, new and improved materials and processing techniques are necessary. In general, the refractory metals and their alloys offer the desired high melting temperatures and an inherent chemical stability, in nonoxidizing environments, needed for these applications. However, the difficulty of forming these materials into complex shapes has limited their application in the past.[3]

Recently, vacuum plasma spray (VPS) forming has been demonstrated as a viable fabrication tool for refractory metal components.[4-6] This technique involves spraying material onto a mandrel of the desired

shape and subsequently removing the mandrel. The plasma is formed by passing gases such as argon and/or hydrogen through an electric arc struck between the an anode and a cathode within a gun. The gas passing through the arc is ionized and results in temperatures on the order of 16,650°C (30,000°F). Powder, which is injected into the hot plasma by an argon carrier gas, is melted and accelerated toward the surface of a part at speeds up to Mach 2-3. Deposition rates can be as high as 9 kg/hr (20 lb/hr). A schematic of this process is shown in Figure 1. Spraying is performed in a large vacuum chamber which has been evacuated and backfilled with a partial pressure of argon to prevent oxidation of oxygen sensitive materials. A primary advantage of VPS forming over other powder metallurgy techniques is that near-net-shape spray forming of components significantly simplifies and promises to reduce the cost of fabricating due to the high material utilization and reduction in laborious machining.

During this investigation, VPS forming was used to fabricate material for evaluation as high temperature containment cartridges for materials science research in microgravity. Possible furnaces in which the cartridge materials will be used include the Crystal Growth Furnace (CGF) and the High Gradient Furnace with Quench (HGFQ) which operate at a maximum temperature of 1400 -1600°C (2552-2912°F). These furnaces are directional solidification furnaces which are used in orbit to determine the effects microgravity has on the solidification of different materials. The requirements of a cartridge material are that it have a high melting temperature and resist attack from the material being processed.[7] High melting temperature and good chemical stability are general characteristics of the refractory metals. Previous research in this area has shown that cartridges can be fabricated from several refractory metals. However, compression testing of the as-sprayed components resulted in relatively low strengths with little ductility. The focus of the current investigation was to fabricate cartridges from several refractory metals by VPS forming and then to perform post-spray thermal treatments on these materials to improve ductility. The effect of the thermal treatments on the microstructures was also noted.

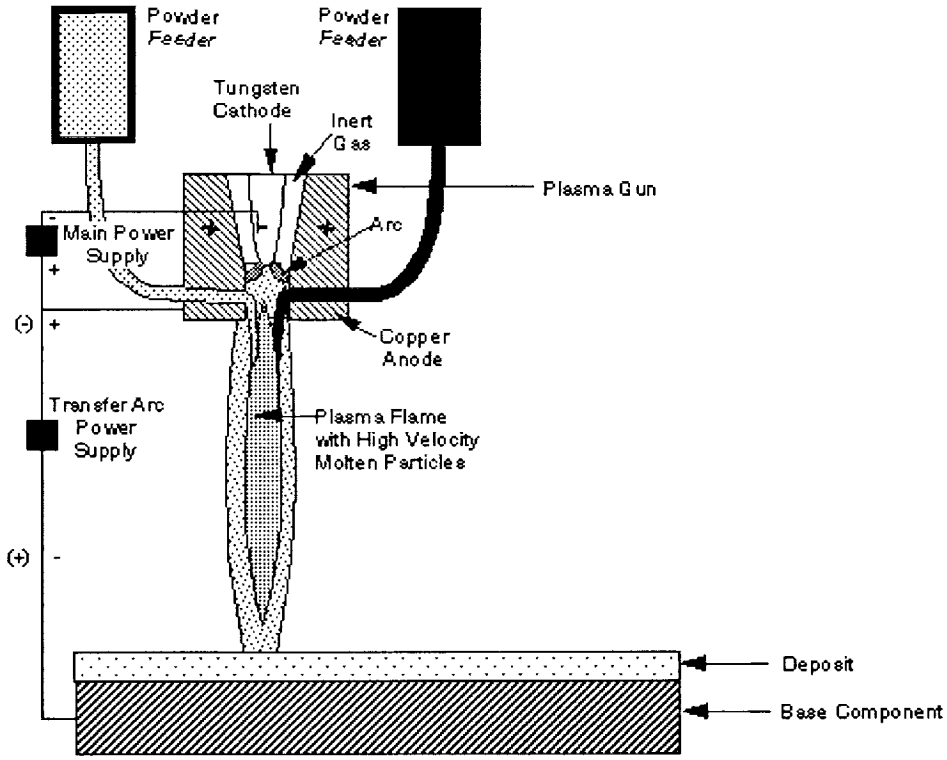


Figure 1 - Schematic of VPS forming process.

