

HEAT-RESISTANT ALLOYS HARDENED WITH Ni<sub>3</sub>Nb

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A systematic study of the effect of alloying elements on the properties of Ni - Cr - Nb alloys [1, 2] led to the development of new alloys with high heat resistance and good weldability.

Here, we give the results from an investigation of the structure and properties of alloy KhN60MBVYu (14.5% Cr, 8% Mo, 5.5% W, 5% Nb, 1.1% Al, 0.04% C, the rest Ni) and the effect of replacing part of the nickel with iron (from 3.5 to 20.5%).

The alloys were melted in a 30-kg laboratory induction furnace. The ingots, weighing 7 kg, were forged into bars with a section of 15 × 15 mm at 1160-1000°C. Increasing the iron concentration to 10% had little effect on the workability of the metal in hot working. With 15% Fe or more the workability decreased and the yield of suitable metal was small.

In the metallographic examination of the alloy with 3.5% Fe we noted an intermetallic phase of the M<sub>7</sub>W<sub>6</sub> type; its size and quantity increased gradually with the iron concentration (Fig. 1). In contrast to the carbide phase, the intermetallic compound M<sub>7</sub>W<sub>6</sub> was not stained a dark color when etched in Murakami's reagent. In the alloy with 15.5% Fe the M<sub>7</sub>W<sub>6</sub> phase was of fairly large size and was observed radiographically. Located mainly in the grain boundaries (Fig. 1d), it prevents grain growth, and therefore one notes a tendency to grain refining with increasing concentrations of iron. When the alloy with 15.5% Fe is quenched from 1150°C (2 h) the grain size of the solid solution is grade 10 on the GOST 5639-65 scale.

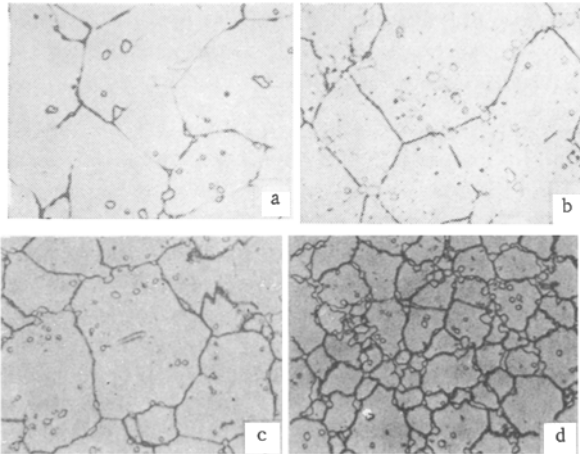


Fig. 1. Microstructure of alloys with different amounts of iron after quenching from 1150°C (2 h) (× 500). a) 0% Fe; b) 5.5% Fe; c) 10% Fe; d) 15.5% Fe.

The effect of iron on the mechanical properties of the alloy at room and elevated temperatures was determined after quenching from 1150°C and aging at 750°C for 15 h (Fig. 2). As the iron content is increased up to 5.5% the ultimate and yield strengths at 20°C are almost unchanged, and then they increase sharply, reaching a peak at 8% Fe, and then remain at the same level up to 15.5% Fe. The specific elongation and impact strength decrease continuously with increasing iron concentrations from 3.5 to 15.5% at 20°C. At 750°C the alloy with 10% Fe has the best combination

TABLE 1

Alloy	Weight increase (g/m <sup>2</sup> ·h) in test of (h):	
	100	1000
KhN60MBVYu. . . . .	0.09	0.034
KhN50MBVYu. . . . .	0.19	0.041
KhN67VMTYu (ÉP 202) . . . . .	0.27	0.072

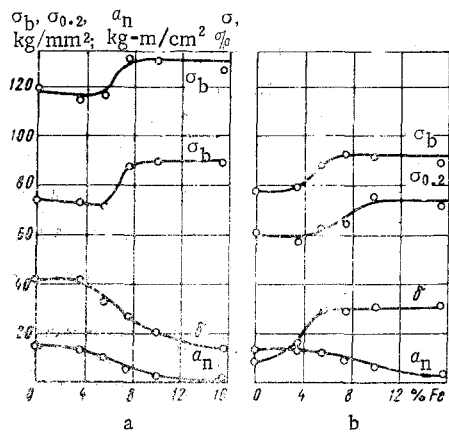


Fig. 2. Mechanical properties of alloys with different amounts of iron. a) Tested at 20°C; b) at 750°C.

