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MASTER

CVD TUNGSTEN AND TUNGSTEN-RHENIUM ALLOYS
FOR STRUCTURAL APPLICATIONS
PART III. RECENT DEVELOPMENTS

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ABSTRACT

Results obtained since the Gatlinburg Conference on a continuing program to develop CVD tungsten and tungsten-rhenium alloy structures with mechanical properties comparable to those of wrought products are presented. New data relating the effects of all the plating parameters on the deposition rate, deposit composition, topography, microstructure, and mechanical properties, together with effects of various heat treatments on the microstructure and properties, are reviewed. New developments in CVD equipment and techniques are also included.

INTRODUCTION

Preliminary results of a program aimed at producing free-standing shapes of tungsten and W-Re alloys by hydrogen reduction of the metal fluoride(s) were reported at the Gatlinburg Conference.^{1,2} Since that time, a scaled-up modification of the original equipment was developed and used to demonstrate the feasibility of producing CVD alloy tubing for the Space Power Reactor program.³ Experiments with the short-tube apparatus used in the earlier work^{1,2} were continued as part of that program. Emphasis was placed on optimizing the mechanical properties of CVD tubing containing about 25% rhenium. Other compositions were also produced, however; and a wide range of plating conditions were investigated in an effort to understand the effects of all the parameters on composition, microstructure, and mechanical properties.

*Work performed under the auspices of the U.S. Atomic Energy Commission.

This paper will summarize results that have been obtained with the short-tube apparatus since the Gatlinburg Conference. The long-tube fabrication equipment will be discussed only briefly, since a detailed report of that work will be found elsewhere.⁴

EXPERIMENTAL PROCEDURES

The short-tube apparatus and the evaluation techniques used throughout the program were described in Parts I and II of this series.^{1,2} Briefly, premixed metal fluorides (MF_6) were metered into a flash evaporator, mixed with hydrogen, and directed vertically upward across a rotating mandrel (0.5 in. diam). A vertical ceramic flue inside the chamber confined the crossflow of the gas to about a $1\frac{1}{4}$ -in. length of the mandrel. An induction coil wrapped around the tube heated the mandrel uniformly throughout the 1-in. length exposed to the flowing gas.

A sketch of the long-tube fabricating apparatus is shown in Fig. 1. This is basically the same design as that used for short tubes.¹ Major modifications include: (1) a rectangular-cross-section flue that directs the feed gas across a 4-in. length of the mandrel and (2) a cam driven ratchet mechanism for slowly translating the rotating mandrel along its axis through the plating region for a distance of 16 in. The cross-sectional view showing the gas flow pattern around the mandrel illustrates the technique that was developed for shielding the tungsten-wire bristle brush¹ from the plating gas.

Mechanical properties of the deposits were measured with a hydraulically actuated ring tensile tester.^{2,5} Ring-shaped tensile specimens were prepared by slicing the coated mandrels into short cylinders (rings) and leaching away the molybdenum substrate.²

RESULTS AND DISCUSSION

TUBE FABRICATION

The tube fabrication apparatus (Fig. 1) was developed to the point where 12-in. lengths of homogeneous W-25Re alloy tubing could be produced.⁴ Microstructure, composition, and room temperature tensile properties of the tubes were found to be the same as those produced under the same conditions with the short-tube apparatus. This was fortunate in that it permitted direct application of short-tube results to the larger equipment.

Long-tube fabrication activities were limited to the design, construction, and debugging of equipment and to the production of a few samples for evaluation. The original short-tube apparatus was used for all of the work presented here.

Fig. 1. Long-tube fabrication apparatus.

MECHANICAL PROPERTIES

The effect of rhenium content on the room temperature tensile properties of ductile specimens is shown in Fig. 2. This graph summarizes the data for all unbrushed deposits that showed measurable elongation after a 1-hr anneal at 1400°C. Since the ultimate strength/yield strength ratio was generally between 1.0 and 1.1, the correlation can also be applied to yield strength. Other annealing temperatures (1000 to 1800°C for 1 hr) resulted in similar correlations: Uniform elongation increased with increasing annealing temperature to a maximum of 4.5% at 1600°C and strength increased with decreasing annealing temperature to a maximum of about 250,000 psi at 1200°C.

