EFFECT OF HEAT TREATMENT ON THE DISTRIBUTION OF ELEMENTS IN THE PHASE COMPONENTS OF A W—Ni—Fe ALLOY

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In a W—Ni—Fe alloy produced by high-temperature liquid-phase sintering round grains of a main tungsten phase containing small amounts of dissolved nickel and iron are distributed in a matrix which is a supersaturated solid solution of iron and tungsten in nickel (γ phase) [1, 2]. According to [1], during annealing the γ phase decomposes, the process being accompanied by the precipitation of excess tungsten. In [3] it was found that the tungsten phase of such an alloy has a much larger solubility for iron than for nickel and that the boundary zones of this phase become enriched in these elements. In a similar alloy produced by sintering at 1450°C and cooled with the furnace precipitated inclusions of an intermetallic phase, W(NiFe), responsible for a marked deterioration in the mechanical properties of the alloy, have been found at grain boundaries [4]. Clearly, a strong influence on the phase transformations accompanying the heat treatment of alloys of this type will be exerted by the character of distribution of the elements between the phase components of the alloys.

In the work described below, a study was made of the effect of various heat treatments on the distribution of elements between the tungsten phase and matrix grains in specimens of the heavy alloy investigated in [1]. Unetched microsections of alloy specimens were examined using a Cameca MS-46 electron probe microanalyzer with a probe diameter of $\sim$ 1 μm (operating conditions of the apparatus: U = 20 kV and I = 20 nA).* Analysis was based on L_{α_1} tungsten lines and K_{α_1} nickel and iron lines. As reference standards the pure metals were used. Examinations were made of tungsten phase grains of size not less than 30 μm and matrix fields of size not less than 10 μm. In radiation intensity measurements use was made of a fixed-step displacement device and of manual movement of specimens. Pulse reception time was 1 min.

Each radiation intensity determination was the average of nine measurements of nine tungsten phase grains and six measurements on six matrix fields. In calculations of element concentrations a noise correction was introduced. The correction, allowing for the atomic number, absorption, and fluorescent excitation, was calculated using data reported in [5]. Maximum relative errors in the determinations of tungsten, nickel, and iron contents were, respectively, 2, 3.5, and 5% for the tungsten phase and 2, 1.5, and 3.5% for the matrix. In addition, electron-absorption photographs of some parts of microsections were taken in the MS-46 apparatus.

The amounts of elements in the structural components of specimens subjected to various heat treatments are given in Table 1. The heat treatments employed induced appreciable changes in the amounts of elements both in the tungsten phase grains and in the matrix. The tungsten phase of the alloy was found to show preferential solubility for iron. The combined iron and nickel content of this phase varied, depending on the heat treatment conditions, in the range between 0.4 and 1.3 wt. %.

During the aging of quenched specimens and their prolonged annealing at 900°C there was a tendency for the concentration of iron and, to a smaller extent, nickel in the tungsten phase to fall. Annealing for 5 h at 1000°C appreciably increased the nickel and iron concentration in the tungsten phase. The boundary zone of the tungsten phase was as a rule enriched in nickel and iron.

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TABLE 1. Results of Electron Probe Microanalysis of Structural Components of Alloy

Alloy component	Aging conditions	Elements determined	Amounts of elements, %*														
			Conditions of annealing before aging (cooling in water)†														
			—			1200° C, 5 h			1000 °C, 5 h			900° C, 0,5 h			900° C, 5 h		
			A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
Tungsten phase	—	Ni	0,3	0,3	0,5	0,3	0,4	0,7	0,4	0,5	0,8	0,3	0,4	0,6	0,2	0,2	0,7
		Fe	0,6	0,7	0,8	0,6	0,7	0,8	0,8	0,8	0,9	0,6	0,6	0,7	0,3	0,5	0,6
	800° C, 24 h	Ni	0,3	0,3	0,6	0,4	0,4	0,7	0,4	0,5	0,7	0,3	0,3	0,5	0,2	0,2	0,8
		Fe	0,4	0,4	0,5	0,6	0,6	0,7	0,8	0,8	0,9	0,3	0,4	0,5	0,2	0,3	0,5
Matrix	—	Ni	51,5			56,8				45,0—53,0		50,9			50,0		
		Fe	20,6			22,4				17,0—21,8		21,5			22,4		
		W	27,9			20,8				26,0—37,0		27,6			27,6		
		Ni/Fe	2,50			2,53				2,4		2,37			2,23		
	800° C, 24 h	Ni	52,3			53,7				40,6—55,5		50,6			49,2		
		Fe	20,0			23,5				22,8—24,7		21,8			22,2		
		W	27,7			22,8				23,0—43,0		27,6			28,6		
		Ni/Fe	2,61			2,27				2,3		2,32			2,21		

*Measurements in the central zones of grains (A) and at distances of ∿5 (B) and 2 μm (C) from grain edges (in addition, nickel-to-iron percentage ratios are given for the alloy matrix).
†Starting heat treatment — sintering at 1550°C and quenching in water.

