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Abstract

Full Text

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INVESTIGATION OF THE NIOBIUM–BERYLLIUM SYSTEM

(Presented by Academician A. A. Bochvar, 11 I 1963)

The niobium–beryllium system has been little studied. No phase diagram has been constructed; there are only isolated data on the existence of the phases $NbBe_{12}$ with a tetragonal structure of the $ThMn_{12}$ type and parameters: $a = 7.357$ kX, $c = 4.247$ kX, $c/a = 0.577$ ($^{1-3}$); Nb_2Be_{17} with a rhombohedral structure and parameters in hexagonal coordinates $a = 7.409$ Å, $c = 10.84$ Å; Nb_3Be_2 with a tetragonal structure and parameters $a = 6.49$ Å, $c = 3.35$ Å (^{5}), and $NbBe_2$ with a h.c.p. structure ($a = 6.53$ Å) (^{6}).

Fig. 1. Microstructure of diffusion layers obtained at a saturation temperature of 1100°C for 6 hours.

In the present work the results are given of an investigation of the niobium–beryllium system by the method of thermodiffusion alloying, in combination with microstructural, X-ray structural, X-ray spectral methods of analysis and measurement of microhardness. Niobium of 98.8% Nb purity, $H_\mu = 200\ \text{kg/mm}^2$, was used for the study. Thermodiffusion surface alloying of niobium specimens was carried out in a solid charge using beryllium of 99.8% purity, in the temperature range 900–1300°C with various holding times. In the process of interaction of beryllium with niobium at the indicated temperatures, a diffusion zone of complex structure and composition forms on the surface of the latter. The microstructure of the diffusion layers obtained at a saturation temperature of 1100° for 6 hours is shown in Fig. 1 (450×). A similar structure is also obtained at other saturation temperatures.

Diffusion proceeds frontally with the parallel formation of a series of layers of different thickness. The inner layer, directly adjacent to the core of the metal, has clearly delineated boundaries. X-ray structural studies found that this layer has a hexagonal structure with parameters $a = 4.516$, $c = 7.387$ kX. X-ray spectral

Fig. 2. Phase diagram of the niobium—beryllium system

Figure 2: Fig. 2. Phase diagram of the niobium—beryllium system

local chemical analysis of this phase, carried out at the Institute of Metallurgy, showed a chemical composition corresponding to the $NbBe_2$ phase. The second layer, adjacent to the $NbBe_2$ phase on the outside, has a greater thickness. According to X-ray spectral analysis, the composition of this layer corresponds to the compound $NbBe_5$. The microhardness of the phase is 1580 kg/mm^2. There is no information on this compound in the literature; it is a new compound in the niobium—beryllium system.

The next phase in the diffusion zone is the compound $NbBe_8$, which has a rhombohedral structure with parameters in hexagonal coordinates $a = 7.56$, $c = 10.73$ kX. The microhardness is about 1430 kg/mm^2. The outer light layer has the greatest thickness. According to X-ray spectral local chemical analysis, it corresponds to the compound $NbBe_{12}$. The X-ray pattern of the layer is well indexed on a body-centered tetragonal system, with parameters $a = 7.376$ and $c = 4.280$ kX. These data agree with the literature. The microhardness of the layer is 1200 kg/mm^2.

Thus, by studying the diffusion zone obtained at saturation temperatures of 900–1300° C, the presence of four compounds was established: $NbBe_{12}$, $NbBe_8$, $NbBe_5$, and $NbBe_2$. The phase with a tetragonal structure, the existence of which is indicated in work (5), is not detected.

Studying the kinetics of phase growth in the diffusion zone at constant temperature and variable duration of the process, it was found that phase growth proceeds according to a parabolic law. On the basis of the data obtained, approximate temperature dependences of the diffusion coefficients for $NbBe_{12}$ and $NbBe_8$ were calculated; they are expressed, respectively, by the equations:

$$D = 7.66 \times 10^{-4} \times$$

$$\times \exp(-32\,000/RT); \quad D = 5.7 \times$$

$$\times 10^{-9} \exp(-14\,740/RT).$$

Fig. 2. Phase diagram of the niobium—beryllium system

For a more detailed study of the niobium—beryllium system, alloys were melted from the above-mentioned starting materials. The alloys were prepared in an arc vacuum button furnace with a water-cooled hearth. In this case, four alloys corresponded to the compounds obtained by us in the diffusion zone. In order to distribute the elements uniformly, the ingots were remelted 5-6 times. The loss of beryllium in these alloys was 1-2 wt.%.

The alloys obtained were subjected to thermal, metallographic, and X-ray diffraction study. The melting temperature of the alloys was determined by the droplet method using an OP-48 optical pyrometer on cast specimens measuring $7 \times 5 \times 5$ mm. The pyrometer was calibrated using pure metals under the same conditions. The melting temperatures were determined as the average of 2–3 measurements. The accuracy of the temperature measurement was $±$50°. The temperature at which a 1 mm diameter hole made in the specimen by the electric-spark method was filled was taken as the melting temperature of the alloy.

Analysis of the cast specimens showed that the compounds $NbBe_{12}$, $NbBe_8$, and $NbBe_2$ melt with an open maximum at temperatures of 1645, 1750, and 2080°, respectively. The compound $NbBe_5$ forms by a peritectic reaction at 1820°. At a temperature of 1260° an eutectic is formed between the compound $NbBe_{12}$ and beryllium, its microhardness being ~260 kg/mm^2. At 1490° an eutectic is formed between $NbBe_8$ and $NbBe_{12}$, whose hardness is $H_\mu = 950$ kg/mm^2. The eutectic of the compounds $NbBe_5$ and $NbBe_8$ melts at temperature of 1675°. At 2000°, between the compound $NbBe_2$ and the niobium-based solid solution of beryllium, a eutectic is formed, the microhardness of which is equal to ~1079 kg/mm^2. The heats of formation of the indicated compounds were measured on cast specimens*. The data obtained are given in Table 1.

Table 1

Compound	$NbBe_{12}$	$NbBe_8$	$NbBe_5$	$NbBe_2$
Q, kcal/mol	29.8 ± 9.6	20.5 ± 3.2	46.4 ± 3.8	14.6 ± 1.9
Q, kcal/g-at	2.20 ± 0.74	2.28 ± 0.36	7.73 ± 0.63	4.87 ± 0.63

As a result of the investigations carried out, the phase diagram of the niobium—beryllium system was constructed (Fig. 2). It represents a system with limited solubility in the solid state, three eutectics, and four compounds, of which three melt with an open maximum, and one by a peritectic reaction.

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* With the participation of Yu. N. Surovyi.

Note: Figure translations are in progress. See original paper for figures.