The strength of brittle specimens varied from very low values up to those given for ductile deposits. The results from brittle deposits were omitted from the graph to show that strength is strongly related to rhenium content if the microstructure is such that brittle failures are avoided. The increase in maximum value of uniform elongation with increasing rhenium content suggests that room temperature ductility is also a function of rhenium content. Similar results have been reported for wrought alloys.⁶

A variety of plating conditions resulted in strong, ductile deposits. In the composition range where federite (A-15 structure)^{1,2} was present in the as-deposited microstructure, most of the ductile deposits were free of grown-in porosity, and annealing had produced a fine grained, equiaxed, single phase microstructure (Fig. 3). At lower rhenium concentrations, where federite was absent, the ductile deposits were also free of porosity; but very little recrystallization occurred on heat treating at 1400°C, and the structures were relatively coarse grained with a feathery columnar appearance. Brushing^{1,2} these federite-free deposits during plating resulted in a fine grained, more equiaxed deposit with improved ductility and strength (Table 1).

The deposition conditions that produced ductile deposits and the relationships between microstructure, composition, and the independent variables are discussed below.

MICROSTRUCTURE

Prior to heat treatment, the microstructures of ductile deposits containing about 25% rhenium were always characterized by a fine dispersion of federite in a matrix of the tungsten-rich bcc terminal solid solution (Fig. 4). These deposits were brittle in the as-deposited condition. On heat treatment, the finely dispersed federite grains transformed to the equilibrium structure and in effect acted as nuclei for recrystallization. The resulting single phase structures were equiaxed, fine grained, and ductile if there was no grown-in porosity. Deposits containing large columnar federite grains (Fig. 5) or grown-in porosity were generally weak and brittle regardless of the heat treating cycle.

Fig. 2. Effect of rhenium content on room-temperature tensile properties. All specimens annealed at 1400°C for 1 hr.

Fig. 3. Ductile ring tensile specimen—27.5% Re. (a) Necking in fracture region. (b) Microstructure.

Conditions: 600°C, 10 Torr, 2800 cc H₂/min, 140 cc MF₆/min, 10.3% ReF₆ in MF₆, annealed 1 hr at 1400°C.

Properties:

Ultimate tensile strength	207,500 psi
Yield strength (0.02%)	206,500 psi
Uniform elongation	0.9%
Reduction in area	75%

Table 1. Room temperature tensile properties of unbrushed and brushed CVD W-Re alloy.

Deposition temp (°C)	Brush rate (cycles/min)	Feed gas flow rate (cc/min)		Deposition rate (mils/min)	Composition (wt% Re)	Ultimate tensile strength (kpsi)	0.02% yield strength (kpsi)	Uniform elongation (%)	No. of tests averaged
		H ₂	MF ₆						
800	None			0.40	8.5	100.9	95.7	0.08	2
	40	500	140	0.44	9.5	126.1	106.3	0.83	2
	60			0.42	8.5	123.8	122.0	0.45	2
	None	1000	280	0.83	10.0	109.3	106.3	0.04	2
	30			0.70	9.0	123.1	118.0	0.54	4
675	None	500	140	0.32	17.5	134.2	124.7	0.75	4
	40			0.23	17.5	165.2	160.4	1.70	2
650	None	500	140	0.24	18.5	137.0	131.4	0.65	2
	60			0.28	20.5	172.9	171.5	1.10	3
	None	420	140	0.25	20.5	149.8	147.2	0.32	2
	60			0.27	21.0	169.3	166.9	1.45	4
600	None	500	140	0.25	24.0	169.3	168.0	1.10	2
	60			0.23	26.0	186.9	183.2	1.47	2

Conditions: Annealed 1 hr at 1400°C
 Pressure 10 Torr
 6.8% ReF₆ in MF₆

(a) 1000X

(b) 1000X

(c) 1000X

(d) 1000X

Fig. 4. Effect of dispersed federite on the microstructure of W-Re alloys. (a) As-deposited structure with very little federite (22.5% Re). (b) Same as (a) after 1 hr at 1400°C. (c) As-deposited structure containing federite dispersion (26.0% Re). (d) Same as (c) after 1 hr at 1400°C. (See Fig. 6 for deposition conditions.)

100X

1000X

Fig. 5. Massive columnar federite (dark) in terminal solid solution.
Average composition 21% Re.

Conditions: 600°C, 40 Torr, 500 cc H₂/min, 70 cc MF₆/min,
14.3% ReF₆ in MF₆; deposition rate 0.34 mils/min.