EXPERIMENTAL PROCEDURE

The five refractory metal alloys used during this investigation included, tungsten-3.5wt% nickel-1.0wt% iron, molybdenum-40wt% rhenium, tantalum-10wt% tungsten, tungsten-25wt% rhenium and niobium-1.0wt% zirconium. Only the Nb-1Zr material was a truly alloyed powder, all of the other compositions were comprised of elemental powders. Compatibility and compression specimens were made for each material. The compatibility specimens were small open-ended tubes which measured 8 mm (0.313") long by 10 mm (0.375") internal diameter by 0.9 to 1.27 mm (0.035 to 0.050") wall thickness. The compression specimens were also small open-ended tubes which measured 2.54 cm (1") long by 2.54 (1") internal diameter by 0.8 to 1.27 mm (0.030 to 0.050") wall thickness. Table 1 lists the five materials and the minimum number of compatibility and compression specimens fabricated for each alloy.

Table 1 - List of Compatibility and Compression Specimens Fabricated

Alloy	No. of Compatibility Specimens	No. of Compression Specimens
W-3.5Ni-1Fe	32	3[a]
Mo-41Re	40	3
Nb-1Zr	32	3
Ta-10W	44	3
W-25Re	40	3[b]

a - three (3) compression specimens were required by the contract, however a total of nine (9) specimens were delivered: six (6) heat treated and three (3) as-sprayed.
b - a total of six (6) specimens were delivered: three (3) heat treated and three (3) as-sprayed.

Vacuum Plasma Spray Forming

The initial step in the fabrication process was the VPS forming of the materials onto preformed graphite mandrels. The mandrels used during spraying were 30.5 cm (12") long with 2.54 cm and 10 mm diameters for the compression and compatibility samples, respectively. Prior to spraying, the vacuum chamber was evacuated and backfilled with a partial pressure of argon. During spraying, powder was delivered to the gun by an argon carrier gas and an argon/hydrogen plasma was used to melt the powder and project it toward the mandrel. The size range of the powders sprayed during this investigation ranged between -45 to +10 μm. The mandrel was rotated during spraying to allow the formation of the tube. Approximately, 25.4 cm (10") of each mandrel was coated resulting in an open-ended 25.4 cm (10") long tube by 0.7 - 1.2 mm (0.03-0.05") wall thickness. These sample configurations were sufficient for this investigation. (For reference, a typical CGF cartridge is 58.4 cm (23") long x 2.54 cm (1") OD x 0.7 mm (0.03") wall thickness with one end closed.) After spraying, the as-sprayed cylinder was removed from the graphite mandrel.

Thermal Treatments

The thermal treatment for each material was selected based on current heat treatments for sintering and annealing conventional powder metallurgy components. Each tube was packed with high purity alumina sand to prevent slumping of the thin walled tubes during heating. Hydrogen was used during the heat treating of three of the alloys (Mo-40Re, W-25Re and W-3.5Ni-1.0Fe) to aid in densification and the reduction of oxides. Both a liquid phase sinter (LPS) and a solid state sinter (SSS) were used on the W-Ni-Fe alloy. Hydrogen was not used when heat treating the Ta-10W and the Nb-1Zr alloys due to the formation of brittle hydrides. These alloys were only given a vacuum anneal (VA). Table 2 lists the heat treatment procedure for each alloy. After heat treating, the samples were cut to size using an abrasive cut-off saw. The compatibility samples were then delivered for testing at the University of Alabama at Birmingham. The results of the compatibility study will be discussed in a different report.

