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**TUNGSTEN BASE ALLOY TREATMENT**

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 3 Claims. (Cl. 148-4)

**ABSTRACT OF THE DISCLOSURE**

A method for producing a tungsten-rhenium alloy containing from about 10 to 35% rhenium comprises mechanically deforming said alloy at least 50% at a temperature of at least 2500° F. followed by subjecting said alloy to at least 5% further mechanical deformation at a temperature below the recrystallization temperature of said alloy.

This invention relates to improvements in the manufacture of tungsten and tungsten base alloy products and relates in particular to a new and novel method for producing a ductile and workable tungsten-rhenium alloy.

Tungsten and tungsten base alloys are known to possess particularly desirable high temperature, physical and mechanical properties. However, the commercially available tungsten and, with one exception (W-Re), the known tungsten base alloys are brittle at ordinary room temperatures so that cast tungsten or tungsten base alloys may not be conveniently worked or formed into desired shapes at such temperatures.

A partial solution to the room temperature brittleness of tungsten and tungsten base alloys has been the discovery of the tungsten-rhenium alloy system. In this system, rhenium additions to tungsten and tungsten base alloys have been found to improve the ductility characteristics of the material. Tungsten-26% by weight, rhenium alloys have been found, under specific processing conditions, to possess improved ductility at room temperature. The degree of room temperature ductility and formability of this alloy, however, is erratic because the composition is borderline or extremely critical in this respect with no safety margin. A sigma phase occurs in large globular form in the matrix of the alloy if the rhenium content is only slightly higher than the 26% maximum, and this phase causes a loss in ductility. Since the ductile-brittle transition temperature of the alloy is lowered as the rhenium content is raised, the 26% concentration cannot be lowered without a corresponding depreciation in room temperature ductility.

The physical and mechanical properties of tungsten and tungsten base alloys, particularly their high temperature strength properties, makes them a particularly desirable material for use in space technology. However, components employed in space hardware such as space capsules, etc., must not only meet the very high temperature mechanical requirements such as are imposed during re-entry, but also must have cryogenic properties. Such components must be particularly resistant to thermal shock since it is not unusual for one surface to be exposed to very high temperatures (as high as 3500° F.) while the opposite surface is subjected to very low temperatures, i.e., the temperatures of liquid oxygen. Although the addition of rhenium to tungsten improves its ductility and hot workability, the alloy remains brittle at low temperatures and is susceptible to fracture when exposed to thermal shock, particularly that imposed by applications such as described above.

I have discovered a method for producing tungsten-rhenium alloy products that possess both highly desired high temperature mechanical properties and exceptional

low temperature ductility. By employing my method, products made from tungsten-rhenium alloys may be consistently formed at room temperature. Rhenium may be effectively employed over an extended range. Tungsten-rhenium products produced by my method possess a combination of high-temperature strength and low temperature ductility that makes them particularly resistant to thermal shock. Products may be produced by my method that are ideal for use in space technology and like environments.

In general, my invention comprises the following sequence of steps:

Step 1—Producing a cast tungsten-rhenium article.

Step 2—Subjecting the cast article to at least a 50% mechanical deformation at a temperature of 2500° F. or above.

Step 3—Subjecting the article to further deformation at a temperature below the recrystallization temperature of the alloy.

Additionally, an optional concluding heat treatment may be desirable depending on the process history of the alloy and its intended use.

By employing the above process cycle, I have been able to produce tungsten-25% rhenium sheet having a ductile-brittle transition temperature below -300° F. This is about 200° F. lower than any of the well-known tungsten base alloys including the tungsten-rhenium system.

My method may be employed to lower the ductile-brittle transition temperature of a tungsten base alloy containing rhenium and is particularly effective in lowering the ductile-brittle transition temperatures of tungsten or tungsten base alloys that contain from about 10% to about 35% rhenium (by weight). The significant lowering of the ductile-brittle transition temperatures permits the utilization of W-Re alloys that exhibit normally embrittling sigma phase in their matrices (W-26 to 35% Re).

By tungsten base alloy, I mean an alloy containing at least about 50% by weight tungsten.

Step 1 of my method encompasses any method for producing a cast W-Re structure as contrasted from the usual sintered powdered metal method of consolidating these alloys. Melting of the material provides a necessary purification and homogenization. I have had particular success by using the vacuum consumable arc cast method in which powder consolidated electrodes of the alloy are melted in water-cooled copper molds. Other suitable methods would include electron beam melting and skull casting.

