The effect of alloying elements on the interaction of boron carbide with aluminum melt

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Thermodynamic assessment of the influence of alloying elements (Si, Cu, Mg, Ni, Mn, and Zn) on the phase formation in cast aluminum matrix composites Al/B_4C is carried out. It is shown that doping with silicon promotes the primary crystallization of Al_8C_7Si phase, the region of existence of which expands with an increase in the silicon content. In the range of 700–900 °C, the addition of silicon slightly changes the B_4C fraction. The effect of copper is manifested in decrease in the solidus temperature and the solid-phase formation of Al_2Cu. Doping with magnesium changes the phase composition, contributing to the additional formation of the AlB_2 phase and free carbon in the four-phase region AlB_2 + B_4C + (Al) + C. The direct effect of zinc is recorded at its concentration of more than 0.7 wt.%, at which a solid solution (Zn) is formed in the solid state. The influence of manganese is fixed in solid-phase transformations; in the range of manganese concentrations up to 1 wt.%, crystallization ends in the $Al_{12}Mn$ + (Al) + B_4C phase region. Nickel addition works similarly; crystallization of alloys containing up to 1 wt.% Ni ends in the phase region Al_3Ni + (Al) + B_4C.

Key words: cast aluminum matrix composites, boron carbide, alloying elements, interaction thermodynamics, thermodynamic modeling.

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Introduction

The creation of advanced materials for structural and functional purposes with controllable properties is currently a key task in the development of such industries as the automotive industry, power engineering, aerospace and defense, nuclear industry, and many others. In recent years, there has been an increase in the attention of researchers to the development and research of aluminum matrix composites, the use of which makes it possible to achieve a significant increase in the level of physical, mechanical and operational properties and the expansion of temperature and power ranges of parts operation [1–3].

Aluminum matrix composites reinforced with particles of boron carbide are promising radiation-absorbing materials that are used in the nuclear industry in the manufacture of containers for storing and transporting nuclear fuel [4]. The high thermal conductivity and thermal stability of Al/B_4C composites provide significant advantages of dry storage facilities for spent nuclear fuel made from them, in particular, mixed oxide fuel and highly enriched fuel [5]. Al/B_4C composites are placed between spent fuel assemblies and provide not only radiation absorption, but also the structural strength of the fuel assemblies, as well as heat removal [6]. Effective absorption of fast and thermal neutrons by boron carbide is caused by the presence of the isotope ^{10}B [4]:

$$^{10}B + n \rightarrow {}^{7}Li + {}^{4}He + \gamma.$$

An increase in the fraction of boron carbide in the aluminum matrix composite is accompanied by an increase in the degree of protection against neutron radiation. Thus, an increase in the volume content of B_4C in alloy 6061 from 5 vol.% to 30 vol.% leads to an increase in the degree of screening of thermal neutrons from 50.9% to 95.6% [7]. Along with the radiation-absorbing effect, the high melting point, low density, high hardness and wear resistance of boron carbide make it an excellent reinforcing phase for aluminum matrix composites for structural and tribological purposes [8–10]. Many studies have confirmed that the degree of increase in the mechanical properties of aluminum alloys reinforced with B_4C particles is higher than reinforced with SiC particles, all other things being equal, which is due to better wettability and stronger adhesive bond in the Al/B_4C system [11–12].

Aluminum alloys of various grades can be used as a matrix material for the manufacture of Al/B_4C composites. It was previously shown that aluminum could interact chemically with boron carbide at temperatures

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above 700 °C with the formation of AlB_2 and Al_3BC phases [13–15]. Alloying elements in an aluminum alloy can have a significant effect on the nature of the interaction with boron-containing phases [16–18]. For example, alloying aluminum with titanium, zirconium and scandium promotes the formation of barrier layers at the interface with boron carbide, preventing its decomposition due to interaction with the aluminum melt [19]. However, information on the influence of the main alloying elements in aluminum alloys of standard composition on the interfacial interaction in the Al/B_4C system is presented in the literature rather limited. At the same time, understanding the processes of phase formation in this system in the presence of various alloying elements will make it possible to purposefully control the structure of cast aluminum matrix composites reinforced with boron carbide particles.

The aim of this work is a thermodynamic assessment of the influence of alloying elements (Si, Cu, Mg, Ni, Mn, and Zn) on the processes of phase formation in the Al/B_4C system.