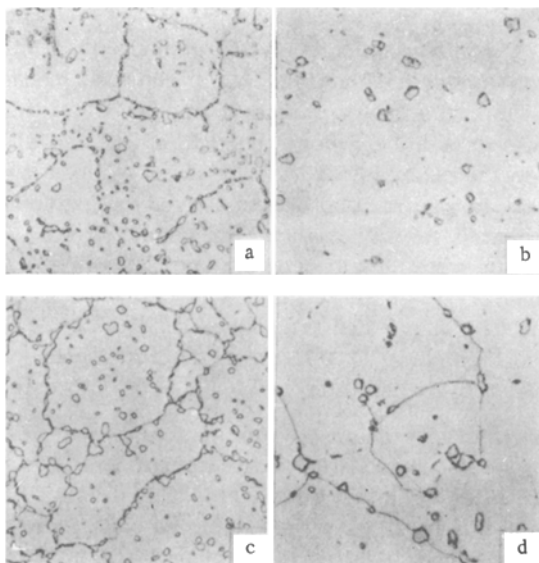


Fig. 3. Microstructure of alloys KhN50MBVYu (a, c) and KhN60MBVYu (b, d) after heat treatment at 1150°C with cooling in air (a, b) and in the furnace (c, d) ( $\times 500$ ).

An investigation of precipitation hardening at 700–900°C showed (Fig. 6) that the hardness increases monotonically with the aging time at 700 and 750°C. At 800, 850, and 900°C the hardness increases sharply during a certain period; the higher the temperature, the earlier this increase occurs. The hardness of the KhN60MBVYu alloy is below that of the KhN50MBVYu alloy both as quenched and after aging. The hardness curves of the alloys after aging at 800–900°C indicate a two-stage aging process, which was confirmed by microstructural and x-ray structural analyses.

The increase of the hardness in the first minutes of aging at 700–800°C is due to precipitation of a dispersed hardening phase distinguishable only in the electron microscope. Limited area electron-diffraction patterns were made\* to determine the crystal structure of the precipitating phase from the particles in

\* Under the direction of A. V. Smirnova.

of properties:  $\sigma_b = 90 \text{ kg/mm}^2$ ,  $\sigma_{0.2} = 75 \text{ kg/mm}^2$ ,  $\delta = 30\%$ ,  $a_n = 7 \text{ kg-m/cm}^2$ . Subsequently, we investigated two alloys: KhN60MBVYu and KhN50MBVYu (with 10% Fe).

As quenched from 1150°C the KhN50MBVYu alloy differs from the alloy without iron by the presence of the phase  $M_7W_6$  (Fig. 3a, b), higher hardness (20–25 HB units), and larger lattice parameters of the solid solution (5–6 kX), which is due to the distortion of the solid solution resulting from partial substitution of nickel atoms ( $r = 1.24 \text{ \AA}$ ) with iron atoms ( $r = 1.27 \text{ \AA}$ ). During slow cooling of alloy KhN50MBVYu from 1150°C (in the furnace) the  $M_7W_6$  phase coalesces (Fig. 3c) and a dispersed hardening phase distinguishable only in the electron microscope is precipitated in the boundaries and within the grains. The lattice constant of the solid solution in this case decreases from 3.602 to 3.598 kX (the precision of the measurements was  $\pm 0.0005 \text{ kX}$ ). After slow cooling of the KhN60MBVYu alloy the grain boundaries are barely distinguishable (Fig. 3d). The lattice constant of the solid solution is the same (3.596 kX) after cooling in air or in the furnace. The increase of the hardness (Fig. 4) during fairly slow cooling to lower temperatures from 1150°C and then quenching in water to fix the high-temperature structure indicated that the precipitation of the hardening phase began at 975°C in the KhN60MBVYu alloy and at 1000°C in the KhN50MBVYu alloy.