Table 2 - Heat Treatment Procedures

Alloy	Heat Treatment Description
W-Ni-Fe	SSS: Cold wall batch furnace with dry hydrogen; cycle 10°C/min to 1000°C, hold 30 min (deoxidation), 5°C/min to 1430°C, hold 200 min. VA: 10^{-6} torr at 900-1100°C for 240 min, cool to room temp. in vacuum.
W-Ni-Fe	LPS: Stoker furnace with dry H_2 with stoke rate of 6.4 mm/min at 1480°C. VA: 10^{-6} torr at 900-1100°C for 240 min, cool to room temp. in vacuum.
Mo-40Re	H_2 Sinter: Industrial H_2 furnace to 1730°C and hold for 5-10 hrs.
Ta-10W	VA: 10^{-6} torr, ramp up 10°C/min to 1000°C and hold for 30 min, 5°C/min to 1500°C hold for 24 hrs, cool to room temperature in vacuum.
Nb-1Zr	VA: 10^{-6} torr, ramp up 10°C/min to 1000°C and hold for 30 min, 5°C/min to 1500°C hold for 24 hrs, cool to room temperature in vacuum.
W-25Re	H_2 Sinter: Industrial H_2 furnace to 1730°C and hold for 24 hrs.

Testing

Standard metallurgical polishing techniques were used to prepare samples in the as-sprayed and heat treated conditions for each material. These samples were then examined in the as-polished and etched conditions using an optical microscope. Quantitative microscopy was used to determine the density of the samples. Helium leak tests were performed on the as-sprayed and heat treated samples to determine if any interconnected porosity was open to the surface. The helium leak rate specification for materials containment cartridges is no greater than $1x10^{-6}$ cm^3 of He/sec. Also, a limited number of compression tests were performed on the heat treated materials to determine any improvements in mechanical properties. Samples were cut from the heat treated tubes [three 2.5 cm (1") long segments x 2.5 cm (1") ID x 0.7-1.2 mm (0.030-0.050") wall thickness] to get a qualitative comparison of each materials robustness or toughness. The segments were laid on their side and compressed until failure at a crosshead speed of 0.025 cm/min (0.010"/min). Testing was conducted at room temperature.

RESULTS AND DISCUSSION

Table 3 shows the results of compression testing. For comparison, the values of W-Ni and Mo-40Re in the as-sprayed condition are shown from a previous investigation.[5] The maximum loads for the LPS and SSS W-Ni-Fe samples were 284 kg (627 lb) and 71 kg (155.7 lb), respectively. These values were 1593% and 320% of an increase over the maximum load for the as-sprayed W-Ni, 17 kg (37.04 lb). The compression tests of the LPS specimens resulted in the largest average amount of displacement of all the samples tested, 17.8 mm (0.7"). In contrast, the H_2 sintered Mo-40Re maximum load was 54 kg (118.4 lb) which was approximately a 2% increase over the as-sprayed maximum load of 53 kg (115.9 lb). Also, the amount of displacement was greatly reduced for the heat treated Mo-40Re material, 1.7 mm (0.068"), as compared to 8.9 mm (0.349") for the as-sprayed material. The maximum load for the vacuum annealed samples were 100 kg (221.4 lb) for Nb-1Zr and 58 kg (127.7 lb) for Ta-10W with displacements of 7.8 mm (0.306") and 0.7 mm (0.027"), respectively. W-25Re samples have been delivered to NASA for compression testing.

Table 3 - Results of Compression Tests

Description	Maximum Load, kgf (lbf)	Maximum Displacement, mm (in)
As-sprayed W-Ni[1]	17 (37.04)	1.4 (0.056)
As-sprayed Mo-40Re[1]	53 (115.9)	8.9 (0.349)
LPS W-Ni-Fe	284 (627.0)	17.8 (0.700)
SSS W-Ni-Fe	71 (155.7)	2.5 (0.097)
H_2 Sintered Mo-40Re	54 (118.4)	1.7 (0.068)
VA Nb-1Zr	100 (221.4)	7.8 (0.306)
VA Ta-10W	58 (127.7)	0.7 (0.027)

[1] - values from previous study

Leak testing results are shown in Table 4. Note that the Ta-10W and the Nb-1Zr both had acceptable helium leak rates before post spray heat treating. However, the W alloys and the Mo-40Re alloy tubes needed the post spray heat treatment to meet the required $1x10^{-6}$ cm^3 of He/sec specification.

