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PROPERTIES OF TUNGSTEN WIRE IN POWDER METALLURGICAL PROCESSING WITH ULTRAFINE POWDER

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The paper presents the results of the investigation of properties of the tungsten wire obtained from powders with particle sizes from 1 to 5 μm with the addition of ultrafine powders with particle sizes of less than 0.5 μm produced by the reduction of hydrogen plasma at a high temperature. Different compositions of mixtures were studied and a new optimal composition was developed. Methods of its compressing and presintering of bars were also developed. The results showed that mechanical and performance properties of the wire with a new composition have higher stability compared to the properties of the wire obtained by the standard technology. They allowed:

– to lower the creep for the wire with the diameter of 1250 μm,
– to achieve a stapel structure for the wire with the diameter of 650–1450 μm;
– to improve the spiralability of the wire;
– to increase the good-to-bad yield to 3.79 % for the wire with the diameter of 120 μm.

Keywords: sintering; ultrafine powder; wire; properties.

Introduction

The tungsten wire used for the production of filaments should have the non-sagging property which is normally achieved by the addition of small quantities of KO_2, Al_2O_3, or Si elements.

However, the usual BA-grade of the non-sagging tungsten wire does not meet the requirements of industries manufacturing electrical lamps and electronics. The production of some new items, such as halogen lamps, requires the creation of super heat-resistant tungsten alloys.

An optional solution of this problem is the creation of original tungsten powder compositions with higher surface-active properties as compared to those of standard tungsten powder fillers, i.e. the production of the non-sagging tungsten wire made of a two-component molding powder mixture consisting of standard tungsten-BA powder and ultra fine or ultra dispersed (UD) powder gained by the process of reduction in high-temperature hydrogen plasma [1–3]. The powder made in such a way includes up to 40 percent of β-W and exhibits a much higher activity [4, 5].

In this work the following questions are discussed: preliminary and high-temperature processes of sintering pressed billets; the consequent pressure treatment of sintered billets; the structure and properties of the W-wire produced.

1. Preliminary and high-temperature sintering of billets

The specific surfaces of molding powder mixtures made of standard W-powders with different quantities of UD powders added are much wider than those of standard W-powders. That is why, to wet qualitatively such a mixture with a plastificator, a higher quantity of glycerin-in-alcohol solution (a plasticizing agent) was introduced into the mixture as compared to the standard powder.

As a rule, presintering of pressed tungsten bars is performed at the temperature of 1200 °C ± 50 °C, in hydrogen atmosphere, in one-stage operation. Presintering of bars made of standard-W-BA-UD-powder mixtures in one step of operation under such temperature leads to vigorous withdrawal of a high amount of the plastificator from the entire volume of the bars. As a consequence, this withdrawal causes expansion, delamination and full destruction of the bars.

Ultra dispersed (UD) powders, especially those with β-W modification, contain a high amount of oxygen and offer a high chemical activity which leads to a more intensive grain growth in the process of one-stage presintering at 1200 °C ± 50 °C. A grain is a conglomerate consisting of the particles of standard BA and UD tungsten powders. The intensive grain growth inhibits the withdrawal of the carbon excess from the billet and the reduction of oxide films because of the hindered access of hydrogen in the conglomerate.

In accordance with the existing technology, the solution of glycerin in alcohol containing carbon serves as a plastificator. Due to a high chemical activity of UD powders, mainly, when they contain β-modification of tungsten, the temperature of the tungsten powder carbidization decreases. Hence, in the process of one-step presintering at 1200 °C ± 50 °C carbon cannot be fully withdrawn from the bars but forms carbides where the access of hydrogen is hindered. This causes lower deformability of the bars under their pressure treatment. Therefore, the process of presintering of the bars of the standard W-BA and UD powder mixture carried out in one stage of operation at 1200 °C ± 50 °C leads to the production of billets unfit for further pressure treatment.

In this connection, the method of sintering of the bars made of powder mixtures with UD powders was developed and brought to a commercial level. The essence of the method consists in three-stage presintering:

– heating up to 300 to 500 °C and soaking during 0.3 to 0.7 hours;

– heating up to 600 to 750 °C and soaking during 0.3 to 1.0 hours;

– heating up to 1000 to 1200 °C and soaking during 0.6 to 1.0 hours.

This method allows to produce bars of UD powder mixtures fit for further pressure treatment.

After preliminary sintering carried out according to the new method the pressed bars were subjected to high-temperature sintering by the direct conduction of the electric current through them. In Table 1 characteristics of original materials, pressing parameters and parameters of the bars after high-temperature sintering are given. In the table: ρ_{st} – density of the standard powder; ρ_{udp} – density of the UD powder; ρ_{mix} – density of mixture; ρ_{com} – density after pressing; ρ_{sint} – density after sintering.

