

THE INFLUENCE OF BORON ON THE KINETICS OF PHASE FORMATION, THE DISLOCATION STRUCTURE, AND THE DIFFUSION PARAMETERS OF NICKEL

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The comparative characteristic of the processes of the kinetic formation of nickel borides, the dislocation structure and the state of interphase boundaries in nickel alloys obtained by the electrochemical method and unidirectional crystallization is carried out. The effect of boron on the diffusion parameters of micro- and macro-alloyed nickel alloys is definitely determined.

INTRODUCTION

At the present time widely used in the construction of rocket-space and nuclear power alloys are formed by transition metals, mainly, IV–VIA of the periodic system with interstitial phases (carbides, borides, oxides, nitrides) [1, 2]. The processes of structural changes in high-temperature alloys under the influence of high temperatures and stresses occur by mass transfer diffusion in the bulk, at the boundaries of phases and grains. Diffusion controls the physical and mechanical properties of materials, in particular, coarsening of the structure. One of the factors that determine the course of this process is the increased energy of the interphase surface, in comparison with the volume. However, the available experimental data on diffusion show that even special boundaries in eutectic (in-situ) compositions still differ from existing volumetric characteristics. The existence on the boundary of the defective areas phases of accumulation of linear clusters and point defects is stimulator for radiation-stimulated processes [3]. The diffusion of interstitial atoms in solids creates significant distortions of the crystal lattice, which, summing up in the region of the diffusion flux, cause the formation of a field of elastic stresses. It is important to study the effect of this field on the kinetics of phase formation, the diffusion coefficient, and the structural features of the diffusion zone. The formation of dislocations ahead of the diffusion fronts was observed in the zone of nickel diffusion into iron, upon saturation with a FeSe_{0.01}, etc. [4]. Of great interest is the mechanism of action on the boundary of the phases of rare-earth elements, surface-active impurities. However, such studies are very limited [5–7].

In the present study, boride nickel alloys obtained by solid-state (electrochemical) and liquid-phase directional crystallization are investigated. The choice of the nickel-boron system is due to the fact that on this basis constructional materials with the highest parameters of heat resistance and heat resistance were obtained [8–10].

In work [11], based on thermodynamic data, the possibility of calculating the equilibrium conditions and creating a wear-resistant Ni-Cr-Si-B coating obtained by directional crystallization was shown [the basis of the Ni-Ni₃B eutectic coating]. Such coatings are used to protect against corrosion and mechanical wear of heat power equipment components, increase short-term,

long-term and cyclic strength at temperatures up to 600...1200 K.

The problem of solid-phase coating of coating fibers can be used as a final technological operation, which allows to obtain finished parts of complex shape, for example, the nose of an airplane made of nickel hardened with boron (carbon) fibers. A method of electrochemical deposition of a dense, non-porous nickel layer on non-metals is known for manufacturing high-pressure chambers, a rocket engine [12]. As is known, eutectic compositions (in-situ composition) are characterized by structural stability up to pre-melting temperatures, Ni-W type alloys are used as parts of gas turbine engines [13].

The nickel used as the main component in modern structural materials is used in a number of cases of technical purity, contains low-melting impurities Bi, S, Pb, Sn, which, even at low concentrations (approximately 10⁻³ wt.%), drastically reduce ductility in the temperature range of -1000 K by 50% [8]. However, microalloying with chemically active elements [REM, boron] linking fusible impurities to hard melting compounds leads to refinement of the matrix and grain boundaries, increasing ductility. According to [14] alloying of nickel and alloy XH77TIOP ≈ 0.01%B increases the rate of boundary diffusion of tin and self-diffusion of nickel by 5...10 times, increases the activation energy and does not significantly affect the characteristics of bulk diffusion. Boron increases the ratio of Q_{gp}/Q_{sp} by 0.52 to 0.59. It should be noted that the lowering of the diffusion permeability of the boundaries of nickel grains during doping with boron correlates with the growth of the sublimation energy of nickel atoms from the boundaries (the heat of sublimation increases from 214 to 258 kJ/g atom) [5, 15]. Thus, boron in nickel alloys approximates the state of grain boundaries in thermodynamic and kinetic relations to the state of grain volume. However, the mechanism of influence on the properties of alloys of the morphology of boride phases, the nature of their location, the state of interphase boundaries (coherent, semi-coherent) is not clear.