The effects of the deposition variables on microstructure can be discussed in terms of changes in the slope of a typical Arrhenius graph relating temperature and deposition rate (Fig. 6). This graph compares maximum rate data for unalloyed tungsten¹ with the results of a series of codeposition experiments in which temperature alone was varied.

The location of the upper rate break in Fig. 6 was determined by the gas supply rate as discussed in Part I.¹ Deposits formed near or above this temperature were usually porous, nodular, and brittle. Between the upper and lower rate breaks the rhenium content increased with decreasing temperature and the curve was parallel to, but slightly above, the curve for unalloyed tungsten. From the top to the lower end of this range the free surface topography gradually changed from a coarse to an extremely fine faceted appearance. At the lower end of this range the average rhenium concentration increased to the level where significant quantities of federite could nucleate and grow, and the slope again changed noticeably to form the lower rate break point. This was the region where finely dispersed federite and optimum properties were observed. The topography here became markedly smooth, glossy, and featureless on a finer scale. At still lower temperatures, a hillock surface with nodules developed, and as federite became predominant the topography again became faceted.

Rate breaks occurred at different temperatures and rates when other combinations of gas composition, pressure, and gas velocity were used. The upper break moved down the curve as the gas supply rate was decreased, and the lower break moved up the curve when the change in variables increased the rhenium rate (total rate $\times$ fraction of rhenium in the deposit) or decreased the tungsten rate. As the two breaks approached each other, the temperature range suitable for producing porosity-free deposits containing 25% or less rhenium became very narrow. Porous, nodular structures with massive federite grains were produced when the two breaks overlapped or were indistinguishable; i.e., when a portion of the curve was not parallel to the maximum rate curve for unalloyed tungsten.

The distinct change in microstructure that occurs as the temperature is lowered in 10°C steps through the lower rate break temperature is shown in Fig. 7. Accompanying changes in deposition rate, rhenium content, and hardness are given in Fig. 8. These results indicate that the lower rate break is associated with the appearance of federite in the microstructure and that the temperature must be maintained within a narrow range near the lower rate break to produce 25% rhenium deposits with finely dispersed federite in the microstructure.

The occurrence of finely dispersed federite grains near the lower rate-break point is probably related to the fact that the mandrel is rotated. During rotation, each portion of the surface is exposed

Fig. 6. Effect of temperature on deposition rate and alloy composition.
Conditions: 1500 cc/min H_2 , 500 cc/min MF_6 , 10 Torr.

Fig. 7. Effect of deposition temperature on microstructure. As electropolished. (See Fig. 8 for conditions.)

Fig. 8. Effect of deposition temperature on deposition rate, microhardness, and composition.

Conditions: 640-710°C, 10 Torr, 1500 cc H₂/min, 500 cc MF₆/min, 6.8% ReF₆ in MF₆.

cyclically to windward (rhenium rich) and leeward (rhenium poor) environments. This results in a cyclic microscopic variation in the rhenium content of the deposit in the radial direction, as discussed in Part I.¹ If the rhenium concentration on the windward side is high enough to nucleate federite but low enough on the leeward side that the federite grains do not continue to grow, then a fine dispersion of federite may be observed. When the deposit contains considerably more than 25% rhenium, alternate layers of federite and solid solution may be deposited (probably epitaxially) to form rather large two-phase psuedocrystals. Massive federite such as shown in Fig. 5 occurs under conditions for which the upper and lower rate breaks are indistinguishable (discussed earlier).

The microstructure of brushed alloy deposits containing no federite (Fig. 9) was similar to that of the brushed but unalloyed tungsten deposits discussed in Part I.¹ Brushing increased the nucleation rate and resulted in a fine-grained, equiaxed structure with marked improvements in uniform elongation. Deposits containing dispersed federite were not greatly affected by brushing, although there was some increase in strength and ductility (Table 1).

COMPOSITION

The composition of codeposited specimens is determined by the relative deposition rates of tungsten and rhenium if the plating conditions are maintained constant throughout the experiments:

$$\text{Total rate } (R_T) \approx \text{tungsten rate } (R_W) + \text{rhenium rate } (R_{Re})$$

$$\text{Rhenium content} \approx \frac{R_{Re}}{R_T} .$$

The individual rates for tungsten and rhenium are discussed below in terms of rate controlling processes.