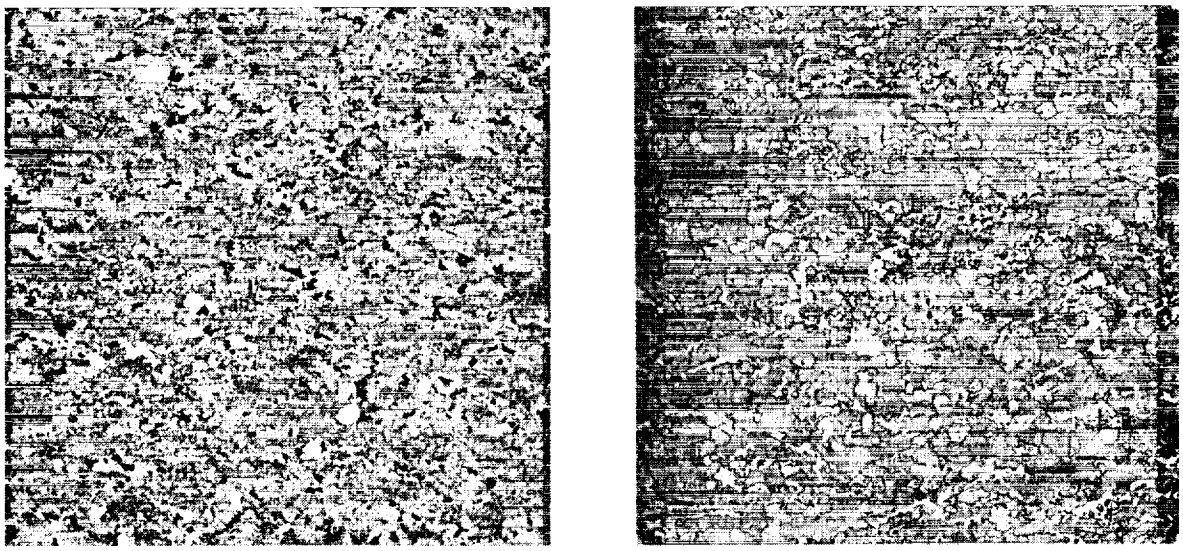
Table 4 - Helium Leak Testing Results

Alloy	Leak Rate (He cm^3/sec) As-sprayed	Leak Rate (He cm^3/sec) Heat Treated
W-3.5Ni-1Fe	10^{-5}	10^{-8} (LPS & SSS)
Mo-40Re*	10^{-4} - 10^{-5}	$<10^{-6}$ - 10^{-7}
Ta-10W	10^{-8}	10^{-8}
Nb-1Zr	$<10^{-7}$	10^{-8}

* - the wall thickness of these tubes were the thinnest of the samples tested which may account for the higher rate.

The microstructures of the materials are shown in Figures 2 - 7. Figure 2a shows the microstructure of the W-3.5Ni-1.0Fe alloy in the as-sprayed condition after etching. Quantitative microscopy measurements of the sample revealed the density to be approximately 91%. Since the W-Ni-Fe alloy was being developed for deposition on a previously plasma sprayed ceramic layer or a preformed ceramic ampoule, a lower set of plasma spray parameters was used during spraying so as not to damage the underlying ceramic. This resulted in the unusually high porosity value for the as-sprayed material (VPS deposits are typically 96 to near 100% dense). Note the as-sprayed microstructure was comprised of a two phase structure. The light gray areas were the Ni-Fe rich binder phase and the larger dark gray area was the W rich phase. Also, there were some large unmelted particles contained in the deposit due to overspray entrapment. The Ni-Fe rich phase was grouped in relatively large islands, but these islands were uniformly dispersed throughout the deposit. With the binder phase mostly contained in these islands, the number of tungsten to tungsten contacts was increased. These tungsten to tungsten contacts greatly reduce the mechanical properties of these alloys in this condition.