The term "cast tungsten-rhenium" as used in the present specification and claims includes any structure resulting from the solidification of molten metal whether such metal is actually cast or teamed into molds or not.

The mechanical deformation of Steps 2 and 3 of my process encompasses any type of working or shaping such as extruding, forging, rolling, swaging, etc. The percent of deformation is the percent of reduction in gauge, diameter, thickness, etc., or the equivalent of such reduction in dimensional change of the cast alloy article.

I have used the extrusion process to achieve particularly significant reductions of 60 to 75% while holding the metal at a temperature of about 3700° F. in the performance of Step 2 of my process.

The initial deformation of at least 50% of Step 2 of my process is preformed at 2500° F. or any temperature above 2500° F. to the melting point of the alloy.

The preferred amount of deformation accomplished by Step 3 of my process is dependent on the ultimate use of the object. Such working must be accomplished at temperatures below the temperature of recrystallization of the subject tungsten-rhenium alloy. Such temperature varies with varying compositions of the alloy and the history of

in fabrication. The temperature of recrystallization may be readily determined, however, by microscopic examination of heat treated specimens or other well-known techniques. This working may take place at temperatures be-

gauge material rolled at 1800° F. and the 3/8" swaged material were subjected to mechanical testing at room and elevated temperatures. The results of these tests are set forth in Table I below.

TABLE I.—AVC TUNGSTEN/25% RHENIUM TENSILE PROPERTIES

Gauge	Form	Test Temp., ° F.	UTS×10 <sup>3</sup>	.2 YS×10 <sup>3</sup>	R.A.	Percent EL
.020	Cold Rolled	R.T.	235.1	229.8	-----	7.4
.020	do	900	165.3	163.4	-----	2.0
.020	do	2000	141.6	132.9	-----	3.1
.020	do	2400	100.9	92.6	-----	17.4
.020	do	3000	31.3	20.1	-----	34.4
.020	do	3500	20.2	18.9	-----	95.9
.040	Hot-Cold Rolled	900	143.7	140.4	36.1	10.5
3/8"	Swaged	R.T.	241.8	225.1	-----	16.0

low room temperature down to such temperatures where adequate ductility for such working is lacking, preferably such temperature will range from room temperature to the temperature of recrystallization.

Any amount of working in the performance of Step 3 will be beneficial in lowering the ductile brittle temperature of the material, so that the percent of working effected depends upon the ultimate use to which the material is to be applied. For example, if particularly low temperature ductility is desired for applications such as the space hardware applications described above, it is necessary to effect minimum reductions of 60% at temperatures up to (but not including) the temperature of recrystallization.

If one's need is to achieve a lesser lowering of the ductile brittle transition temperature, a lesser amount of Step 3 deformation may be required. A reduction of at least about 5% is needed to effect an appreciable improvement in ductility.

If the deformation of Step 3 is a working which results in the production of a finished article (so far as working is concerned) then this article will possess the improved lowered ductile brittle transition temperature and resistance to thermal shock inherent in articles produced in accordance with the present invention.

The optional concluding heat treatment may be desirable to effect stress relief and further enhance the ductility of the article. In some instances such as in fabricating thin sheet (below .020" gauge) and depending on the prior work history, the concluding anneal may be eliminated. A recrystallizing anneal may also be desirable for some specific applications.

To demonstrate my invention, 1 3/4" diameter prealloyed pressed and sintered electrodes of tungsten-25% rhenium were vacuum arc melted. The vacuum arc cast ingots were machined into extrusion billet size (3 1/16" diameter). A chemical analyses of these billets prove them to be tungsten-25%, by weight, rhenium plus minute (less than .05%) quantities of impurities. These billets were extruded to 1.5" diameter rounds at temperatures of approximately 3700° F. The nose and tail of the extrusions were abrasively cropped and the surfaces were abrasively cleaned.

One extruded and conditioned round was press forged to sheet bar at 2400° F., then rolled to .125" thickness at 2300° F. and recrystallize annealed. A portion of this material was then hot-cold rolled (1800° F.) to .040" gauge, and a portion was reserved for testing. A portion of the .040" gauge material was further hot-cold rolled to .030" gauge (1800° F.), and a portion was reserved for testing. A portion of the .030" gauge material was cold rolled to .015" gauge, a second portion of the .030" gauge material was cold rolled to .020" gauge, and a third portion of the .030" gauge material was reserved for testing.

