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Influence of various titanium-containing additives on the modification efficiency of aluminum–silicon eutectic alloy

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Abstract: This study investigates the impact of titanium addition to the eutectic silumin AK12 melt, considering various methods of addition. The research results encompass the sole introduction of titanium (at a calculated amount of 0.1 wt.%) through different forms/methods, such as the Al–4%Ti ligature, TiO_2 oxide, K_2TiF_6 salt, and Ti sponge. Additionally, the study explores the combined addition of titanium and a standard flux (comprising 62.5 % NaCl + 12.5 % KCl + 25 % NaF). The research involved qualitative and quantitative analyses of macro- and microstructures, spectral analysis data, and mechanical properties (tensile strength and relative elongation) of the alloys. The findings highlight that titanium has a positive influence on the structure of eutectic silumin, with the most effective results achieved when combined with the standard flux. However, the efficiency of silumin modification with titanium varies depending on the method of addition. Specifically, the introduction of titanium in the form of K_2TiF_6 fluoride salt, Al–4%Ti ligature, and titanium sponge positively affected macro grain refinement, reduced the spacing between the secondary dendrite arms of the solid solution (α-Al), and enhanced the dispersion of eutectic silicon. The most promising approach for complex silumin modification involves the joint introduction of titanium-containing substances and a sodium salt-based flux. This combination has a multifaceted impact on the silumin structure, leading to the simultaneous modification of various structural components in aluminum–silicon alloys. Depending on the type of titanium-containing substance, when processed alongside flux, the alloy achieves a relative elongation ranging from 9.7 % to 11.1 %, exceeding the same parameter for the unmodified alloy by more than 4 times and surpassing the sodium-modified alloy's relative elongation by 17–37 %. Furthermore, the ultimate strength reaches levels of 171–193 MPa, representing a 22–38 % improvement compared to the unmodified alloy and a 7–21 % increase compared to the sodium-modified alloy.

Keywords: cast aluminum alloys, titanium, sodium, complex modification, eutectic silicon, solid solution, macro- and microstructure, spectral analysis.

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Влияние различных титансодержащих добавок на эффективность модифицирования эвтектического сплава системы алюминий–кремний

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Аннотация: Изучено влияние титана, в зависимости от способа его ввода в расплав, на структуру и механические свойства эвтектического силумина АК12. Приведены результаты исследований как при одиночном введении титана (расчетное содержание Ti – 0,1 мас.%) различными способами (лигатурой Al–4%Ti, оксидом TiO_2, солью K_2TiF_6, Ti-губкой), так и при совместных добавках титана и стандартного флюса (62,5 % NaCl + 12,5 % KCl + 25 % NaF). Исследования осуществляли путем качественного и количественного анализа макро- и микроструктур сплавов, данных спектрального анализа и механических свойств (предела прочности на разрыв и относительного удлинения). Установлено, что титан оказывает влияние на структуру эвтектического силумина и наиболее эффективен при совместном введении со стандартным флюсом. При этом эффективность модифицирования силуминов титаном зависит от способа его ввода в расплав. Отмечено положительное влияние титана, введенного с помощью фтористой соли K_2TiF_6, лигатуры Al–4%Ti и титановой губки, на измельчение макрозерна, уменьшение расстояния между ветвями дендритов второго порядка твердого раствора (α-Al), а также на диспергирование эвтектического кремния. Наиболее перспективным способом комплексного модифицирования силуминов является совместное введение титансодержащих веществ и флюса на основе солей натрия. Такие составы оказывают комплексное влияние на структуру силумина, заключающееся в одновременном модифицировании различных структурных составляющих алюминиево-кремниевых сплавов. В зависимости от вида титансодержащего вещества при совместной обработке с флюсом относительное удлинение сплава достигает 9,7–11,1 %, что более чем в 4 раза превышает этот показатель для немодифицированного сплава и на 17–37 % выше, чем у сплава, модифицированного натрием. Предел прочности составляет 171–193 МПа, что на 22–38 % больше, чем у немодифицированного сплава, и на 7–21 % выше по сравнению со сплавом, модифицированным натрием.