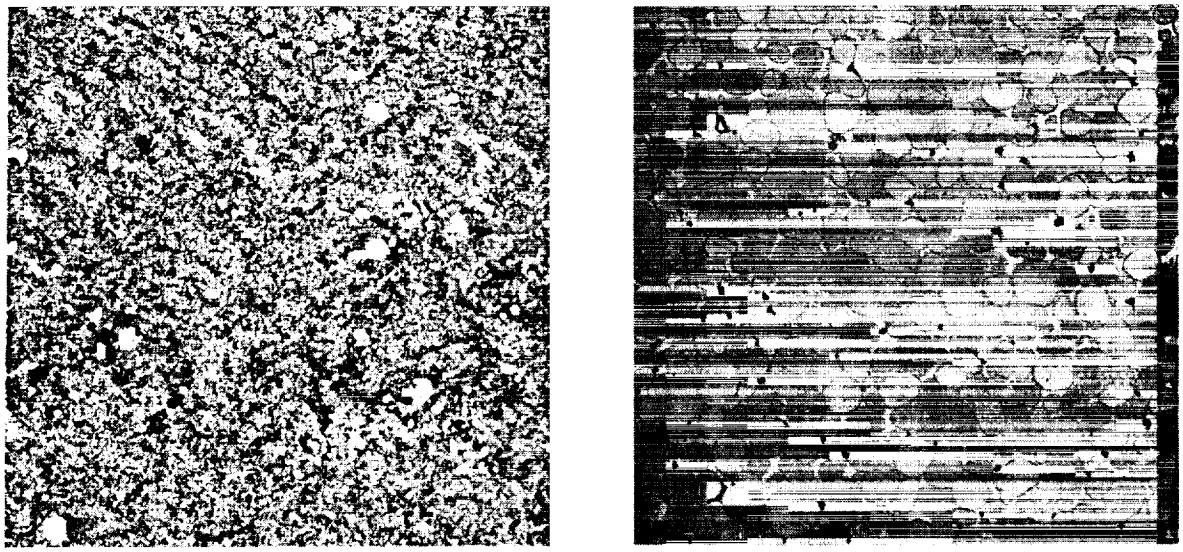
Figure 2b shows the sample after the SSS heat treatment. There was a significant increase in the density of the SSS sample as compared to the as-sprayed condition. Analysis of the SSS W-Ni-Fe showed the density to be >99%. Note that the sample had partially recrystallized, but there were still a significant number of tungsten to tungsten contacts. Also, the tungsten grains were very sharp and angular.



A B

Figure 2 - W-Ni-Fe alloy in the as-sprayed (A) and the solid state sintered (b) conditions; 100x.

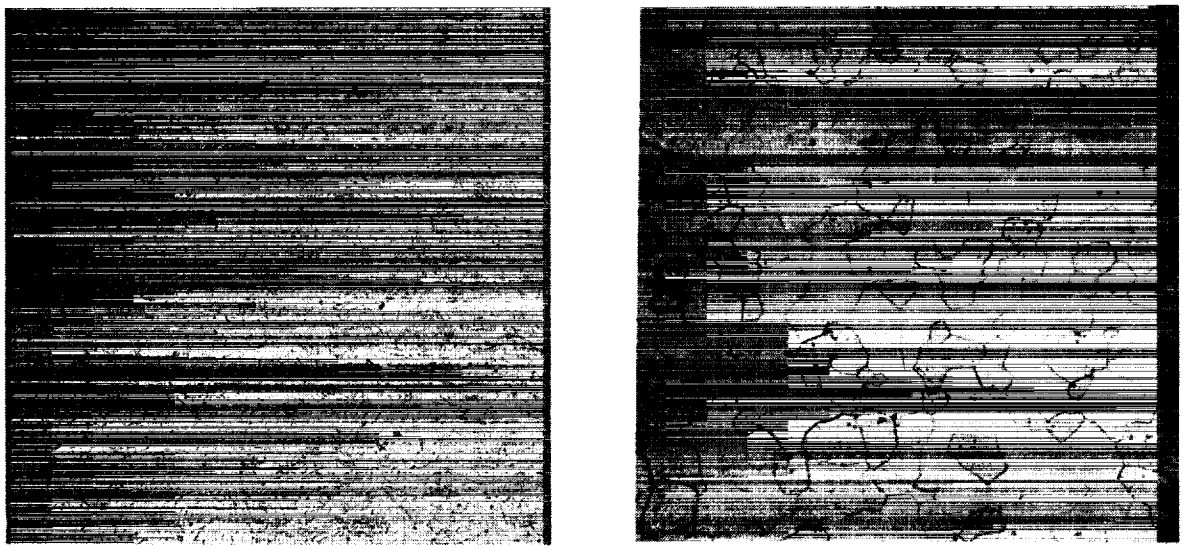
Figure 3b shows the microstructure of the LPS W-Ni-Fe sample. For comparison, the as-sprayed condition is shown in Figure 3a and is essentially the same as Figure 2a. Note the LPS structure consists primarily of large, circular W-rich grains with the Ni-Fe rich phase located between these grains. The density of the sample was increased from 91% to >97% dense. The porosity was contained in the Ni-Fe rich binder phase and is most likely due to void coalescence and binder starvation. The easiest way to alleviate this problem is to add more binder, i.e, increase nickel and iron content. However, the He leak tests showed that the tubes were leak tight, thus the pores must be isolated and not interconnected. As a result of the compression tests data and analysis of the microstructures, it can be concluded that the ductility of the W-Ni-Fe alloy is more dependent on the elimination of tungsten to tungsten contacts than the presence of isolated porosity.