Methods

Polythermal sections were constructed according to the CALPHAD methodology (CALculation of PHAse Diagrams) using the Thermo-Calc program and the TCAl4.0 database. The sections were created based on the concentration of elements and temperature. The general view of the section was a state diagram, which makes it possible to estimate the equilibrium phase composition when the temperature changes from the liquidus line to room temperature. In the case of complex phase transformations accompanied by a significant number of regions of phase equilibria, the region responsible for these transformations was enlarged and presented as a separate fragment of the diagram with characteristic temperatures and concentration of the alloying component.

Based on the stoichiometric B : C ratio in the B_4C compound, we selected the element concentrations of 4.03 wt.% B and 1.09 wt.% C, which, under the condition of suppressing the formation of the Al_4C_3 phase (Al_4C_3 = = SUSPENDED), ensure the stability of the 5 wt.% B_4C fraction. After selecting a number of elements of the Al – B – C – *X* system (where *X* = Si, Cu, Mg, Ni, Mn, Zn), the corresponding element contents were added, in particular, 4.03 wt.% B, 1.09 wt.% C, and 1.00 wt.% *X*. The last value was taken as the boundary concentration, which provides a general representation of the effect of the alloying element on the character of crystallization of the Al – 5 wt.% B_4C composite.

The calculation of the dependences of the mass fraction of phases and their composition on the content of the alloying element *X* (Si, Cu, Mg, Ni, Mn, Zn) and temperature in the range of 700–900 °C was carried out using the Thermo-Calc v.3.1 and database TCAl4.0 in the "Properties diagram" mode. Similar to the constructed polythermal sections, the calculations were performed with the suppression of the formation of the Al_4C_3 phase. After selecting the database and entering the content of the elements, a constant temperature was set, at which the calculation was performed. Then, a step of 0.02 wt.% was used, with which the change in the concentration of the alloying element was calculated. Thus, the specified amount of the alloying element was 0, 0.02, 0.04, 0.06 ... 0.98, and 1.00 wt.%. After the transition to the visualization area, the dependent parameters were selected, in particular, the amount of phase in wt.% and the amount of a component in the phase in wt.%. The iterations were carried out at temperatures of 700, 750, 800, 850, and 900 °C.

Results and discussion

Phase diagram of the Al – B – C ternary system allows one to estimate the critical temperatures and trace the formation of possible phases. With a given composition Al – 5 wt.% B_4C, it is assumed that the B_4C additive will be added into the aluminum melt in the form of a powder material, which does not imply an intense degradation of boron carbide particles with the formation of the Al_4C_3 phase. Dissolution of B_4C particles in liquid aluminum requires excessive overheating of the melt (above ~1500 °C), which is impossible in conventional foundry conditions. Both boron and carbon have extremely low solubility in aluminum (for example, for boron it is less than 0.025 at.% B at the eutectic temperature). The Al_4C_3 phase, in turn, has thermodynamically favorable conditions for the formation in the case of separate addition of B and C. In this case, the constructed polythermal section, taking into account the formation of aluminum carbide, indicates its primary crystallization at a low concentration of C (~0.25%) even with an excess of boron, in particular, at 4.03 wt.% (**Fig. 1**). At the same time, in the absence of boron, at 1.09 wt.% C, the interval of separation of the Al_4C_3 phase from the liquid exceeds 1000 °C, and with an increase in the boron content, this interval narrows significantly, for example, to 100 °C at 6 wt.% B, which is due to the following crystallization of B_4C. Summarizing, it should be noted that the formation of aluminum carbide seems to be undesirable due to a decrease in the proportion of the reinforcing phase B_4C and the formation of free boron, which binds to aluminum in the AlB_2 phase, significantly complicating the phase composition and character of crystallization. On the one hand, the temperature-time parameters of the liquid-phase combining of boron carbide reinforcing particles with an aluminum melt largely determine the composition of the interaction products at the interfacial borders. On the other hand, a change in the composition of interphase layers at the interfaces between the particles and the matrix is possible with the addition of various alloying elements.