Unalloyed tungsten deposition was discussed in Part I,¹ where it was shown that the maximum rate was a function only of pressure and temperature: $R_{max} \propto P^{1/2} \exp (-16,000/RT)$. Rates lower than the maximum were observed if the gas supply rate was below the critical value required to maintain that rate. It was suggested that the maximum rate (R_{max}) was controlled by the slowest surface process; i. e., adsorption, desorption, or surface diffusion. Rates less than R_{max} were said to be controlled by the gas supply rate (reactant supply limited). Mass-transport control is a more acceptable term for the latter process, since gas-phase mass-transport is the slow step in the overall process when the rate is less than R_{max} .

In codeposition experiments, the maximum tungsten rate was also controlled by a surface process. The presence of rhenium influenced the tungsten rate slightly between the upper and lower rate

(a)

(b)

(c)

100X

1000X

Fig. 9. (a) Unbrushed, as deposited. (b) Brushed, as deposited. (c) Brushed, annealed 1 hr at 1400°C. See Table 1 for properties.

Conditions: 800°C, 10 Torr, 500 cc/min H₂, 140 cc/min MF₆, 6.8% ReF₆ in MF₆; deposit composition 8.5% Re.

breaks (Fig. 6). Below the lower break, the tungsten rate was strongly affected by the presence of federite. The change in slope (Fig. 6) suggests that the activation energy of the rate controlling process occurring on the surface is lower if the surface contains federite.

In contrast with the tungsten rates, the rhenium rates were found to be mass-transport controlled over the entire range of deposition conditions investigated. Apparently, the surface processes involved in the reduction of ReF_6 were all relatively fast and the overall rhenium rates were limited by the rate of transport of ReF_6 to the surface. The process can be approximated by applying mass-transport theory, which assumes that the total rate is determined by the rate of diffusion through the boundary layer;⁷ i. e., the rate is proportional to the drop in concentration across the boundary layer (ΔC), the diffusion coefficient (D), and the reciprocal of the boundary layer thickness ($1/\delta$):

$$R_{\text{Re}} \propto \Delta C \cdot D \cdot 1/\delta .$$

When the rates of the surface processes are fast relative to the rate at which ReF_6 arrives at the surface, the concentration at the gas-solid interface will approach zero and $\Delta C \approx C_{\text{Re}}$, the concentration of ReF_6 in the gas stream. C_{Re} , in moles/cc, is proportional to the partial pressure of ReF_6 in the gas stream:

$$C_{\text{Re}} \propto P_{\text{Re}} = \frac{Q_{\text{ReF}_6}}{Q_{\text{total}}} \cdot P_T ,$$

where Q is the mass flow rate (moles or g/min) and P_T is the total pressure. The diffusion coefficient is inversely proportional to P_T and approximately proportional to the $3/2$ power of the temperature ($D \propto T^{3/2}/P_T$). The boundary layer thickness (δ) is inversely proportional to the square root of Reynolds number (N_{Re}), which is in turn directly proportional to velocity (V), density (ρ), and the reciprocal of viscosity ($1/\mu$):

$$\delta \propto \frac{1}{\sqrt{N_{\text{Re}}}} = 1/(V\rho/\mu)^{1/2}$$

Viscosity is approximately proportional to $T^{1/2}$ and independent of P_T and V , and density is proportional to P_T . The boundary layer thickness is therefore proportional to $1/(V P_T^{1/2})^{1/2}$, and

$$R_{\text{Re}} \propto P_{\text{Re}} \cdot (V/P_T)^{1/2} \cdot T^{5/4} .$$

The inlet temperature of the gas was maintained at room temperature in all of the experiments. Variations in the temperature or temperature gradient in the boundary layer (due to heat transfer from the deposit) did not appear to affect the rhenium rate significantly except at higher deposition temperatures (Fig. 6 and Ref. 1, Fig. 13). Temperature can therefore be considered constant, and the rhenium rate should be approximately proportional to $P_{\text{Re}} \cdot (V/P_T)^{1/2}$ if the process is mass-transport controlled.

The correlation given in Fig. 10 includes most of the experiments carried out at 600 and 800°C. The trend is clear, although there is considerable scatter around the 45° line. Some of the scatter can be attributed to nucleation and growth of fine particles (smoke) in the boundary layer. This has been observed at higher pressures, lower velocities, and at higher temperatures. When gas phase nucleation occurs, the transport process becomes very complex and the deposition rate on the mandrel is decreased. Reducing the mole ratio (H_2/MF_6) to the stoichiometric ratio of 3 also appears to have lowered the rhenium rate (Fig. 10). This suggests that mass transport of hydrogen through the boundary layer may also control the rate (mixed control) if the mole ratio does not exceed 3 by some minimum amount.