The purpose of this work is to determine the kinetics of the formation of boride phases, the dislocation structures of the diffusion zone, the effect of boron and the structure of interface boundaries on the diffusion parameters of nickel alloys.

EXPERIMENTAL

Electrolytic nickel was used as starting materials, followed by vacuum remelting (10^{-6} Torr) with purity $>99.99\%$. According to micro-X-ray analysis, the content of each of the impurities of carbon, oxygen, aluminum, copper is less than 10^{-3} mas.%, tin $3 \cdot 10^{-4}$, lead 0.0002, bismuth 0.0003%, antimony, magnesium and manganese are not detected. The value of the residual resistance of pure nickel $R_{300\text{ K}}/R_{4.2\text{ K}} = 258$. Alloys of nickel containing 0.001, 0.003, 0.005, 0.01, and 3.8 wt.% boron (eutectic alloy) were prepared to study the structure of the alloys and the diffusion processes occurring in them. Alloy components were alloyed in a vacuum ($<10^{-6}$ Torr) or in an inert medium of purified helium (40 Torr pressure). The melted samples were cut from the ingot by an electric spark method, subjected to electropolishing in a solution of 60% sulfuric acid (current density $j = 0.1\text{ A/cm}^2$, $T = 300\text{ K}$) to remove the distorted surface layer. Metallographic, X-ray structural using a goniometer texture, as well as electron microscopic analysis (TEM) were carried out. The dislocation structure was determined from the etch pits (thermal method, 1600 K, 2 hours, vacuum 10^{-6} Torr) and electron microscopic method similarly [18].

Various boride coatings on nickel were obtained by electrochemical method by electrolysis from molten salts. The choice of technique is due to the fact that in various conditions, nonmetallic materials, for example boron fibers, can behave as conductors in electrolyte for the deposition of nickel and copper, and in the electrolyte for aluminum deposition, as non-conductive fibers [4]. Borage of nickel was carried out at 1173 K and cathode current density $j = 0.2\text{ A/cm}^2$ for 2, 5, 15, 30, 60, 180 min, followed by analysis of the diffractograms. The survey was conducted on copper CuK α -radiation from a flat section.

To study the diffusion parameters by the absorption method, the isotope nickel-63 (B-radiation, energy 0.067 keV) was deposited on the surface of samples of a given composition. The relative intensity of the hard component of radiation due to the radioactive isotopes Co-60, Co-58, Co-59 present in it was 2%. It is determined that the possible absorption of β radiation in the intrinsic layer on the surface of the samples is the minimum deposition of an isotope layer of $\sim 0.5\text{ }\mu\text{m}$ at a diffusion depth of $\sim 10\text{ }\mu\text{m}$. The thickness of the layer was controlled by a resonance method [16]. To eliminate the allowance for the correction for decay, the isotope layer was applied to control samples followed by diffusion annealing on a special built-in test bench [17].

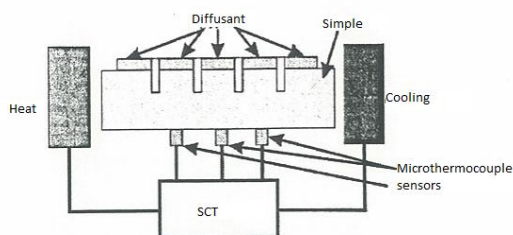


Fig. 1. Scheme of diffusion annealing with a temperature gradient [17]

At given modes of on-line spraying of the thickness of the deposited isotope film, the surface activity of the isotope along the entire length of the sample with accuracy to the statistical error is the same. According to (Fig. 1), the different temperatures of individual sections of the sample are the cause of the difference in the diffusion coefficient and the rates of diffusion processes in the local sample site. After diffusion annealing, the activity is repeated on the surface, the cooled end of the plate is used as a control, which is not subject to diffusion annealing. The received data on activity, after preliminary processing, are transferred to a personal computer for further analysis and output to the user program interface in a graphical (tabular) form.

