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PRODUCING HIGH-TEMPERATURE TITANIUM ALLOYS OF Ti–Al–Zr–Si–Mo–Nb–Sn SYSTEM BY ELECTRON BEAM MELTING

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ABSTRACT

A complex of research work was performed on the base of EBM technology to produce high-strength complex titanium alloys of Ti–Al–Zr–Si–Mo–Nb–Sn system with silicon content, higher than the thermodynamically stable value in the solid solution. Alloys of this system are promising for creation of a new class of materials with a high level of heat-resistant characteristics. It is shown that EBM allows producing ingots of high-temperature titanium alloys of Ti–Al–Zr–Si–Mo–Nb–Sn system, which are characterized by sufficient chemical homogeneity and absence of casting defects. It is found that tin presence lowers silicon solubility in the test alloys and intensifies silicide release. Here the structure is also refined. It is found that additional alloying elements influence silicon solubility in titanium, forming complex silicides of $(Zr, Ti)_5Si$ $(Ti, Zr)_3Si$ type in the test alloys. It is shown that the microstructure of the test cast alloys consists of platelike α-phase packets within primary β-grains, having different crystallographic orientation.

KEYWORDS: high-temperature titanium alloy; ingot; electron beam melting; technological modes; chemical composition; cast metal; structure

INTRODUCTION

In the recent decades significant attention is given to development of alloys based on refractory and highly reactive metals. Today, nuclear power engineering, gas turbine construction, aeronautic and aviation engineering require light and strong materials, which can compliment a list of high-temperature alloys based on nickel, cobalt and iron that are traditionally used in these fields. High-temperature titanium-based alloys are one of the ways of solution of this problem. Complex titanium-based alloys are of particular interest. They have high specific strength, heat- and corrosion resistance properties in various media [1–3].

There are high requirements to the critical designation parts, which are constantly improved and become more rigid. This, first of all, relates to the quality of materials being used. Therefore, for the purpose of widespread use of titanium alloys in different structures it is necessary to develop new titanium-based materials with higher operation characteristics as well as improve production of semi-finished products of these alloys in future[4].

Any imperfections of chemical and structural homogeneity in titanium alloys result in decrease of strength and life of the products. Production of titanium alloys is related with the difficulties caused by high titanium sensitivity to interstitial impurities, in particular, oxygen, nitrogen, hydrogen and carbon and interaction with many chemical elements. This leads to creation of solid solutions or compounds. Moreover one of the main structural imperfections of titanium alloys is the presence of nonmetallic inclusions. High activity results in occurrence of physical-chemical processes of interaction with gases even in solid state. Therefore, non-metallic inclusions, in particular nitrides and oxides, can be generated in a process of ingots melting as well as at various stages of technological redistribution into finished products. Non-metallic inclusions can be introduced in the finished product from charge materials in the process of melting as well as in heat treatment of the finished product. Titanium actively interacts not only with gases, but also with other elements, including alloying components. Therefore, local enrichment of separate volumes of the ingots with alloying elements can result in formation of intermetallic inclusions, for example Ti_3Al, TiAl, TiCr and others [5].

WORK RELEVANCE

Operating temperatures of the modern commercial titanium alloys do not exceed 600 °C that limits their application [6]. Therefore, a problem of improvement of mechanical characteristics of titanium alloys at temperatures exceeding 600 °C is relevant for the moment and requires solution. One of the perspective directions for the problem solution is development of the titanium composites based on Ti–Al–Zr–Si–Mo–Nb–Sn system with silicon content exceeding thermodynamically stable value in solid solution. Such composites have a multiphase structure where strengthening of titanium matrix with refractory compound of Ti_5Si_3 takes place in a natural way in a process of solidification. The alloys of this system are perspective for development of a new class of materials with high level of heat-resistant characteristics [7–11].

Today not all the methods of titanium ingots manufacture allow obtaining quality metal and violation of a technological process of titanium alloy production results in detection in the ingots of defects reducing metal quality. The main factor affecting the material quality, in particular, ones having low technological plasticity, is a high quality of output ingot. An electron beam melting (EBM) can provide proper quality of obtained ingot with uniform, not coarse structure and good chemical homogeneity. The EBM allows a wide range regulation of melting rate due to independent heat source that in turn allows regulation of duration of metal staying in liquid state. The EBM is a technology that permits providing almost complete removal of refractory inclusions of high and low density. Thus, the EBM allows rising a quality of titanium alloy ingots.