A B

Figure 3 - W-Ni-Fe alloy in the as-sprayed (A) and the liquid phase sintered (B) conditions; 100x.

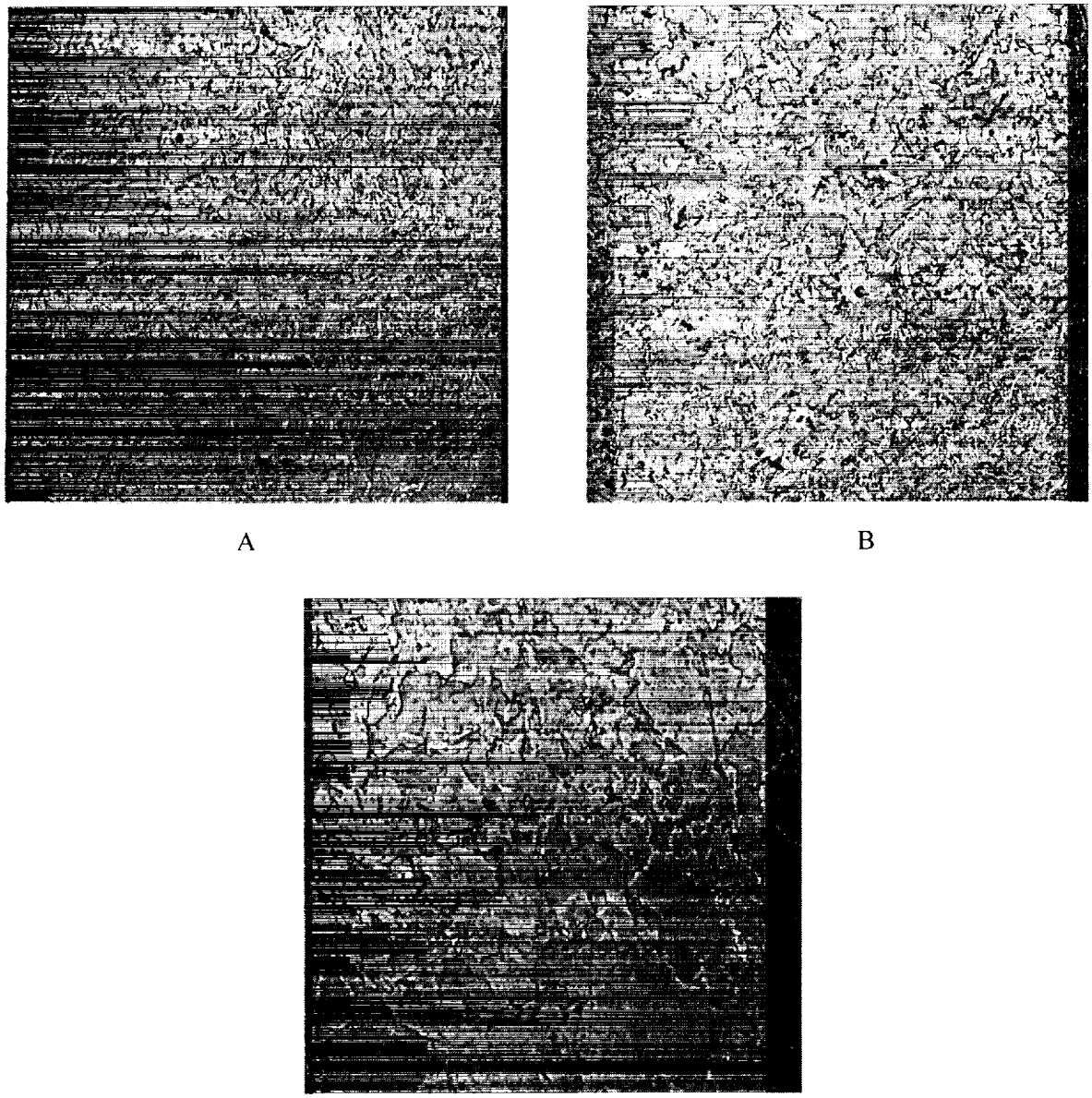
An example of the microstructure of the Mo-40Re sample is shown in Figure 4. Figures 4a and 4b show the Mo-40Re in the as-sprayed and etch conditions. Note the microstructure was comprised of a two phase structure consisting of a Mo-rich matrix and uniformly dispersed Re-rich splats. The density as determined by image analysis was >99%. The microstructure after H_2 sintering at 1730°C (3146°F) is shown in Figure 4c. The as-sprayed microstructure was completely replaced with a recrystallized, homogeneously alloyed microstructure. This microstructure should have resulted in increased displacement measurements during the compression tests when compared to the as-sprayed microstructure. However, this was not the case as stated previously. One explanation for these results are that the Mo-40Re became embrittled during the H_2 sintering process due to the formation of the intermetallic σ phase (68 to 82 wt%Re).