RESULT AND DISCUSSION

The results of the analysis of boride coatings on nickel obtained by electrolysis are shown in Fig. 2. As can be seen, in 2 min of electrolysis a Ni_3B phase forms on the nickel surface. With increasing diffusion saturation time to 15 min (diffractogram 2), the Ni_3B phase concentration increases and embryos appear phase Ni_2B .

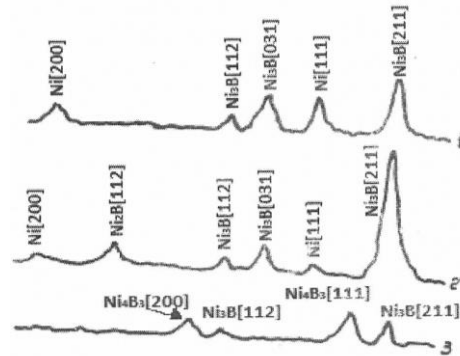


Fig. 2. Diffractograms of nickel samples subjected to electrolysis borings at $T = 1173\text{ K}$ for: 1 – 1 to 2 min; 2 – 15 min; 3 – 30 min

At an electrolysis time of 30 min or more, both low-boron and high-boron phases Ni_4B_3 and NiB are formed on the surface, which is confirmed by metallographic analysis data. In the electrolytic boronization of the first Ni_3B phase is formed, it is of interest to determine the interrelationships of the atomic structure of nickel boride with the shape of the crystallization center.

From simple geometric considerations it follows that, taking into account the size of interstitial atoms of boron and nickel and the presence of sufficient space in the lattice for their arrangement, it is possible to construct an elementary rhombic lattice (Fig. 3).

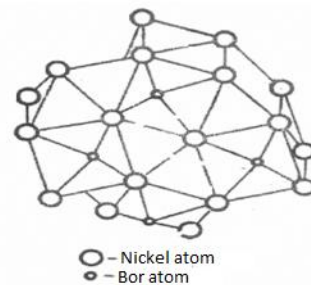


Fig. 3. The scheme of the elementary orthorhombic lattice of nickel

The structure of Ni_3B is identical to that of iron carbide, i.e. the boron atoms are located in the center of slightly distorted triangular prisms formed by 6 nickel atoms. In the nickel lattice, Me-Me distances are 0.249 nm. When the transition from the metal (Ni) to Ni_3B , the lattice becomes noticeably distorted due to the introduction of boron atoms (diameter 0.092 nm). An analysis of elementary orthorhombic cells shows that the lowest free energy corresponds to the hexagonal arrangement of atoms, i.e. the crystallization center of Ni_3B should have a hexagonal shape, which is confirmed by the data of metallographic and electron microscopic analysis (Fig. 4).