Structural transformations in alloys are ordinarily accompanied by changes in the physical properties. The change in the electrical resistivity of the water-quenched alloy KhN50MBVYu during continuous heating at an average rate of 100 deg/h (Fig. 5a) indicates the precipitation of hardening phase at temperatures of 650–950°C. The precipitation of the dispersed phase induces an increase of hardness at 650–850°C (Fig. 5b) and contraction of the sample. On the dilatometric curves (Fig. 5c) made during heating of quenched samples one finds two minimums, the first at 520–620°C, which is evidently due to the K state, and the second at 650–750°C, which is due to the precipitation of hardening phases. The effect is most pronounced on the curve for the water-quenched sample (1). After cooling in air the contraction during heating is considerably smaller (2), since a certain amount of hardening phase was precipitated during the previous cooling. After aging at 750°C the effect is very small (3).

the replica. In both alloys the dispersed phase has a fcc lattice with  $a \sim 3.59 \text{ \AA}$ . This phase, which is a metastable modification of  $\text{Ni}_3\text{Nb}$ , dissolves in itself the elements entering into the composition of the alloy.

The sharp increase in hardness during aging at 800–900°C is due to the transformation of this phase into the stable modification with an orthorhombic lattice. The substantial increase in hardness on precipitation of considerable amounts of stable phase was observed earlier for Ni–Ti alloys [3], where the phase initially precipitated also had a fcc lattice. The equilibrium phase is in the form of plates. After initial precipitation primarily in the grain boundaries (Fig. 7, a, c), the lamellar phase precipitates throughout the grains, forming a complex network resembling a Widmanstatten structure (Fig. 7d). The lamellar phase is formed more rapidly in KhN50MBVYu than in KhN60MBVYu. For example, after aging 50 h at 850°C a network of the lamellar phase ( $\text{Ni}_3\text{Nb}$ ) is visible in alloy KhN50MBVYu, while the phase occupies a considerably smaller volume of the grains in KhN60MBVYu (Fig. 7b).

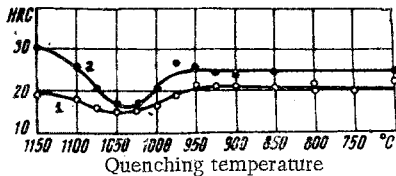


Fig. 4. Variation of hardness of alloys KhN60MBVYu (1) and KhN50MBVYu (2) with slow cooling from 1150°C (in the furnace) to different quenching temperatures.

Apart from the hardening phases, the alloys contain a cubic carbide  $\text{M}_6\text{C}$  that has no part in the hardening of the alloys during aging. According to limited area x-ray spectral analysis, the chemical formula of the carbide is of the type  $(\text{Ni}, \text{Cr}, \text{Fe})_3(\text{Mo}, \text{W}, \text{Nb}, \text{Al})_3\text{C}$ . It should be noted that it was impossible to distinguish the  $\text{M}_6\text{C}$  carbide and the  $\text{M}_7\text{W}_6$  phase by the color metallography method; they were detected by x-ray structural analysis of electrolytic residues.

The structural characteristics of the KhN60MBVYu and KhN50MBVYu alloys determine their mechanical properties and the type of heat treatment.

After quenching from 1200°C and aging at 750°C for 15 h both alloys have almost the same properties:  $\sigma_b = 110 \text{ kg/mm}^2$ ;  $\sigma_{0.2} = 70$

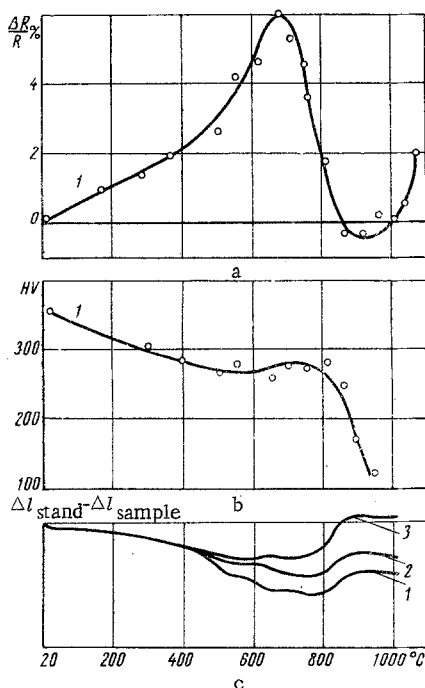


Fig. 5. Variation of resistivity (a), hardness (b), and length of the sample (c) of alloy KhN50MBVYu during continuous heating after heat treatment: 1) 1150°C, 2 h, water; 2) 1150°C, 2 h, air; 3) 1150°C, 2 h + 750°C, 15 h.