Therefore, now mastering of the technology of melting of high-temperature complex titanium alloys of Ti–Al–Zr–Si–Mo–Nb–Sn system using EBM is a relevant problem.

MATERIALS AND INVESTIGATION METHODS

Most of the titanium alloys contain high amount of alloying elements that somewhat complicates their production using electron beam melting. Melting in vacuum promotes random evaporation of alloying elements with high vapor tension. So, melting of the ingots of complex titanium alloys using EBM provokes a problem of assurance of set chemical composition of an ingot [13]. In this case aluminium is referred to such elements. And concentration in the ingot of elements with vapor density lower than vapor density of titanium (Nb, Mo, Zr and Si) can even somewhat rise. PWI of the NASU has carried out the fundamental investigations of the processes of evaporation of components from a melt in vacuum [12]. They were used to calculate a predicted chemical composition of the ingots based on the results of which a calculation of weight of the charge ingot components was carried out. An alloying component with high density of vapor (Al) was charged taking into account compensation of evaporation losses. The charge ingot for melting of ingots of high-temperature complex titanium alloys of Ti–Al–Zr–Si–Mo–Nb–Sn system was formed in a nonconsumable bucket. Electron beam installation UE-208M (Figure 1) [14] was used for melts.

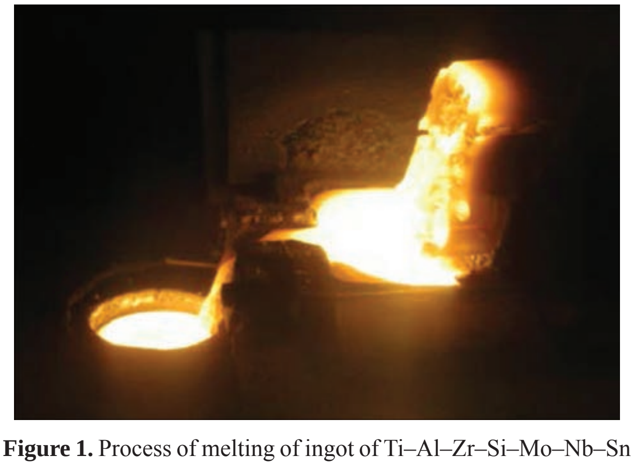

Figure 1. Process of melting of ingot of Ti–Al–Zr–Si–Mo–Nb–Sn system titanium alloy of 110 mm diameter in electron beam unit

To master the technology of melting of high-temperature complex titanium alloys of Ti–Al–Zr–Si–Mo–Nb–Sn system using EBM there were used earlier mastered technological schemes of charging of a consumable billet and melting modes [15].

Technological parameters of melting of Ti–Al–Zr–Si–Mo–Nb–Sn system ingots of 110 mm diameter

Melting rate, kg/h	30
Height of portion simultaneously poured in a mold, mm	10
Power, kW:	
in a mold	20
in an intermediate crucible	90

The composition of metal of the obtained ingots was determined using inductively-coupled plasma method in optical emission spectroscopy (ICP-OES) on ICP-spectrometer ICAP 6500 DUO. Content of oxygen and nitrogen was determined by a method of melting of analyzed sample of chips in vacuum in a graphite crucible, heating of which was performed using 900–1000 A current on RO-316, TN-144, RH-3 type devices of LECO Company (USA).

Quality of the ingot surface as for presence of pores, cavities, non-metallic inclusions, and cracks was determined visually without magnifying devices. An ultrasonic testing method was used to determine in the titanium ingots of internal defects in form of non-metallic inclusions, pores or inhomogeneities.

The structural investigations of metal of the obtained cast ingots were performed on the transverse templates, from which the samples for metallographic investigations were cut out. The sections of investigated alloys were made using the method of mechanical grinding with abrasive paper of different grain size and polishing of the samples by abrasive paste with chemical etching by a reagent of the following composition: 70 % H_2O, 25 % HNO_3, 5 % HF.