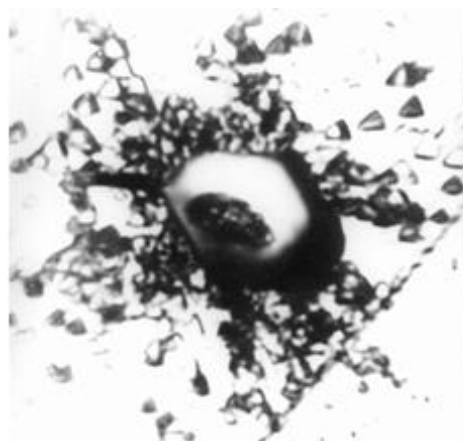


Fig. 4. Morphology of the boride phase Ni_3B , $\times 45000$

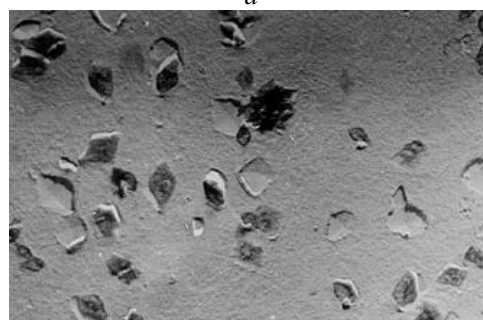
In the alloys of the eutectic composition Ni-3.8 wt.%B obtained directional crystallization, a similar pattern is observed. According to the entropy criterion of Hunt-Jackson under conditions close to equilibrium, substances whose entropy change at melting $\Delta S \leq 4 \text{ cal}/(\text{mol} \cdot \text{K})$ are prone to nucleation in the form of clearly faceted crystals, nickel boride has $\Delta S = 3 \text{ kcal}/(\text{mol} \cdot \text{K})$. Studies of the structure formation of the Ni- Ni_3B eutectic composition show that the leading crystallization of nickel boride grows as a plate in the $\langle 101 \rangle$ direction, crystallizes in a rhombic syngony with four modular units in a unit cell (the Pnma space group).

The dislocation structure of boron nickel was studied, the microstructure of the boride column polished perpendicular to the diffusion front is shown in Fig. 5.

As can be seen (see Fig. 5,a), dislocation clusters are formed under a solid boride layer, identified by triangular etch pits. The presence of such a geometry of etching pits indicates the emergence of such pits on the (111) plane. Such a cluster of dislocations was observed under a continuous diffusion layer. In a matrix located from the boride layer at a distance of 250...300 μm only separately located dislocations are found. Consequently, dislocation clusters are formed as a result of the diffusion of boron in nickel in the solid solution zone. Since the formation of misfit dislocations in this case possible, it should be assumed that they are formed due to joining two gratings – orthorhombic lattice Ni_3B nickel boride ($a = 0.5211 \text{ nm}$, $a = 0.6619 \text{ nm}$, $c = 0.4389 \text{ nm}$) and a cubic nickel. According to Fig. 5, the boride with a matrix has a rather significant interface, consisting of dislocation etch pits. It is established that the dislocation density increases in proportion to the square of the lattice parameter difference.



a



b

Fig. 5. Dislocation structure of boride alloys obtained by electrolysis, $\times 1750$

In the borides Ni_3B , Ni_2B , NiB , the locations of the dislocation exit and the etch pits of the tetrahedral shape have been identified (see Fig. 5,b). This shape of the etching pits indicates the dislocation output to the (100) plane. The location of the dislocation pits of etching in the above-mentioned phases is disposed along the dislocation pits of the etching chaotically.

Dislocations of the discrepancy were found in the eutectic composition Ni_3B , in which the minimum mismatch ($\approx 2\%$) of the lattice parameters of the Ni_3B conjugate phases was observed, similar in shape to those found in nickel boride alloys obtained by electrolysis (Fig. 6).



Ni_3B

Fig. 6. Dislocation disparities in nickel-boron alloys (shown by an arrow), $\times 50000$

It is determined that the interfacial surface energy due to the epitaxial dislocation network is $\approx 200 \text{ erg}/\text{cm}^2$, the distance between them is about 25 nm. It should be noted that a good correspondence between the interfacing phase gratings causes a long

high structural stability up to the pre-melting temperatures of the eutectic (in-situ) compositions.

The analysis shows that due to the small value of the atomic diameter of boron with respect to nickel atoms (see Fig. 3), the formation of a boron-vacancy complex plays an important role in the transport of boron to structural defects of the crystal lattice, as well as to grain boundaries and interphase boundaries. As indicated above, due to the ambiguous influence on the physical and mechanical properties of low boron concentrations and the structure of interphase boundaries in high-alloy alloys (composition Ni-3.8%B), the diffusion parameters of the ^{63}Ni isotope in such materials are determined by the absorption method [17].