The specific concentrations of 4.03 wt.% B and 1.09 wt.% C were selected based on the stoichiometric ratio B : C in B_4C compound. With the intentional elimination of the formation of the Al_4C_3 phase from the calculations performed, this amount of B and C should ensure the formation of ~5 wt.% B_4C practically in the entire crystallization range, which makes it possible to simulate the crystallization of the composite material, starting with a liquid solution (**Fig. 2**). In particular, the separation of this phase from the liquid begins at a temperature of 1570 °C with a monotonic increase in the mass fraction up to 2.3% at 1470 °C, up to 4.2% at 1270 °C and reaching ~ 5% at 1000 °C. Below this temperature, the value of the fraction of the phase changes slightly; in particular, only a small increase of it by 0.12 wt.% is noted.

Alloying with silicon significantly changes the composition of the interaction products. As shown by the polythermal section of the Al – B – C – Si system at 4.03 wt.% B and 1.09 wt.% C (**Fig. 3**), the addition of only 0.02 wt.% Si promotes the process of primary crystallization of the Al_8C_7Si phase. With a further increase in the Si content, the region of formation of this phase expands, which is accompanied, first of all, by an increase in the liquidus temperature of the entire system, in particular, to ~1650 °C at 1 wt.% Si, at an almost constant temperature of separation of the B_4C phase (1570 °C). Further transformations are associated with the separation from the AlB_2 phase at a constant temperature of 655 °C solidus at ~0.12–0.4 wt.% Si and the formation of a four-phase region $Al_8C_7Si + B_4C + AlB_2 + (Al)$ until the end of crystallization, at ~0.42 wt.% Si peritectic reaction $B_4C + L \rightarrow Al_8C_7Si + AlB_2 + (Al)$. It is important to note that with an increase in Si above 0.42 wt.%, the solidus temperature sharply decreases, reaching 620 °C at 1 wt.% Si. At more than 0.8 wt.% Si, the transformation $Al_8C_7Si + B_4C + (Al) + L \rightarrow Al_8C_7Si + AlB_2 + (Al) + L$ occurs. After these transformations, accompanied by the decomposition of B_4C, crystallization is accompanied by the formation of eutectic (Si) at temperatures below ~410 °C. In the technologically significant range of 700–900 °C, the addition of silicon slightly changes the fraction of the B_4C phase, in particular, at 1 wt.% Si, it decreases by 0.2 wt.% at 0.6 wt.% of the Al_8C_7Si phase, which is also accompanied by a change in the composition of the boron-containing phase by 2 wt.% in the direction of increasing B to 80% and decreasing C to 19 wt.%.

The effect of the addition of copper manifests itself much easier in comparison with the addition of silicon, since copper does not form intermediate phases with boron and carbon. At an almost constant liquidus temperature of 1570 °C, the separation of the B_4C phase occurs up to the line responsible for the onset of crystallization

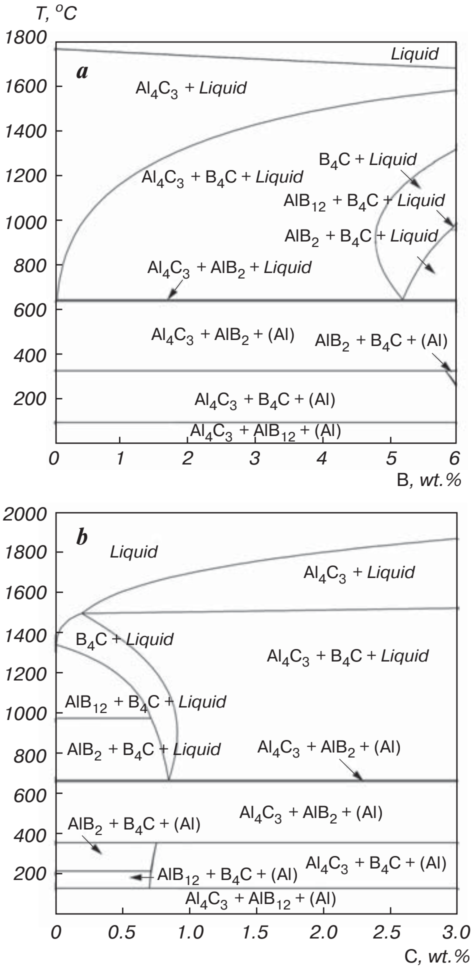

Fig. 1. Polythermal sections of the Al – B – C phase diagram at a constant carbon content of 1.09 wt.% (*a*) and boron of 4.03 wt.% (*b*) without suppressing the formation of the Al_4C_3 phase