From Table 1 it is well seen that the density of the bars made of the UD powder mixture is lower than the density of the bars made of standard tungsten powders. The higher is the content of UD powders in the mixture, the lower is the density of the bars. The high pinhole porosity of the bars of powder mixtures is the result of the increased content of the remaining oxygen and the filler which are removed in the process of high-temperature sintering. Those processes which retard the bars' shrinkage are to a great extent to be overcome due to a high activity of UD powders in sintering. The bars containing UD powders have pronounced plasticity for the subsequent pressure treatment.

2. Treatment of sintered bars, W-wire structure and characteristics

Experimental master batches of the W-wire were prepared from tungsten-BA bars which in their turn were made of molding powder mixtures of standard tungsten-BA and UD powders. The studies of the W-wire microstructure revealed that UD powders contained in a molding powder mixture stabilize a stapel structure of the wire.

The balance batch of the W-wire with the diameter of 69 μm was fabricated from the bars pressed and sintered with the results given in Table 1. The results of microstructure investigations for the recrystallized wire are shown in Table 2.

These results clearly illustrate that the wire made from the bars with 12 percent content of UD powders exhibits a stable coarse-crystal structure with long grain boundaries for all wire diameters as compared with the wire produced from standard tungsten powders which does not meet requirements of The National Standards (GOST). If the diameter of the standard W-wire is equal to or below 120 μm, the structure of the wire is in fact the same as the structure of the wire obtained from the mixture of standard W-BA and UD powders.

The wire produced from the bars made of the mixture containing 6 percent of the UD powder could not withstand the drawing operation. Starting with the diameter of 510 μm the wire was torn on all bobbins during drawing.

The wire with the diameter of 1250 μm was tested for creep according to the technique described in GOST 19671–81. The results of testing for creep are given in Table 3.

From these results one can conclude that the W-wire made of the standard W-powder with more than 500 μm in the diameter does not fit for manufacturing filaments intended to stand up to 2500 °C and higher than working temperatures.

Table 1

Parameters of pressing and sintering bars with UD powders

Characteristics of original materials				Pressing parameters				Sintering parameters			
ρ_{st}, t/m^3	ρ_{udp}, t/m^3	D_{udp}, %	ρ_{mix}, t/m^3	Spec. press., MPa	ρ_{com}, t/m^3	Bar size, mm		Weld. current, A	Bar size, mm		ρ_{sint}, t/m^3
						h	*b*		*h*	*b*	
3.043	0.439	0	3.043	403	12.273	12.01	12.24	3600	10.61	10.62	17.680
		6	4.599	402	12.676	11.60	12.26	3600	10.38	10.80	17.673
		12	4.746	400	12.572	11.58	12.30	3600	10.38	10.83	17.447
		18	4.765	400	12.439	11.57	12.34	3600	10.26	10.92	17.409

Table 2

The microstructure of the recrystallized wire

Wire diameter, µm	Standard W-wire GOST 19671–81		UDP W-wire GOST 19671–81	
	Fit*	Unfit*	Fit*	Unfit*
1450	–	–	–	–
780	–	–	–	–
630	–	22, 25	12, 13	–
510	17, 18, 16	–	12, 13, 15	–
470	13, 16 17, 18	–	12, 13	–
320	27, 28, 30	–	26, 29	–
300	26, 27, 28	–	26, 29	–
185	–	36, 34, 37	33, 34	–
120	36, 34	–	33, 34	–
112	33, 34	–	33, 34	–
73.5	41, 40	–	41, 39	–
68.9	39, 41	–	41, 39	–
46.0	40, 41	–	41, 39	–
30.0	46, 49	–	46, 48	–

* Numbers of figures, appendix 7 of GOST 19671–81.

Table 3

Testing-for-creep results for the wire of 1250 µm diameter

Batch	Item No	Soaking time, hour	Elongation, mm	Structure according to GOST 19671–81, Appendix 7
Standard W-powder batch	1	4	4.5	Fit draught 3
	2	4	4.7	–
	3	4	4.7	–
	4	4	4.6	–
UD powder batch	1	4	2.4	Fit draught 1
	2	4	2.3	
	3	4	2.2	
	4	4	2.3	

Table 4

The good-to-bad yield for the standard W-BA powder wire and the wire containing UD powder

Wire diameter, µm	Good Wire Yield, %		
	Balance batch	UDP Batch	Current Shop-batches
510	78.52	80.25	77.7
300	72.16	74.52	70.83
120	67.61	71.40	65.70

Spiralability was tested according to the regimes specified in GOST 19671–81 using the wire with the diameter of 300 µm and less and winded on 10 bobbins. The spiralability of the wire made of the mixture of standard W-BA and UD powders is better than that of the wire produced from the standard W-BA powder only. Thus, the UDP W-wire is suitable for winding the spirals about the core with two and fewer factors over the entire range of diameters studied. The standard W-wire with 30 µm in the diameter fits for winding the spirals about a die core with 2.5 factors and more.