It should be noted that the structure of the investigated samples was studied both as a result of preliminary diffusion annealing, and immediately after the experiment. It was found that in low-alloyed nickel alloys with boron (0.001; 0.003; 0.005; 0.01 wt.% B) the grain size during annealing is practically independent of the boron content and decreases by a factor of 2 compared to pure nickel, which is not significantly affects diffusion processes [5].

Tabl. 1 shows the mean values of the coefficients of self-diffusion in pure nickel and its slightly ligated alloys.

Table 1
Mean values of self-diffusion coefficients
in nickel-boron alloys

Self-diffusion coefficients in nickel-boron alloys, $D \times 10^{13} \text{ cm}^2/\text{s}$				
$T, \text{ K}$	Clear Ni (1)	Ni+0.001 B (2)	Ni+0.003B (3)	Ni+0.005B (4)
1125	0.39	0.23	0.27	0.46
1225	4.2	2.8	3.6	6.3
1325	38.1	26.3	45.7	66.1
1425	210	182	354	501

The temperature dependence of the coefficients of self-diffusion of nickel is determined, the graphical dependence is shown in Fig. 7

As can be seen (see Fig. 7), at boron concentration ≈ 0.002 wt.% the coefficients of self-diffusion in alloys are much less (especially at low temperatures) of the coefficients of self-diffusion of pure nickel. The presence of small concentrations of boron in pure and ultrapure nickel leads to a change in the mobility of the lattice atoms. At all annealing temperatures, minimal mobility is observed at a concentration of $\approx 0.001\%$ B max. In the future, with increasing boron concentration, the mobility of boron increases. At high temperatures ≈ 1400 K, the self-diffusion coefficient of nickel at boron concentrations above 0.05 wt.% changes insignificantly and its value tends to a value of $\approx 10 \dots 11 \text{ cm}^2/\text{s}$. The probable cause-the contribution of diffusion along dislocations and grain boundaries to the main diffusion flux at high temperatures becomes small and diffusion by volume becomes the main one. The latter is confirmed by the obtained data on the activation energies of self-diffusion in alloys with a maximum boron content of about 0.01% by weight. The activation energy of the self-diffusion of such an alloy coincides

with the activation energy of self-diffusion in pure nickel, where bulk self-diffusion gives the main contribution to the overall diffusion flux.

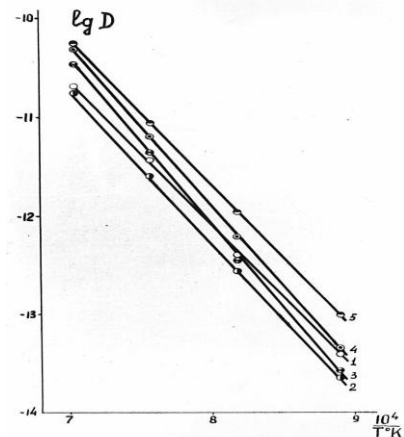


Fig. 7. Temperature dependence of self-diffusion coefficients in nickel alloys. Values 1, 2, 3, 4, 5 correspond to the alloy compositions given in Tabl. 1

As can be seen (see Fig. 7), at boron concentration ≈ 0.002 wt.% the coefficients of self-diffusion in alloys are much less (especially at low temperatures) of the coefficients of self-diffusion of pure nickel. Numerical values of the energy activation are 68700, 73400, 68500 cal/mol (respectively for allows 1, 3, 5).

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The observed growth (see Fig. 7, curve 3) in nickel-boron alloys is probably due to a significant decrease in the permeability of the boundaries due to the separation of boron along the grain boundaries, which is especially noticeable at low temperatures. The obtained results explain the available literature data on the influence of boron on the strength characteristics of nickel alloys. According to [9], boron not only increases creep and long-term strength, but also, to a certain extent, increases ductility.

Due to the small size of the atomic diameter of boron with respect to nickel atoms, the formation of an atomic-boron-vacancy complex (binding energy ~ 0.4 eV) plays an important role in the displacement of

boron atoms to structural defects of the crystal lattice, as well as grain boundaries and interphase boundaries.