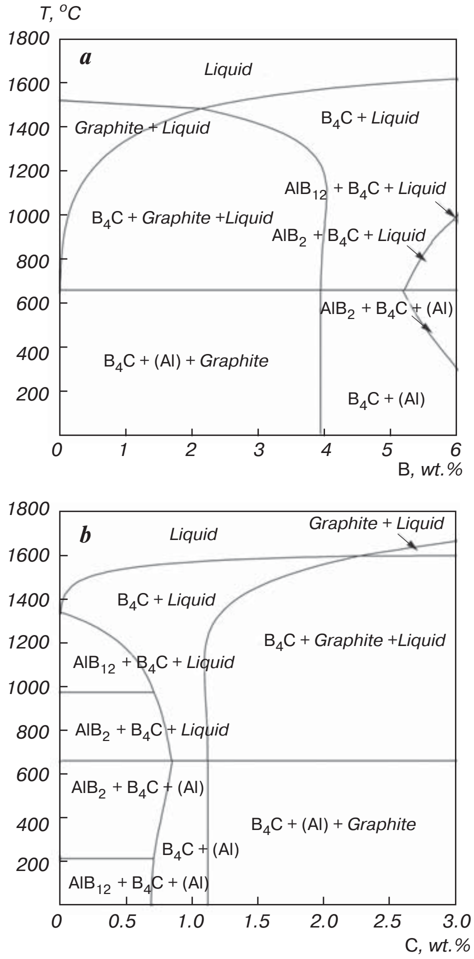

Fig. 2. Polythermal sections of the Al – B – C phase diagram at a constant carbon content of 1.09 wt.% (*a*) and boron of 4.03 wt.% (*b*) under the condition of suppressing the formation of the Al_4C_3 phase

of the solid solution (Al). This line, like the solidus line, decreases monotonically with an increase in Cu, which is associated with the occurrence of a eutectic reaction similar to that in the Al – Cu binary system. So the solidus temperature at 1 wt.% Cu is about 580 °C. With a decrease in temperature, the phases (Al) and B_4C are in equilibrium in the solid state together (**Fig. 4**). Solid-phase precipitation of the Al_2Cu phase occurs in the temperature range 225 °C (at 0.2 wt.% Cu) to 390 °C (at 1 wt.% Cu), which is also comparable to transformations in the binary system. In the temperature range 700–900 °C, the amount of B_4C in the liquid at 4.03 wt.% B and 1.09 wt.% C is practically unchanged and amounts to 5.0–5.1 wt.%. With an increase in the concentration of copper, its equivalent increase in a liquid solution occurs.

Similarly, it was found that with the addition of magnesium there is no change in the liquidus temperature, and the B_4C phase is formed up to the onset of crystallization of the solid solution (Al) at ~660 °C (**Fig. 5**). At more than 0.12 wt.% Mg, free carbon is formed in the four-phase region $AlB_2 + B_4C + (Al) + C$. It should be noted that at more than 0.7 wt.% Mg, the AlB_2 phase is formed earlier than the solid solution (Al), and an increase in concentration to 1 wt.% is accompanied by an expansion of the crystallization range of boron aluminide in the three-phase region $AlB_2 + B_4C + L$ approximately up to 80 °C. In the temperature range 700–900 °C, the

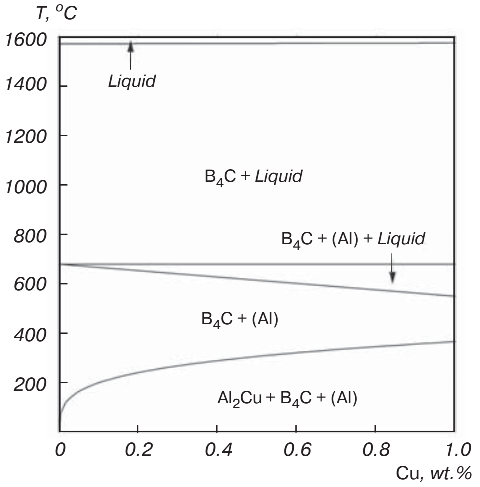

Fig. 4. Polythermal section of the phase diagram of Al–4.03 wt.% B –1.09 wt.% C – Cu in the range of copper concentrations from 0 to 1 wt.%