Further evidence that the expression $R_{\text{Re}} \approx (V/P_T)^{1/2} \cdot P_{\text{Re}}$ is approximately correct is given in Fig. 11, where the effects of several gas compositions and total pressures are compared. In this series the total pressure and the ratio of ReF_6 to WF_6 were varied, while temperature and the mass flow rates of MF_6 and H_2 were maintained constant. Under these conditions Q_T is constant and, since $Q_T = \rho \cdot V \cdot \text{area} \propto P_T V$, the rhenium rate should be approximately proportional to Q_{ReF_6} :

$$\begin{aligned} R_{\text{Re}} &\propto (V/P_T)^{1/2} \cdot P_{\text{Re}} = (V/P_T)^{1/2} \cdot \frac{Q_{\text{ReF}_6}}{Q_T} \cdot P_T = \frac{(VP_T)^{1/2}}{Q_T} \cdot Q_{\text{ReF}_6} \\ &= \frac{(Q_T)^{1/2}}{Q_T} \cdot Q_{\text{ReF}_6} = \text{Const.} \cdot Q_{\text{ReF}_6} \end{aligned}$$

In the lower ranges of ReF_6 concentrations where most of the recent experiments were carried out, the ratio $R_{\text{Re}}/Q_{\text{ReF}_6}$ was constant, within experimental error, and pressure had no effect on R_{Re} .

Figure 11 also shows the effect of pressure and gas composition on the tungsten rate. The presence of rhenium has raised the rate slightly above that obtained for unalloyed tungsten, but the pressure dependence is approximately the same: $R_W \propto P^{1/2}$. At higher pressures the upper

Fig. 10. Correlation between R_{Re} and $(V/P_T)^{1/2} P_{Re}$.

Range of deposition conditions		
Temperature (°C)	600	800
V, velocity (cm/min)	1,400-83,000	2,700-57,000
P_T , total pressure (Torr)	5-80	5-40
P_{Re} , partial pressure ReF_6 (Torr)	0.01-0.68	0.04-0.33
H ₂ flow rate (cc-STP/min)	210-4000	105-1600
MF ₆ flow rate (cc-STP/min)	35-500	35-500

Fig. 11. Effect of pressure and gas composition on composition and deposition rates.

% ReF₆ in MF₆: $\diamond$ 6.8%, $\circ$ 10.5%, $\square$ 14.3%.

Conditions: 500 cc/min H₂, 70 cc/min MF₆, 600°C.

rate break region was approached and the tungsten rate dropped slightly below $R_{\max}$ as the process shifted toward mass transport control (Fig. 11). The shift was much greater when the same series of experiments was repeated at higher temperatures. In these experiments the tungsten rates all leveled off at about the same value and, since the rhenium rates were independent of pressure, the deposit compositions also became independent of pressure (Ref. 1, Fig. 4).

CONCLUSIONS

Recent developments have resulted in improved techniques and procedures for predicting and controlling composition, microstructure, and mechanical properties of CVD tungsten-rhenium alloy tubing. Density and composition can be related to processes controlling the deposition rates of the components. Porosity-free alloys can be deposited by operating under conditions that force the tungsten deposition rate into the surface controlled region; i. e., conditions under which the mass flow rate of WF_6 is greater than that required to deposit tungsten at the maximum rate

$$\left[R_{\max} \propto P^{1/2} \exp - \frac{16,000}{RT} \right].$$

Rhenium deposition rates are mass transport limited and can be approximated by the expression $R_{Re} \propto (V/P)^{1/2} P_{Re}$.

Brushing the surface during deposition results in fine-grained, equiaxed microstructures. Similar structures can be generated without brushing by taking advantage of the metastability of federite. Alloys in the 25% rhenium range can be deposited with federite present as a fine dispersoid. During subsequent heat treatment, transformation of the federite provides nuclei for grain refinement, and the final structure is equiaxed, fine-grained, and single phase.

Using these techniques, CVD tubing can be produced with mechanical properties equal to those obtained in wrought W-Re alloys.

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