A B

Figure 4 - Mo-40Re in the as-sprayed (A) and the H_2 sintered (B) conditions; 200x.

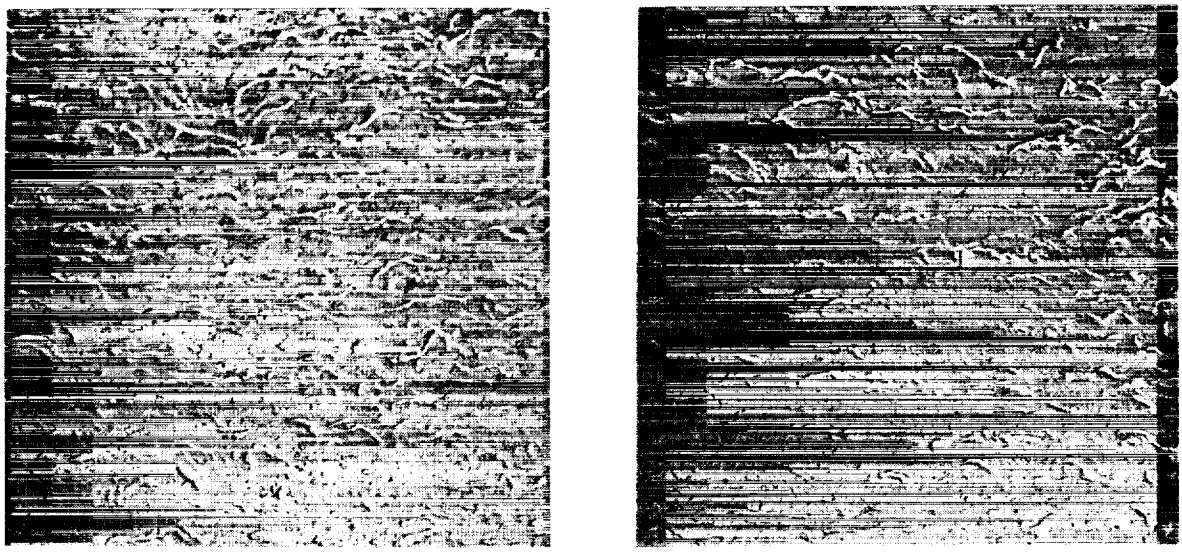
Figure 5a shows the microstructure of the Nb-1Zr alloy in the as-sprayed, etched condition. In contrast to the previously discussed samples, the Nb-1Zr sample has a recrystallized microstructure in the as-sprayed condition. In addition to the lower melting temperature of the material, the prealloyed powder aided in the formation of this microstructure because all the heat input during spraying could go to removing the as-sprayed grain boundaries as opposed to diffusion of elemental powders. Figures 5b and 5c show the microstructure of the alloy after vacuum annealing at 1316°C (2400°F) and 1500°C (2732°F), respectively, for 24 hours. Note there was little change in the microstructure from the as sprayed sample to the sample heat treated for 24 hours at 1316°C. In contrast, grain growth has began for the sample vacuum annealed at 1500°C, which should aid in ductility. The ductility of this alloy is evident by the large amount of displacement during the compression tests shown in Table 2.



A B C

Figure 5 - Nb-1Zr in the as-sprayed (A), 1316°C vacuum anneal (B) and 1500°C vacuum annealed (C) conditions; 200x.