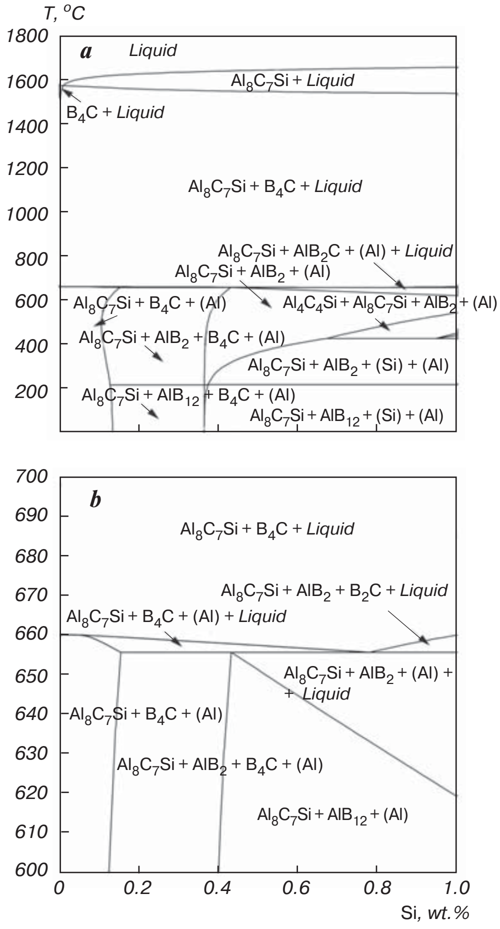

Fig. 3. Polythermal section of the phase diagram of Al–4.03 wt.% B –1.09 wt.% C – Si in the silicon concentration range from 0 to 1 wt.% (*a*) and its enlarged fragment in the temperature range 600–700 °C (*b*)

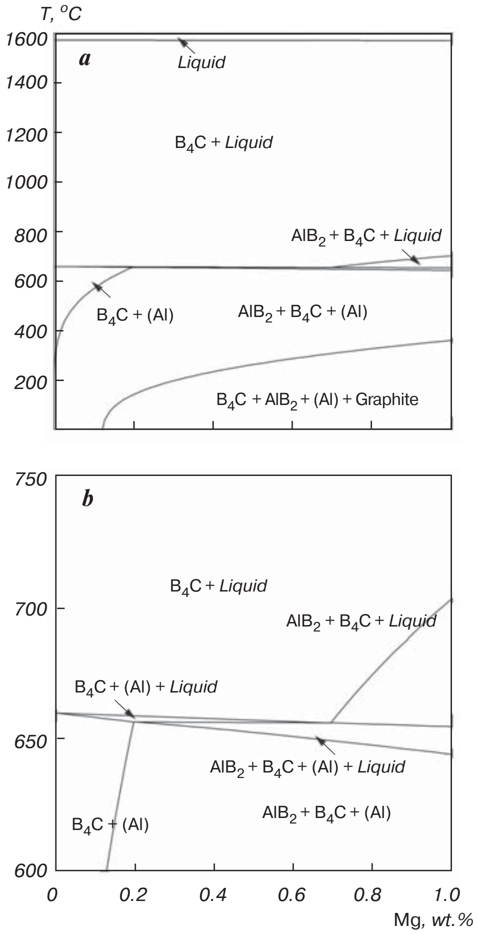

Fig. 5. Polythermal section of the phase diagram for Al – 4.03 wt.% B – 1.09 wt.% C – Mg in the range of magnesium concentrations from 0 to 1 wt.% (*a*) and its enlarged fragment in the temperature range 600–750 °C (*b*)

amount of B_4C in the liquid is practically unchanged and amounts to ~5.1 wt.%. Magnesium is predominantly found in liquid solution. At 700 °C and 0.97–1.00 wt.% Mg, it is also partially dissolved in the AlB_2 phase in an amount of more than 38 wt.%.

Zinc has slight effect on the phase composition of the Al – 4.03 wt% B – 1.09 wt% C alloy (**Fig. 6**). The temperatures of liquidus and solidus are practically constant at 1570 and 660 °C, respectively. When the latter is reached, a solid solution (Al) is released. The direct effect of zinc is recorded at its concentrations of more than 0.7 wt.% Zn and a temperature of ~10 °C, at which a solid solution (Zn) is formed in the solid state. In the temperature range of 700–900 °C, the amount of B_4C in the liquid is practically unchanged and amounts to 5.0–5.1 wt.%. An increase in zinc concentration is accompanied by its equivalent increase in a liquid solution.