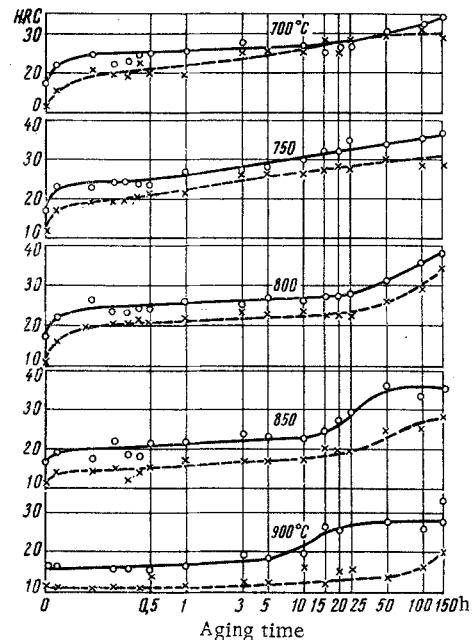


Fig. 6. Variation of hardness with aging time. The aging temperatures are given on the curves —) KhN50MBVYu; ---) KhN60MBVYu.

kg/mm<sup>2</sup>;  $\delta = 35-40\%$ ;  $a_n = 10-15 \text{ kg-m/cm}^2$ . Changes in quenching temperature have little effect on the mechanical properties of KhN60MBVYu, but a considerable influence on the properties of KhN50MBVYu. Reducing the quenching temperature from 1200 to 1100°C and aging at 750°C increases the ultimate and yield strengths by 20 kg/mm<sup>2</sup>, and the elongation drops from 32 to 20%.

The mechanical properties of the alloys at room temperature and at 600-1000°C were determined after quenching from 1100°C and aging at 750°C for 15 h. The properties were determined at 1050-1250°C after quenching (without aging), since heating in this temperature range would remove the effect of aging.

The strength and plasticity of the alloys are high (Fig. 8). The higher strength of the alloy containing iron is due to the intermetallic phase  $M_7W_6$ .

The KhN60MBVYu alloy has a distinct minimum on the specific elongation curve at 650-800°C, which is characteristic of most precipitation-hardening alloys. The elongation of alloy KhN50MBVYu increases continuously with the temperature up to 1000°C. The reduced plasticity of metals at elevated temperatures is usually associated with the development of intergranular deformation and cracks in the grain boundaries. Most theories link intergranular damage with slip and the means by which it occurs [4, 5]. Studies have shown [6] that the substantial reduction in the plasticity of the heat resistant alloy KhN80TBVYu at working

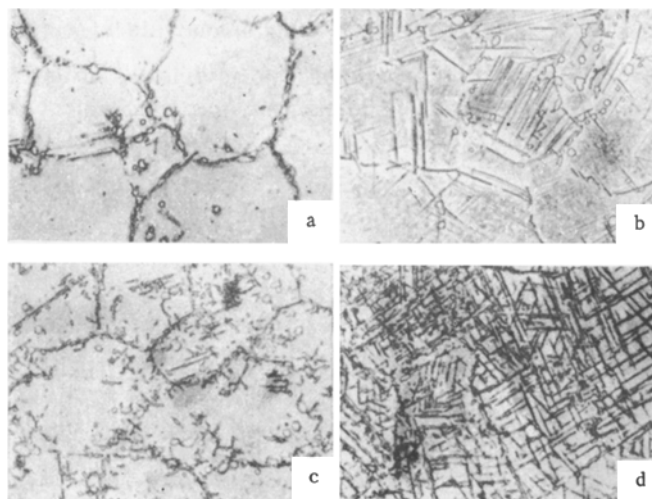


Fig. 7. Microstructure of alloys KhN60MBVYu (a, b) and KhN50MBVYu (c, d) after quenching from 1150°C and aging at 850°C for 15 h (a, c) and 50 h (b, d) ( $\times 500$ ).