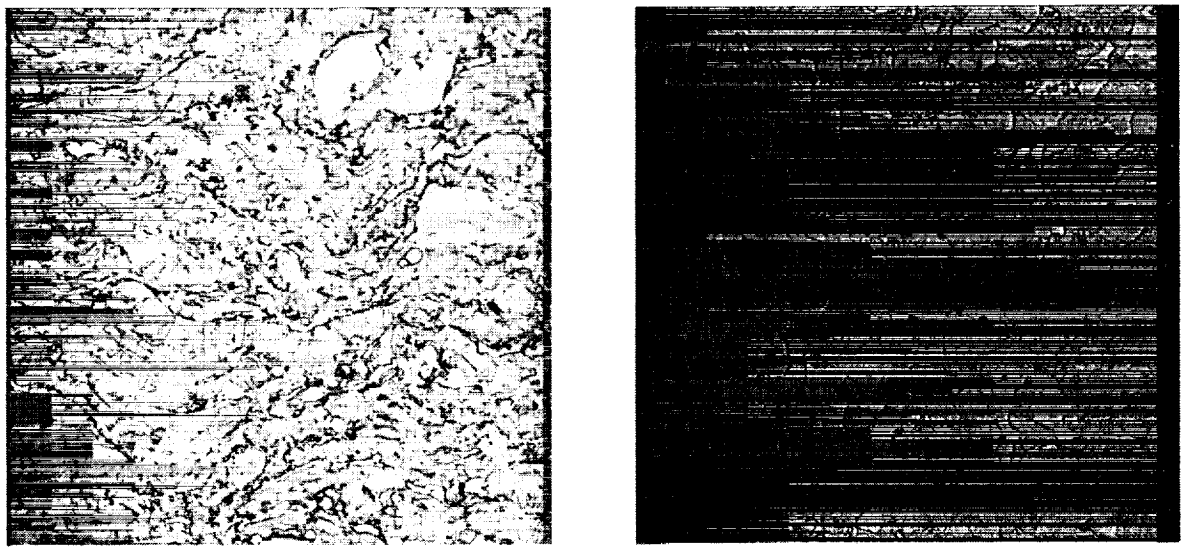
Figure 6a shows the microstructure of the Ta-10W sample in the as-sprayed, etched condition. This figure shows a two phase structure comprised primarily of a Ta matrix with uniformly dispersed W splats. Figure 6b shows the microstructure after vacuum annealing at 1500°C (2732°F). Again, the two phase structure comprised of a Ta matrix with the W splats was still present. Thus, the heat treatment hold time and temperature were insufficient to result in any noticeable diffusion between the two elemental powders.



A B

Figure 6 - Ta-10W in the as-sprayed (A) and vacuum annealed (B) conditions; 200x.

Figure 7a shows the W-25Re alloy in the as-sprayed, etched condition. The photomicrograph shows the as-sprayed material was comprised of a partially recrystallized tungsten matrix with islands of rhenium splats. Similar to the other elementally blended powders used in this study, no alloying occurred in



A B

Figure 7 - W-25Re in the as-sprayed (A) and the H_2 sintered (B) conditions; 400x.

the as-sprayed deposit. Figure 7b shows the material after heat treating in hydrogen at 1730°C for 24 hours. Note the previously as-sprayed microstructure was completely replaced with a recrystallized, homogeneously alloyed microstructure. Compression tests will be performed on this material to determine the effect of the heat treated microstructure on the materials mechanical properties.

CONCLUSIONS

During this investigation, the parameters and techniques for fabricating spray formed tubes from several refractory metal alloys were developed. Post spray thermal treatments were then performed to determine the effect on the microstructure and mechanical properties of the materials investigated. Changes in the microstructure and the mechanical properties were related. The following list is a summary of these results:

1. The SSS and LPS heat treatments significantly improved the toughness and ductility of the W-Ni-Fe alloys and resulted in acceptable leak rates ($1x10^{-8}$ He cc/sec).
2. Heat treating the Mo-40Re alloy reduced the amount of interconnected porosity to acceptable levels as evident by the leak test results, but decreased the ductility. Efforts to alleviate this loss of ductility are currently being investigated.
3. The Nb-1Zr alloy in the as-sprayed condition had an acceptable leak rate and recrystallized microstructure.
4. The 1500°C (2732°F) heat treatment was insufficient for alloying of the two constituents in the Ta-10W samples.
5. The 1730°C hydrogen sinter of the as-sprayed W-25Re deposits resulted in a recrystallized, homogeneously alloy microstructure.

This work has shown that vacuum plasma spray forming, in conjunction with post spray heat treating, is a viable method for fabricating new and improved materials for high temperature furnace cartridges.

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