Figure 2. Ingots of Ti–Al–Zr–Si–Mo–Nb–Sn system titanium alloys of 110 mm diameter after EBM: *a* — 812-208 melt; *b* — 814-208 melt; *c* — 815-208 melt

The structure of alloys was examined using optical and electron microscopy methods. Metallographic investigations were carried out using optical microscope Jenaphot-2000 by the method of scanning electron microscopy on scanning microanalyzer JEOL Superprobe-733. Standard Vickers method was used for measurement of hardness at room temperature according to the State Standard 2999–75 at 30 kg loading.

INVESTIGATION RESULTS AND THEIR DISCUSSION

In order to provide high physicochemical characteristics of the experimental titanium alloys at room and elevated temperatures a complex alloying was performed for the purpose of hardening of solid solution by α-phase and dispersed particles of secondary phase, in particular silicides. Following the specification a series of melts was carried out (Figure 1) and cast ingots of high-temperature complex titanium alloys of Ti–Al–Zr–Si–Mo–Nb–Sn system of 110 mm diameter and 1000 mm length (Figure 2) with different content of alloying elements were obtained, namely 812-208 melt — Ti–5Al–5Zr–0.8Si–0.3Mo–0.1Nb alloy; 814-208 melt — Ti–6.5Al–5Zr–0.5Si–1.5Sn–0.1Nb alloy; 815-208 melt — Ti–6.5Al–5.5Zr–0.8Si–0.6Mo–0.5Nb–1.7Sn alloy.

A side surface of the obtained cast ingots after cooling in vacuum to temperature below 300 °C is clean, increased concentration of impurity elements on the surface in form of oxidized or alpha layer is absent. Depth of the surface defects of corrugation type makes 2–3 mm, defects in forms of tears, cracks or lack of fusion are absent.

In order to evaluate quality of metal of the obtained ingots there were carried out the investigations of chemical composition of the samples taken on ingot length from upper, middle and lower parts. The results of analysis of chemical composition of metal of the obtained ingots showed that the distribution of alloying elements on ingot length is uniform.

As a result mechanical processing of the side surface was performed and upper and bottom parts of the ingots were cut (Figure 3). The templates were cut out for performance of metallographic investigations and production of the samples for mechanical properties testing (Figure 4).

Figure 3. Appearance of ingots of Ti–Al–Zr–Si–Mo–Nb–Sn system titanium alloys of 110 mm diameter after side surface machining: *a* — 812-208 melt; *b* — 814-208 melt; *c* — 815-208 melt

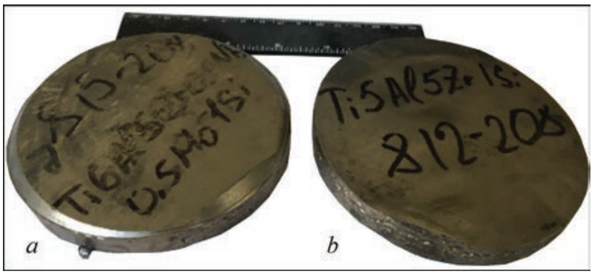

Figure 4. Cut templates of ingots of 815-208 (*a*) and 812-208 (*b*) melts for samples manufacture

Melted alloys corresponding to their composition can be referred to pseudo-α-alloys: they consist of α-phase and small amount of residual β-phase, the structure also contains silicides (Figures 5, 6).

Microstructure of the investigated cast alloys represents itself the batches of lamellar α-phase in the limits of primary β-grains that have different crystallographic orientation. Formation of the structure takes place in the process of cooling, namely internal volume of β-grains is filled with chaotic α-plates as well as collected in the batches (α-colonies), that indicates inhomogeneity of the cast structure. The intermetallics start to segregate first of all on structural defects, boundaries of grains and α-plates stimulating their growth. Primary silicides and silicide layers are formed at decrease of silicon solubility in β-phase according to the diagram of phase equilibrium of Ti–Si system as a result of liquation processes at ingot solidification (Figure 7) [16].