A second extruded and conditioned round was swaged to 3/8" diameter at temperatures ranging from 2000° F. to 2400° F.

Portions of the 0.20" gauge cold rolled sheet, the .040"

The .015" gauge and .020" gauge cold rolled and the .030" hot-cold rolled (1800° F.) sheet were tested to determine ductile brittle transition temperatures. The results are set forth in Table II below.

TABLE II.—DUCTILE BRITTLE TRANSITION OF TUNGSTEN-25% RHENIUM SHEET (ALL SAMPLES IN TRANSVERSE DIRECTIONS)

Gauge (in.)	Bend Rate 1" per minute		Remarks
	T-Radius	DBTT <sup>1</sup> (° F.)	
.030	4	-225	Hot-Cold Rolled.
.020	4	<sup>2</sup> < -300	Do.
.020	4	< -300	Cold Rolled.
.020	1	-225	Do.
.015	4	< -300	Do.
.015	1	-225	Do.

<sup>1</sup> Ductile Brittle Transition Temperature.  
<sup>2</sup> < Symbol indicates samples were not broken and brittle range was not determined.

It will be readily noted from the data of Tables I and II that the practice of the present invention is particularly effective in lowering the ductile brittle transition temperature of tungsten-rhenium alloys. The tungsten-rhenium alloy was vacuum consumable electrode melted so as to meet the requirement of having a cast structure as defined in Step 1 of my process. The cast ingots were extruded to 1.5" diameter from 3 1/16" diameter at 3700° F. which constitutes a reduction in diameter exceeding 50% (Step 2). All of the flat rolled specimens were deformed or worked in a manner to effect a reduction greater than 60% at temperatures below the recrystallization temperature when forged to sheet bar at 2400° F. and rolled to .125" at 2300° F. (Step 3). The swaged specimens were also mechanically deformed at temperatures below the recrystallization temperature (2000–2400° F.) at a deformation exceeding 60%.

From Table I it may be seen that the .020" gauge cold rolled material, the .040" hot-cold rolled material and the 3/8" swaged material possess particularly desirable high temperature and room temperature mechanical properties. Ductility as measured by elongation and reduction in area is particularly impressive.

From Table II it may be seen that the .030", .020" and .015" gauge hot-cold rolled and cold rolled materials exhibited exceedingly low temperature ductility by successful bending over a 4 T-Radius at temperatures as low as -300° F.

While I have described certain presently preferred embodiments of my invention, it is to be understood that they may be otherwise embodied within the scope of the appended claims.

I claim:

1. A method for producing a tungsten-rhenium product containing about 10% to about 35% rhenium and characterized by a low temperature ductile brittle transition temperature comprising:

(a) producing a cast tungsten-rhenium alloy article;

- (b) subjecting said article to at least 50% mechanical deformation at a temperature of at least 2500° F.; and
- (c) subjecting said article to at least about 5% further mechanical deformation at a temperature below the recrystallization temperature of said alloy. 5
2. The method set forth by claim 1 wherein said mechanical deformation at a temperature below the recrystallization temperature of said alloy amounts at least to 60% deformation.
3. A tungsten-rhenium alloy product characterized by a low temperature ductile brittle transition temperature, produced by the process of claim 1.

**References Cited**

## UNITED STATES PATENTS

3,236,699 2/1966 Pugh et al. ----- 148—11.5

## OTHER REFERENCES

Plansee Proceedings, 1955, Geach et al., "The Alloys of Rhenium with Molybdenum or With Tungsten and Having Good High Temperature Properties," pp. 245-253 relied on.

10 Plansee Proceedings, 1958, Jaffee et al., "The Effect of Rhenium on the Fabricability and Ductility of Molybdenum and Tungsten," pp. 394-406 and 410 relied on.

CHARLES N. LOVELL, *Primary Examiner*.

UNITED STATES PATENT OFFICE  
CERTIFICATE OF CORRECTION

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It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, line 66, "succeptible" should read -- susceptible --. Column 3, line 1, "in" should read -- its --; line 75, "0.20" should read -- .020 --. Column 4, line 60, "0.20" should read -- .020 --.

Signed and sealed this 12th day of August 1969.

(SEAL)

Attest:

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Commissioner of Patents