At a constant temperature of liquidus (1570 °C) and solidus (660 °C), the effect of manganese on the phase composition of aluminum matrix composites Al – B_4C is fixed in solid-phase transformations (**Fig. 7**). In particular, at more than 0.4 wt.% Mn below the solidus line, the Mn_3B_4 phase precipitates, the homogeneity region of which decreases with increasing Mn in favor of the formation of the Al_6Mn phase. Moreover, the Mn_3B_4 phase dissolves at a constant temperature of 525 °C with the formation of the composition Al_6Mn + (Al) + B_4C. At 510 °C, the Al_6Mn phase is replaced by the $Al_{12}Mn$ phase. Thus, in the range of manganese concentrations up to 1 wt.%, crystallization ends in the $Al_{12}Mn$ + (Al) + B_4C phase region. In the temperature range of 700–900 °C, the amount of B_4C in the liquid is practically unchanged and amounts to 5.1 wt.%. With an increase in the concentration of Mn, its equivalent increase in the liquid solution occurs, respectively.

By analogy with manganese, at a constant temperature liquidus (1570 °C) and solidus (660 °C), the effect of nickel is fixed in solid-phase transformations (**Fig. 8**). At a concentration of up to 0.4 wt.% Ni, the Al_3Ni phase appears from the solid solution (Al), and at a higher content, it falls out of the liquid together with the solid solution (Al). Crystallization of alloys in the range of nickel concentrations up to 1 wt.% ends in the Al_3Ni + + (Al) + B_4C phase region. In the temperature range of 700–900 °C, the amount of B_4C in the liquid is practically unchanged and amounts to 5.1 wt.%. With an increase in the concentration of Ni, respectively, its equivalent increase in the liquid solution occurs.

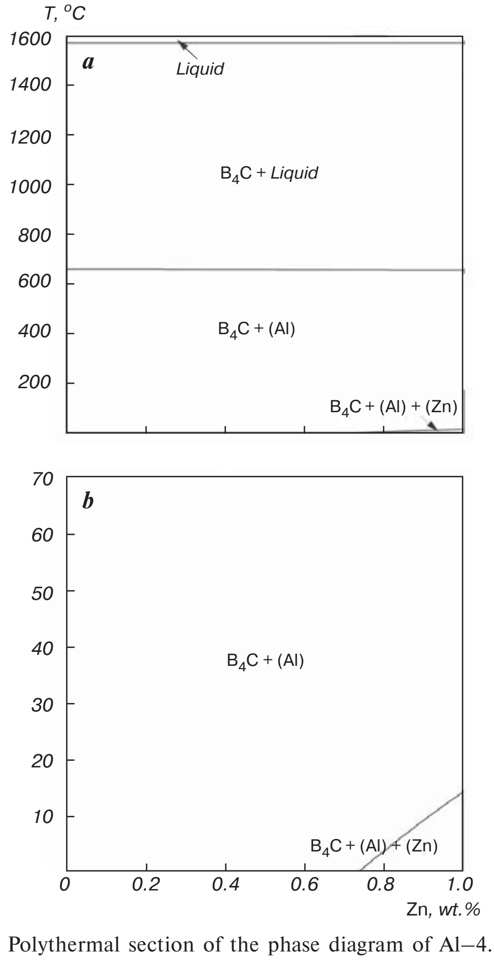

Fig. 6. Polythermal section of the phase diagram of Al–4.03 wt.% B –1.09 wt.% C – Zn in the range of zinc concentrations from 0 to 1 wt.% (*a*) and its enlarged fragment in the temperature range 0–70 °C (*b*)