Fine silicides at the boundaries of α-plates are formed in the process of eutectoid transformation and further decrease of silicon solubility in the titanium

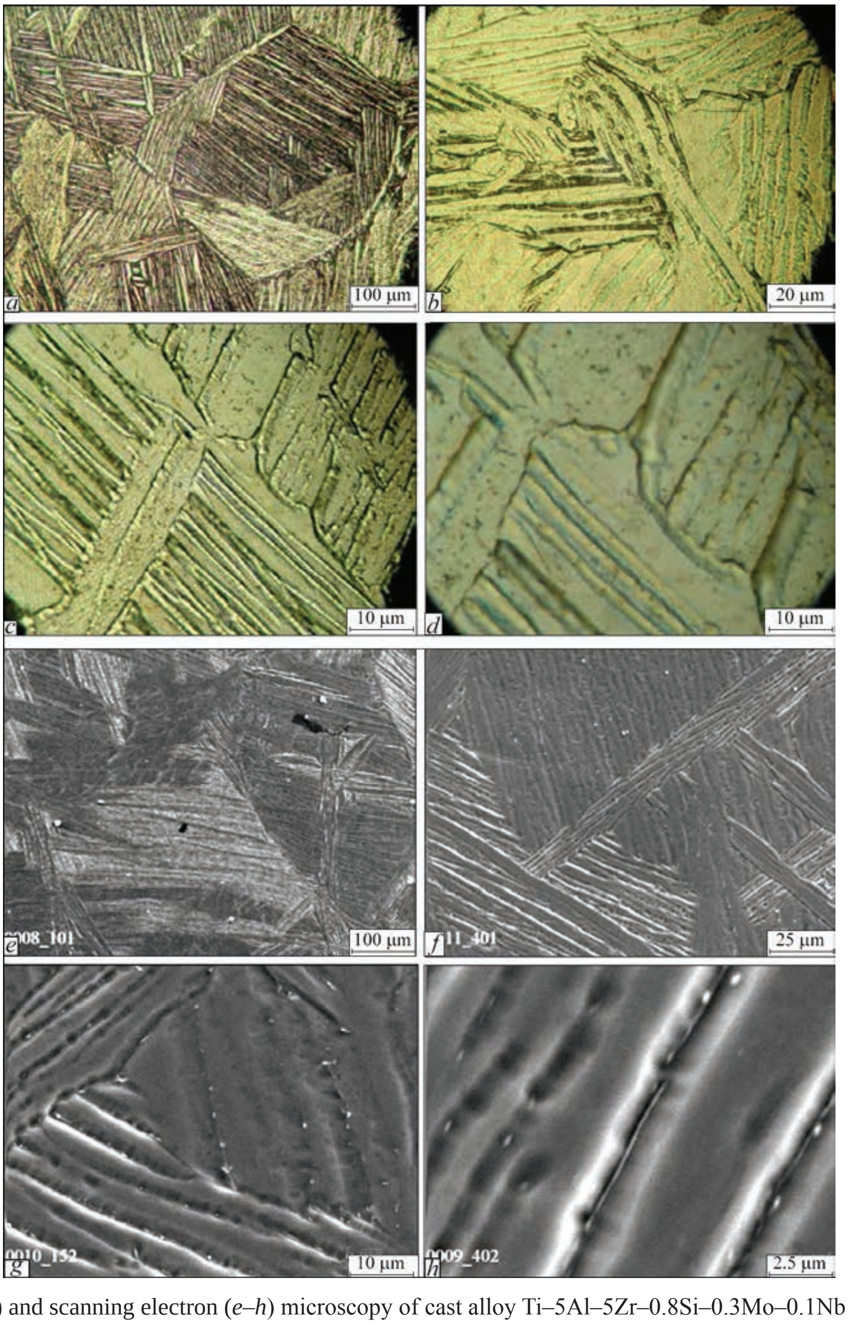

Figure 5. Optical (*a*–*d*) and scanning electron (*e*–*h*) microscopy of cast alloy Ti–5Al–5Zr–0.8Si–0.3Mo–0.1Nb (melt 812-208)