To clarify the diffusion permeability of interface boundaries in boride nickel alloys, the data obtained for the eutectic composition Ni-Ni₃B at the crystallization rates $R = (0.3...2.07) \cdot 10^{-5}$ m/s are of interest. The composition has a perfect lamellar microstructure, consisting of a single-crystal nickel solid solution and nickel boride occupying approximately 78 vol.% (Fig. 8). Direction of crystallization parallel to $\langle 113 \rangle$ Ni₃B and $\langle 101 \rangle$ Ni. The phase conjugation plane of the $\{031\}$ Ni₃B phases is $0.166 \text{ at.}/\text{\AA}^2$, $\{101\}$ Ni $\approx 0.1632 \text{ at.}/\text{\AA}^2$, that is, the inconsistency on the semi-coherent phase surface is a minimum of about 2%.

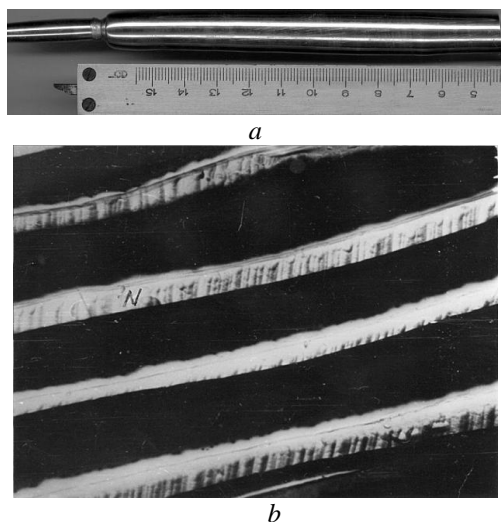


Fig. 8. Bimono-crystalline eutectic composition Ni₃B (a), microstructure of the composite, longitudinal section, $\times 750$ (b)

Specifically, for the plate composition along and across the growth axis, the activation energy is close (46 kcal/mol) and substantially higher (Fig. 9, Tabl. 2) of bulk diffusion in a single-crystal nickel matrix (≈ 56.36 kcal/mol) than in single crystals of nickel (69.97 kcal/mol). The observed anisotropy of the diffusion coefficient is due to the nonequivalence of the crystallographic directions of the interphase boundary plane.

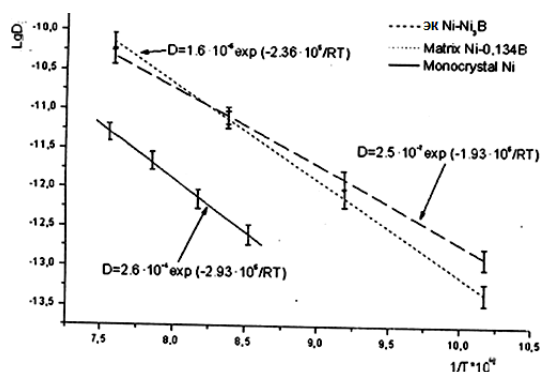


Fig. 9. The temperature dependence of the diffusion parameters in the eutectic composition Ni-Ni₃B

Table 2
Diffusion parameters in a eutectic composition

Material $\text{\AA K Ni-Ni}_3\text{B}$	The rate of crystallization, m/s	The diffusion coefficient $D_0, \text{cm}^2/\text{s}$	Activation energy $E, \text{kJ/mol}$ (kcal/mol)
EC _{II} – along the growth axis	10^{-3}	$7.3 \cdot 10^{-7}$	196 (46.805)
EC – isotropic growth structure	–	$1.1 \cdot 10^{-6}$	202 (48.238)
Ni + 0,134 B (matrix)	10^{-3}	$1.6 \cdot 10^{-5}$	236 (56.35)
EC ₊ – across the growth direction	10^{-3}	$2.5 \cdot 10^{-7}$	193 (46.08)
Ni monocrystal	10^{-3}	$2.6 \cdot 10^{-4}$	293 (69.97)

It is not excluded that this effect is due to the anisotropy of bulk diffusion in single-crystal Ni₃B plates having a rhombic lattice. Particular interest are data on the different diffusion permeability of the matrix solid solution and the composition oriented along the growth axis (see Fig. 9).

