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DUCTILE TUNGSTEN ALLOYS

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ABSTRACT OF THE DISCLOSURE

A wrought tungsten-base alloy containing up to 1.0 weight percent rhenium and between 1 and 35 parts per million manganese. The alloy exhibits a low ductile-to-brittle transition temperature and substantially the same strength and high temperature properties as unalloyed tungsten.

This invention relates to tungsten-base alloys characterized by ductile-to-brittle transition temperatures which are at or below room temperature.

It is well known that the ductile-brittle transition temperature of tungsten can be reduced by the addition of rhenium. While tungsten wire having room temperature ductility has been produced without the addition of rhenium, the fabrication of sheet, tubing and other wrought tungsten forms require in the neighborhood of 22–25% rhenium to achieve room temperature ductility.

In copending U.S. patent applications Ser. No. 443,690 and Ser. No. 443,692, both filed Mar. 29, 1965, there are disclosed improved tungsten-base alloys which incorporate between 1% and 10% rhenium yet can be fabricated into sheet having ductility at or below room temperature. These alloys also exhibit ultimate tensile strengths and recrystallization temperatures which are superior to those of unalloyed tungsten.

However, due to the scarcity and high cost of rhenium, it is desirable to have available an alloy which is ductile at or below room temperature yet employs even less of this relatively rare metal. Further, the alloy should be at least as strong and have a recrystallization temperature which is not less than that of unalloyed tungsten.

Accordingly, it is an object of our invention to provide a tungsten-base alloy which employs less than 1% rhenium and yet has a low ductile-to-brittle transition temperature.

Another object is to provide a tungsten-base alloy which is ductile at room temperature yet has substantially the same strength and high temperature properties as unalloyed tungsten.

In the present invention, tungsten-base alloys are produced by blending less than 1% rhenium powder and between 0.1% and 0.3% manganese powder with tungsten powder. (All percentages given are by weight of tungsten.) The blend is hydrostatically pressed at about 50,000 pounds per square inch to form a billet after it is presintered at about 1300° C. for approximately 4 hours in vacuo at a pressure of about 2×10^{-6} torr. Following the 1300° C. presintering, the blend is again presintered in vacuo at 1650° C. for an additional 4 hours. The 1300° C. presinter permits the manganese to combine with the residual oxygen in the blend to form manganese dioxide and the 1650° C. presinter volatilizes part of the manganese dioxide and much of the residual manganese.

Alternatively, a flowing atmosphere of hydrogen or an inert gas may be used in place of vacuum presintering. In such an atmosphere, the rate of gas flow should be sufficient to remove both the manganese and manganese dioxide vapors so that their vapor pressure at the billet is less than the normal vapor pressure of these elements at the sintering temperature.

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Following the two presintering steps, the blend is sintered at about 2350° C. for 6 hours under the same atmospheric conditions as was used in the presintering steps. During sintering the alloy is densified and almost all of the remaining oxygen combined with the manganese and removed as manganese dioxide. After sintering, the alloy is rolled to desired thickness and cleaned.

Analysis of samples of the resultant alloy by means of emission and mass spectrometry show that they contain between 1 and 35 parts per million manganese. This is considerably less than the amount of manganese initially introduced into the blend although it is more than the 0.3 to 0.4 parts per million residual manganese found in a typical unalloyed tungsten billet. The percentage of rhenium in the alloy remained substantially unchanged from the percentage initially incorporated in the blend. The oxygen content of the alloy samples is significantly less than that of unalloyed tungsten which contains between 50 and 100 parts per million.

Measurement of ductile-to-brittle transition temperatures were carried out for a number of compositions by attempting to bend at several temperatures specimens 1 inch long by $\frac{3}{8}$ inch wide by 0.04 inch thick over a radius four times the thickness of the specimen through a 105° angle. The minimum temperature at which the sample cracked after a small but perceptible amount of bending (nil ductility temperature) was measured as was the minimum temperature at which the sample bent 105° without cracking (ductility temperature).

Alloy sheet 0.04 inch thick was fabricated in the manner previously described by blending element al manganese, rhenium and tungsten in the percentages given in the following table. Samples were tested for ductile-brittle transition temperatures, oxygen content, tensile strength and recrystallization temperature.

TABLE I

Alloy, percent by weight			Ductile-to-Brittle Transition Temperature (° C.)		Oxygen Content, parts per million
Mn	Re	W	Nil Duct. Temp.	Ductility Temp.	
0.10	None	Bal.	100	125	22
0.10	0.20	Bal.	10	25	9
0.10	0.50	Bal.	—5	0	16
0.10	0.75	Bal.	—20	—5	38
0.15	None	Bal.	25	40	-----
0.20	None	Bal.	50	70	13
0.20	0.50	Bal.	0	10	2
0.30	None	Bal.	35	50	28
0.30	0.50	Bal.	0	10	26
0.30	1.00	Bal.	25	35	-----

The above alloys exhibited ultimate tensile strength at 1650° C. of approximately 14,000 pounds per square inch and recrystallization temperatures in the range 1400° C. to 1550° C. These values are slightly higher than those obtained from unalloyed tungsten made by the described process. The nil ductile-to-brittle transition temperature of unalloyed tungsten is 75° C. and the ductility temperature 80° C.

Analysis of the alloys by emission spectrometry indicated that the residual manganese content was less than 35 parts per million in all cases. Mass spectrometer analyses of selected alloys obtained by blending manganese and rhenium elemental powders with tungsten prior to presintering indicate that the residual manganese increases with the manganese addition as shown in the following table.

TABLE II

Parts per Million in Blend before Sintering			Parts per Million in Sintered Alloy		
Mn	Re	W	Mn	Re	W
1,000	5,000	Bal.	1	Unchanged	Bal.
1,000	7,500	Bal.	2	do	Bal.
3,000	None	Bal.	3	do	Bal.
3,000	5,000	Bal.	13	do	Bal.

As indicated in the table, the amount of manganese in the final alloys analyzed with the mass spectrometer was between one and 13 parts per million. Also, the percentage of rhenium in the final alloy was found by emission spectroscopy to be substantially unchanged from that employed in the initial blend.

As many changes could be made in the above described process and many different compositions could be produced without departing from the scope thereof, it is intended that all matter contained therein shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. A sintered alloy characterized by high strength at elevated temperatures and a low ductile-to-brittle transition temperature in the range -5° C. to $+35^{\circ}$ C. consisting essentially of up to 1 weight percent rhenium, between 1 and 35 parts per million manganese, and the balance tungsten.

2. A sintered alloy characterized by high strength at elevated temperatures and a low ductile-to-brittle transition temperature in the range -5° C. to $+35^{\circ}$ C. consisting essentially of approximately 0.5 weight percent rhenium, approximately 1 part per million manganese and the balance tungsten.

3. A sintered alloy characterized by high strength at elevated temperatures and a low ductile-to-brittle transition temperature in the range -5° C. to $+35^{\circ}$ C. consisting essentially of approximately 0.75 weight percent rhenium, approximately 2 part per million manganese and the balance tungsten.

4. A sintered alloy characterized by high strength at elevated temperatures and a low ductile-to-brittle transition temperature in the range -5° C. to $+35^{\circ}$ C. consisting essentially of not more than 1 weight percent rhenium, at least 1 part per million manganese, and the balance tungsten.

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