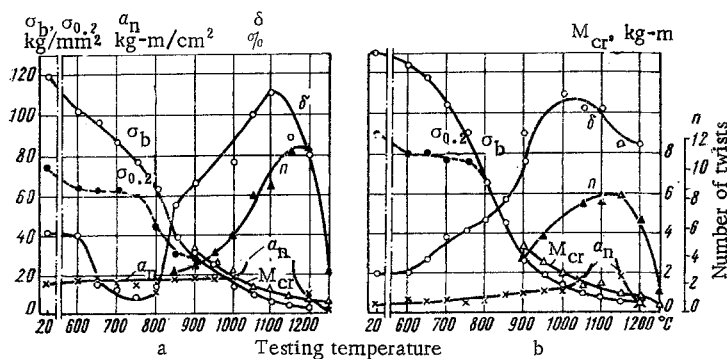


Fig. 8. Mechanical properties of alloys KhN60MBVYu (a) and KhN50MBVYu (b) at elevated temperatures.

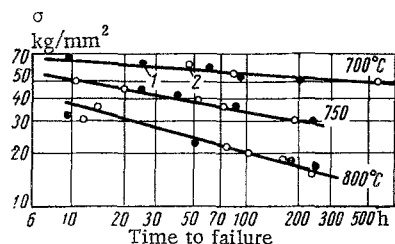


Fig. 9. Long-term strength of KhN50MBVYu (1) and KhN60MBVYu (2). The testing temperatures are given on the curves.

temperatures is due to the maximum development of intergranular slip. This is probably the reason for the minimum plasticity observed on the specific elongation curve for the KhN60MBVYu alloy, while the intergranular slip in the KhN50MBVYu alloy is considerably less. The reason for this must be sought in the structural condition of the grain boundaries, which differs in the two alloys. In the KhN60MBVYu alloy the grain boundaries are thin and rarely contain carbides, and a considerable portion of the boundaries is free of inclusions. The grain boundaries of the KhN50MBVYu alloy have a network of  $M_7W_6$  precipitates after quenching from 1100°C. These precipitates can serve as a substantial barrier to slip of the grains with respect to each other, thus increasing the strength of the boundaries, which leads to redistribution of the strain from the boundaries to the grain bodies. The process of failure is preceded by considerable plastic deformation of the grain bodies. In addition, the solid solution is impoverished in such elements as molybdenum and tungsten during formation of  $M_7W_6$ , which promotes additional solution of niobium in the solid solution and a reduction of the amount of hardening phase  $Ni_3Nb$ , with a consequent increase of plasticity.

The impact toughness of the KhN60MBVYu alloy is higher at all temperatures tested. The impact toughness of the KhN50MBVYu alloy at 20–800°C is relatively low ( $\sim 5$  kg-m/cm<sup>2</sup>), although the level is the same as that of most nickel heat-resisting alloys hardened with titanium and aluminum.

The plasticity of the alloys is high at 1000–1150°C, which permits hot working.

The long-term strength of the two alloys is the same (Fig. 9), i. e., the addition of 10% Fe does not reduce the long-term strength in tests up to 300 h.

Heat resistance tests of the alloys in air at 1000°C showed that they are oxidized less than alloy KhN67VMTYu, which is hardened with titanium and aluminum (see Table 1).

## CONCLUSIONS

1. Heat-resisting alloys KhN60MBVYu and KhN50MBBYu, hardened by precipitation of  $Ni_3Nb$ , have high mechanical properties at room and operating temperatures.
2. The addition of 10% Fe promotes the formation of  $M_7W_6$ , grain refining of the solid solution, and accelerates the transformation of the metastable  $Ni_3Nb$  phase with a fcc lattice into the equilibrium modification with an orthorhombic lattice.
3. The  $M_7W_6$  phase in the grain boundaries of the KhN50MBVYu alloy provides high plasticity at maximum hardening temperatures.

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