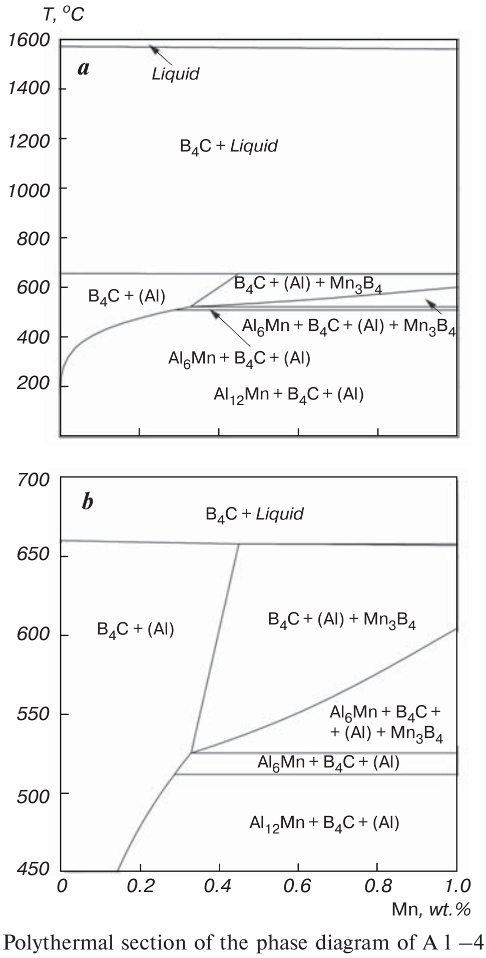

Fig. 7. Polythermal section of the phase diagram of A l –4.03 wt.% B – 1.09 wt.% C – Mn in the range of manganese concentrations from 0 to 1 wt.% (*a*) and its enlarged fragment in the temperature range 450–700 °C (*b*)

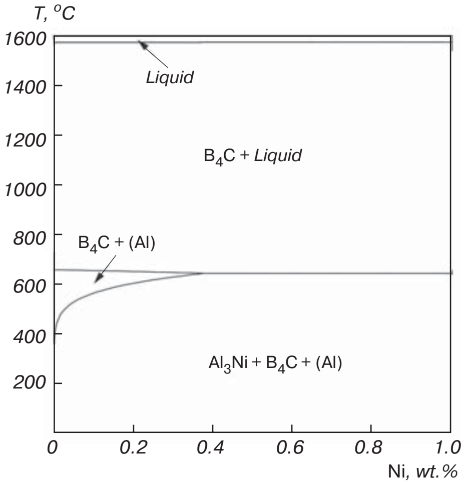

Fig. 8. Polythermal section of the phase diagram of Al – 4.03 wt.% B – 1.09 wt.% C – Ni in the range of nickel concentrations from 0 to 1 wt.%

The results of thermodynamic modeling of quaternary systems Al – B – C – X (where X is an alloying element) can be used in the selection of compositions and development of technologies for obtaining cast aluminum matrix composites with boron-containing phases with increased mechanical properties and functional characteristics. In particular, the data obtained make it possible to trace the possible composition of the interaction products at the interphase boundaries, as well as to assess the likelihood of intensification or suppression of the degradation processes of the exogenous reinforcing phase B_4C upon the addition of certain alloying elements. The application of the CALPHAD methodology for analyzing the interphase interactions of the components of aluminum matrix composites will help to reduce the development time for new materials, reduce energy, material and labor costs due to the rapid obtaining of sufficiently detailed and accurate information about the structure formation in multiphase systems.

Conclusion

Using the CALPHAD methodology, an assessment of the influence of alloying elements (Si, Cu, Mg, Ni, Mn, Zn) on the phase formation processes in the preparation of cast aluminum matrix composites Al/B_4C was carried out. It is shown that doping with silicon provokes the primary crystallization of the Al_8C_7Si phase, the region of existence of which expands with an increase in the silicon content in the alloy. In this case, in the technologically significant range of 700–900 °C, the addition of silicon slightly changes the fraction of the B_4C phase, in particular, at 1 wt.% Si, it decreases by 0.2 wt.% at 0.6 wt.% of the Al_8C_7Si phase. The effect of copper is manifested in decrease in the solidus temperature of the entire system and in the solid-phase formation of the Al_2Cu compound. Doping with magnesium changes the phase composition of the material, contributing to the additional formation of the AlB_2 phase and free carbon in the four-phase region AlB_2 + B_4C + (Al) + C. The direct effect of zinc is recorded at its concentration of more than 0.7 wt.% Zn and a temperature of ~10 °C, at which a solid solution (Zn) is formed in the solid state. The effect of manganese on the phase composition of Al – B_4C aluminum matrix composites is recorded in solid-phase transformations; in the range of manganese concentrations up to 1 wt.%, crystallization ends in the phase region $Al_{12}Mn$ + (Al) + B_4C. Nickel addition works similarly; crystallization of alloys in the range of nickel concentrations up to 1 wt.% ends in the phase region Al_3Ni + (Al) + B_4C.

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