At low temperatures (less than 1200 K), preferential diffusion is observed over semi-coherent phase interfaces, less diffusion permeability of the solid solution of boron in nickel is due to the inhibitory effect of boron, which agrees with the data obtained above (see Fig. 7). At high temperatures ($T > 1200$ K), the reverse picture is observed because of the high thermal stability of the interphase boundaries in the regular lamellar composition.

It should be noted that the analysis of the ratios $E_{act}/R \cdot T_m = 16...20$ and the magnitude of the frequency factor $D_0 \approx 7.3 \cdot 10^{-7}...1.6 \cdot 10^{-5} \text{ cm}^2/\text{s}$ shows that in this case there is a vacancy diffusion mechanism.

CONCLUSION

The kinetics of the formation of boride phases in alloys obtained by various methods – electrolysis and directional crystallization – has been studied. The interrelation between the atomic structure of borides and the shape of the crystallization centers is established.

The dislocation structure of borides is determined, dislocation clusters are formed just below the continuous boride layer. In the matrix there are individual dislocations, which are formed as a result of diffusion of boron in nickel in the zone of solid solution. In boride alloys, obtained by liquid-phase and solid-phase methods, misfit dislocations are revealed on the semi-coherent interface. The ideal lattice mismatch ($\approx 2\%$) of the Ni-Ni₃B conjugate phases is found in a regular bimolecular composition.

The study of diffusion parameters in low-alloy nickel alloys shows that when small additions of boron are used to improve the strength characteristics of the alloys, it is likely that small concentrations of boron should be limited from 0.001 to 0.002% by weight. With a further increase in the boron concentration, its separation occurs along the boundaries, which worsens the ductility and strength. The activation energy of self-

diffusion has the highest value 73.4 kcal/(g·at.) at a boron concentration of 0.002%.

A temperature interval is determined in which predominantly bulk and interphase diffusion is observed. At low temperatures, a predominant diffusion along semi-coherent phase interfaces is observed in the plate-like composite (less than 1200 K), while the diffusion permeability of the solid solution of boron in nickel is explained by the inhibitory effect of boron. At high temperatures ($T > 1200$ K), an inverse picture is observed because of high thermal stability interphase boundaries in a regular lamellar composition. The small value of the atomic diameter of boron with respect to nickel atoms leads to the formation of a boron-vacancy complex, which plays an important role in the transport of boron to structural defects of the crystal lattice, as well as to grain boundaries and interphase boundaries.

The results obtained are important when small amounts of surface-active impurities are introduced into high-temperature materials and estimates of the high-temperature stability of the structure of eutectic composites.

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ВЛИЯНИЕ БОРА НА КИНЕТИКУ ОБРАЗОВАНИЯ ФАЗ, ДИСЛОКАЦИОННУЮ СТРУКТУРУ И ПАРАМЕТРЫ ДИФФУЗИИ НИКЕЛЯ

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Проведена сравнительная характеристика процессов кинетики образования боридов никеля, дислокационной структуры и состояния межфазных границ в никелевых сплавах, полученных электрохимическим методом и направленной кристаллизацией. Определено влияние бора на параметры диффузии микро- и макролегированных сплавов никеля.

ВПЛИВ БОРУ НА КІНЕТИКУ ФОРМУВАННЯ ФАЗ, ДИСЛОКАЦІЙНУ СТРУКТУРУ ТА ПАРАМЕТРИ ДИФУЗІЇ НІКЕЛЮ

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Проведена порівняльна характеристика процесів кінетики утворення боридів нікелю, дислокаційної структури і стану міжфазних границь у нікелевих сплавах, отриманих електрохімічним методом і спрямованою кристалізацією. Визначено вплив бору на параметри дифузії мікро- і макролегованих сплавів нікелю.