The good-to-bad yield for the wire obtained from the standard W-BA and the UD powder bars was cheeked using the wire of 510, 300 and 120 µm in diameters. Table 4 shows that the yield of good wire made of the UD powder bars is 1.73 % higher for the wire of 510 µm in the diameter, 2.36 % higher for the wire of 300 µm in the diameter and 3.79 % higher for the wire of 120 µm in the diameter than the yield of good standard W-BA powder wire.

Conclusion

The application of the bars made of the standard tungsten-BA and the tungsten- BA-UD-powder mixture for manufacturing the W-wire allows:

– to lower the creep for the wire of 1250 µm in the diameter,

– to achieve a stapel structure for the wire of large diameters, from 650 to 1450 µm,

– to obtain a stable staple structure along the wire length over the entire range of diameters from 30 to 1450 μm,

– to improve the spiralability of the wire,

– to increase the good-to-bad yield by 3.79 % for the wire of 120 μm in the diameter.

References

1. Haberko K. Characteristics and Sintering Behaviour of Zirconia Ultrafine Powders. *Ceramurgia International*, 1979, vol. 5, no. 4, pp. 148–154. DOI: 10.1016/0390-5519(79)90023-1

2. Kalamazov P.U., Tsetkov Yu.V., KalkovA.A. *Ul'tradispersnye poroshki vol'frama i molibdena* [Ultrafine Powders of Tungsten and Molybdenum]. Moscow, Metallurgiya Publ., 1988. 192 p.

3. Andrievskiy R.A. *Poroshkovoe materialovedenie* [Powder Materials Science]. Moscow, Metallurgiya Publ., 1991. 205 p.

4. Mymrin S.A., Leonov S.A. [Investigation of Pressing Tungsten with Ultrafine Powders]. *Trudy kafedry MiTOMD YuUrGU* [Proceedings of Machines and Technologies of Material Deformation Processes Department of the South Ural State University]. Chelyabinsk, SUSU Publ., 1998, pp. 137–143. (in Russ.)

5. Barkov L.A., Mymrin S.A., Chaplygin B.A. *Sposob polucheniya vol'framovykh zagotovok* [Method of Tungsten Blanks Receiving]. Patent RF, no. 2361697, 2009.

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СВОЙСТВА ВОЛЬФРАМОВОЙ ПРОВОЛОКИ ИЗ ПОРОШКОВ С ДОБАВЛЕНИЕМ УЛЬТРАДИСПЕРСНЫХ ЧАСТИЦ

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Представлены результаты исследования свойств вольфрамовой проволоки, полученной из порошков с размерами частиц от 1 до 5 мкм с добавлением ультрадисперсного порошка с размерами частиц менее 0,5 мкм, произведенного восстановлением в высокотемпературной водородной плазме. Изучались различные композиции смесей и разработан оптимальный состав новой композиции. Разработаны способы ее прессования и предварительного спекания штабиков. Результаты исследований показали, что механические и эксплуатационные свойства проволоки из новой композиции выше и стабильнее по сравнению со свойствами проволоки, полученной по стандартной технологии:

– ниже ползучесть для проволоки диаметром 1250 мкм;

– получена стапельная структура на проволоке диаметром 650–1450 мкм в диаметрах;

– улучшена спирализуемость проволоки;

– увеличен выход годного на проволоке диаметром 120 мкм на 3,79 %.

Ключевые слова: спекание; ультрадисперсный порошок; проволока; свойства.

Литература

1. Haberko, K. Characteristics and Sintering Behaviour of Zirconia Ultrafine Powders / K. Haberko // Ceramurgia International. – 1979. – Vol. 5, no. 4. – P. 148–154. DOI: 10.1016/0390-5519(79)90023-1

2. Каламазов, П.У. Ультрадисперсные порошки вольфрама и молибдена / П.У. Каламазов, Ю.В. Цветков, А.А. Калков. – М.: Металлургия, 1988. – 192 с.

3. Андриевский, Р.А. Порошковое материаловедение / Р.А. Андриевский. – М.: Металлургия, 1991. – 205 с.

4. Мымрин, С.А. Исследование прессования вольфрама с ультрадисперсными порошками / С.А. Мымрин, С.А. Леонов // Труды кафедры МиТОМД ЮУрГУ. – 1998. – С. 137–143.

5. Пат. 2361697 Российская Федерация, МПК B 22 F 3/12. Способ получения вольфрамовых заготовок / Л.А. Барков, С.А. Мымрин, Б.А. Чаплыгин. – 2007134474/02; заявл. 20.03.09; опубл. 20.07.09, Бюл. № 20. – 4 с.

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