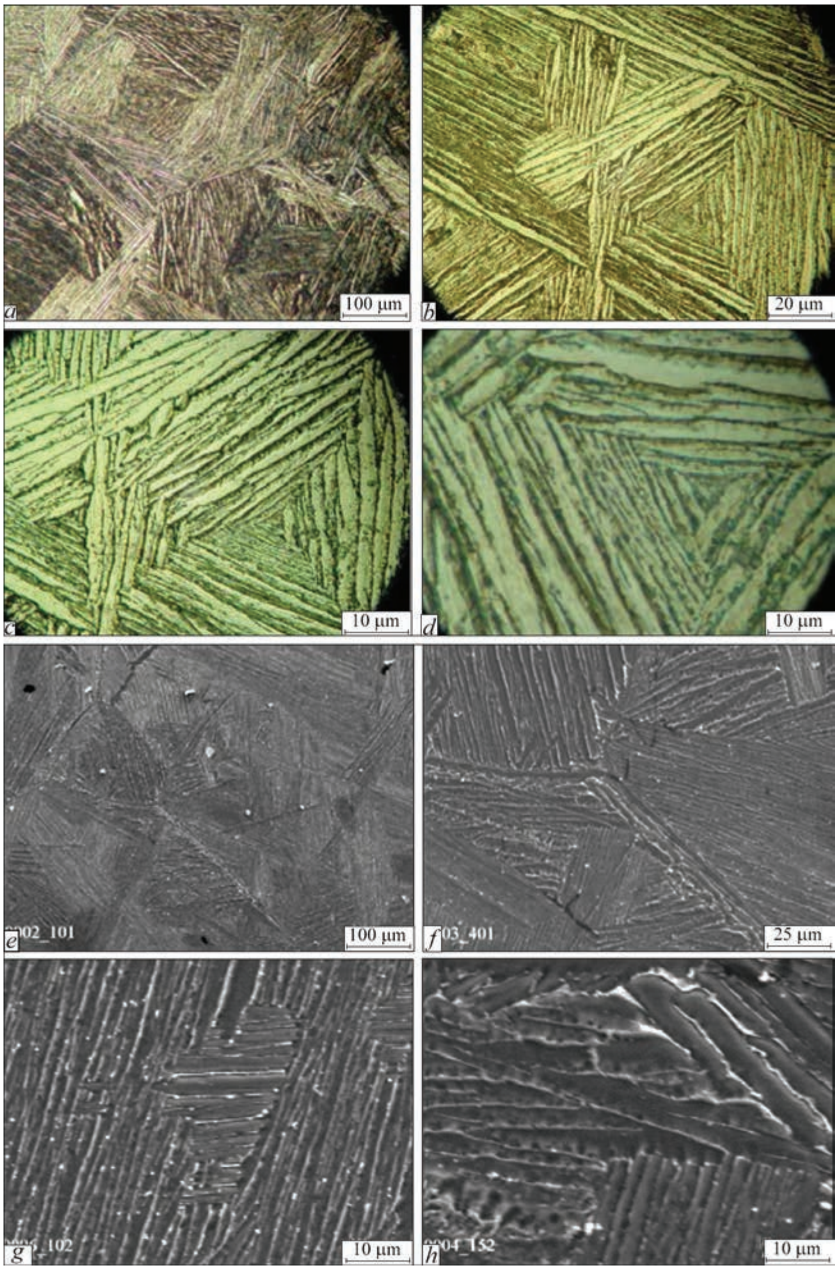

Figure 6. Optical (*a–d*) and scanning electron (*e–h*) microscopy of cast alloy Ti–6.5Al–5.5Zr–0.8Si–0.6Mo–0.5Nb–1.7Sn (melt 815-208)

α-matrix. Figure 8 shows a distribution of the main alloying elements in the cast alloy of 812-208 melt. It can be seen that except of titanium the silicides and their interlayers at the grain boundaries also contain zirconium, i.e. in the investigated alloys there is formation of the complex silicides of $(Zr, Ti)_5Si_3$ and $(Ti, Zr)_3Si$ type, which were earlier found in Ti–Zr–Si–Nb system alloys [17]. Zirconium and silicon are in solid solution as well as in hardening silicide phase that is distributed along the boundaries of former β-grains and α-plates. Thus, alloying elements have significant effect on silicon solubility in titanium.

Aluminium has uniform enough distribution in the structure. It is virtually completely located in the solid solution, almost absent at the boundaries of grains and