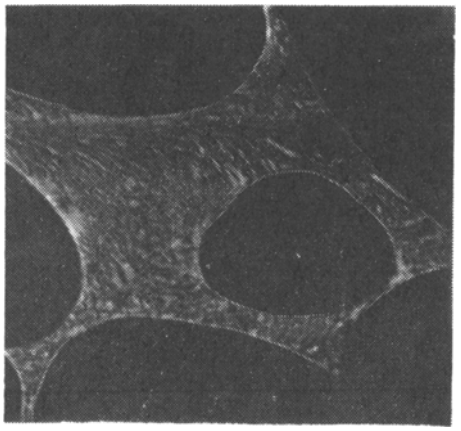

Fig. 1. Absorbed-electron photograph of structure of W–Ni–Fe alloy specimen, × 1500. Heat treatment: quenching after sintering at 1550°C + 5-h annealing at 1000°C, cooling in water.

According to results obtained, the solubility of tungsten at 1550°C in the matrix — a quenched γ phase — amounted to ∿ 28%. Annealing for 5 h at 1200°C lowered the tungsten content of the matrix to ∿21%, which was in accord with data cited in [6]. Excess tungsten precipitating out of the matrix under these conditions coagulated on the tungsten phase grains, and the nickel concentration in the boundary zone rose.

Of particular interest are the results of an investigation into the effect of 5-h annealing of quenched specimens of the alloy at 1000°C on the composition of its matrix. After this heat treatment an appreciable local heterogeneity of the matrix was observed, which was due to the fact that the size of the particles precipitated as a result of the decomposition of the supersaturated solution was commensurable with the size of the electron probe. Clearly, the matrix was heterogeneous also after annealing at 900 and 800°C, but, as the particle sizes in these cases were smaller than the probe diameter, the heterogeneity did not manifest itself clearly during microanalysis. A slight relative enrichment of the matrix in tungsten during annealing at 1000°C was due to the diffusion of nickel and iron from the matrix to the grains of the tungsten phase, rather than the coagulation of excess tungsten precipitating out of the γ phase on the grains of the tungsten phase being the dominant process. A study of the structure of such a matrix in absorbed electrons revealed that it contained very fine precipitated lamellar particles which (judging from the fact that they gave an absorbed-electron current similar to that obtained from the tungsten phase) corresponded in composition to the tungsten phase. An absorbed-electron photograph of a characteristic fragment of an alloy specimen is shown in Fig. 1.

Annealing for a short time (0.5 h) at 900°C had only a slight effect on the distribution of nickel and iron between the tungsten phase and matrix. In an alloy specimen annealed for 5 h at 900°C, because of a sharp fall in the solubility of iron in the tungsten phase, the relative amount of iron in the matrix was much higher compared with the state prevailing after quenching. In this case the nickel-to-iron concentration ratio in the matrix (Table 1) was equal to 2.23 (as against 2.50 for the alloy in the quenched condition). With rising iron content the decomposition of the supersaturated γ phase induced by aging would be expected, judging by data reported in [6], to become more intense, bringing about the precipitation of a substantial amount of excess phase.

Aging the alloy for 24 h at 800°C had virtually no effect on the solubility of nickel in the tungsten phase. At the same time, this treatment lowered slightly the amount of iron in the tungsten phase and as a rule raised its concentration in the matrix. An analysis of a quenched alloy specimen whose matrix had experienced additional decomposition as a result of aging after annealing (5 h at 900°C) revealed a slight fall in the nickel content of the matrix, a small rise in its tungsten content, and a further enrichment of the boundary zone of the tungsten phase in nickel.

The observed redistribution of elements in the alloy investigated was apparently due not only to the temperature dependence of the solubilities of the components in the phases but also to the fact that an equilibrium had not been attained in its structural constituents during sintering and heat treatment. The findings of this work should be taken into account in investigations into the relationship between the structure of alloys of this type and their properties.

LITERATURE CITED

1. S. S. Kiparisov, L. S. Vodop'yanova, et al., "Effect of heat treatment on the structure and properties of W–Ni–Fe alloys," in: Hard Alloys and Refractory Metals [in Russian], Vol. 16, Metallurgiya, Moscow (1976), pp. 280-287.
2. L. Ekbom, "The influence of microstructure of liquid-sintered tungsten-base composites on the mechanical properties," Scand. J. Metall., 5, No. 4, 179-184 (1976).
3. I. Ya. Dzykovich, R. V. Makarova, et al., "Distribution of elements during the formation of sintered alloys of the system W–Ni–Fe," Poroshk. Metall., No. 8, 62-69 (1965).
4. D. W. Edmonts and P. Jones, "Interfacial embrittlement in liquid-phase sintered tungsten heavy alloys," Metall. Trans., 10A, No. 3, 289-295 (1979).
5. N. A. Kudrya, Yu. F. Rudakov, et al., "Quantitative analysis of phase compositions in the microprobing of multicomponent alloys with the aid of an MIR-1 computer," in: Refractory Metals [in Russian], Vol. 19, Metallurgiya, Moscow (1978), pp. 5-12.
6. V. M. Agababova and I. N. Chaporova, "Boundary of the single-phase γ solid solution region in the ternary system W–Ni–Fe," Poroshk. Metall., No. 7, 65-72 (1969).