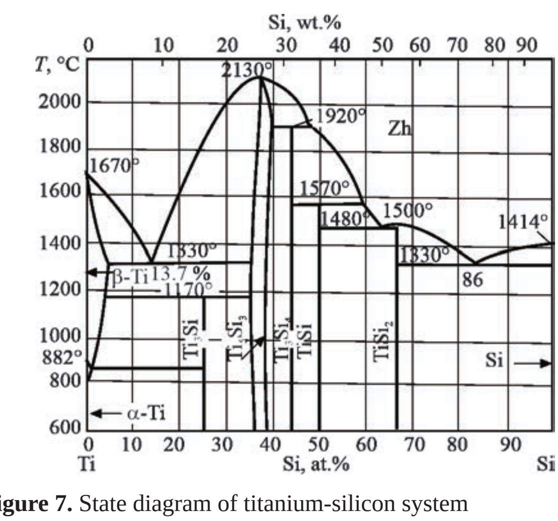

Figure 7. State diagram of titanium-silicon system

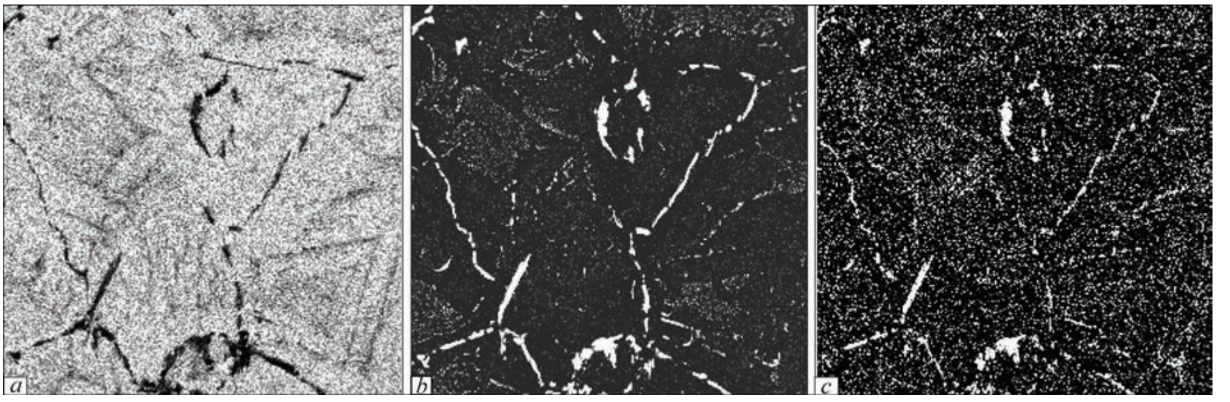

Figure 8. Distribution of doping elements (*a* — Al; *b* — Si; *c* — Zr) in cast alloy Ti–5Al–5Zr–0.8Si–0.3Mo–0.1Nb (815-208 melt)

present in smaller quantity sometimes in the spaces of α-plate batches in the places, where, probably, residual β-phase is present. The size of grains and plates of α-phase depends on silicon content as well as additional alloying in investigated cast titanium alloys. For quantitative evaluation of the lamellar structure the following parameters are used *D* (β-grain size), *d* (size of α-plates colonies) and *b* (width of α-plates). The average size of the grains of cast alloys makes approximately 300–500 μm, alloy 815-208 has finer structure elements (α-plates and their colonies) in comparison with alloy 812-208. Thus, in alloy 812-208 the width of α-plates makes 3–5 μm and in alloy 815-208 it is 2-3 μm (see Figures 5, 6).

In the structure of alloy 812-208 the amount of silicides on the boundaries of former β-grains and between α-plates is smaller than in alloy 815-208. Composition of the latter differs by additional content of tin, which is often used for titanium alloys doping for the purpose of increase of their heat resistance. Addition of tin promotes slowdown of processes of redistribution of the alloying elements between α- and β-phases that rises thermal stability of high-temperature alloys [18].

CONCLUSIONS

1. Performed set of works showed that the electron beam melting allows obtaining metal of ingots of high-temperature Ti–Al–Zr–Si–Mo–Nb–Sn system titanium alloys being characterized with sufficient chemical homogeneity and absence of defects of cast origin (pores, cavities, inclusions of low and high density).

2. It is shown that microstructure of examined cast alloys represent itself the batches of lamellar α-phase in the limits of primary β-grains that have different crystallographic orientation.

3. It is determined that additional alloying elements have significant effect on silicon solubility in titanium. They form complex silicides of $(Zr, Ti)_5Si_3$ and $(Ti, Zr)_3Si$ type in the investigated alloys.

4. It is shown that presence of tin reduces solubility of silicon in the investigated alloys and therefore increases precipitation of silicides, at that the structure is also refined.

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CONFLICT OF INTEREST

The Authors declare no conflict of interest

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