Ключевые слова: литейные алюминиевые сплавы, титан, натрий, комплексное модифицирование, эвтектический кремний, твердый раствор, макро- и микроструктура, спектральный анализ.

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Introduction

Aluminum—silicon alloys find widespread applications in various industries such as aircraft and automotive due to their suitability for producing intricate castings. Their excellent casting properties enable the creation of complex, thin-walled, and impermeable castings. However, a disadvantage of these silumin alloys is their relatively low mechanical properties in sand casting, primarily attributed to the development of a coarse microstructure in the alloy. It is worth noting that Al-Si cast alloys are typically used for sand casting only in a modified form [1; 2].

In order to enhance modification, surfactants like sodium and strontium are introduced into the melt of pre-eutectic and eutectic silumins [1; 2]. These modifying additives have the effect of refining eutectic silicon particles, causing them to adopt a globular shape during crystallization. Consequently, the mechanical properties of the alloy, especially its relative elongation, improve.

For refining the grains in the solid aluminum-based (α-Al) solution, crystal-nucleating elements such as Ti, Zr, and Sc are added to the melt of deformable and foundry aluminum alloys, including silumins [1; 3; 4]. Notably, the efficiency of grain refinement in deformable alloys differs from that in pre-eutectic silumins. This discrepancy is due to the known fact that increased silicon content in the alloy reduces the degree of grain refinement [5].

Among the various modifiers for α-Al in aluminum-based alloys, titanium is recognized as one of the most effective, particularly for pre-eutectic alloys with silicon content less than 7 % [1]. When introduced into the melt in the range of 0.05—0.15 %, titanium leads to the formation of additional $TiAl_3$ crystallization centers resulting in the refinement of α-Al solid solution grains and significant improvements in the casting and mechanical properties of the alloys [1; 6].

However, the existing scientific and technical literature provides contradictory information regarding the modifying impact of titanium (up to 0.2 wt.%) on pre-eutectic (containing more than 7 % Si) and eutectic silumins. It is generally believed that the modification of such alloys with titanium should not yield significant structural refinement, as titanium primarily modifies α-Al dendrites, while the key structural component determining the complex mechanical properties of silumins is the silicon eutectic [7; 8].

Moreover, as demonstrated in [3; 9—11], a substantial amount of silicon (7—13 wt.%) diminishes the grain refinement efficacy of titanium-modified casting Al—Si alloys. This phenomenon can be explained by the formation and growth of TiAlSi intermetallides, such as $(Al,Si)_3Ti$ and $(Al,Si)_2Ti$, in the melt. Consequently, the number of $TiAl_3$ particles, which act as crystallization centers, decreases.

Nonetheless, there is a discernible beneficial impact of titanium on the structure of both pre- and eutectic silumins, as documented in [12—17]. For instance, in [12], the influence of titanium introduced through the Al—5Ti alloy on the structure and mechanical properties of the Al—10%Si alloy was investigated. The study revealed that the addition of 0.5 wt.% Al—5Ti contributes to the refinement of dendrites (α-Al) and leads to a maximum increase in the solid solution fraction across thin sections. Consequently, the tensile strength and relative elongation of the alloy increase by 9 % and 49 %, respectively, compared to the unmodified alloy.

In [13; 14], the favorable impact of titanium introduced via Al—10%Ti and Al—5%Ti—1%B alloys, combined with strontium, on grain (α-Al) refinement in the Al—7%Si—Mg alloy was demonstrated. Comprehensive treatment of the melt with titanium and strontium amplifies the degree of refinement of eutectic silicon particles compared to using Sr alone. As described in [2], this effect arises because the silicon eutectic undergoes a modified transformation; being a solidified phase, it crystallizes within the restricted space between dendrite arms, and the spacing between them decreases under the influence of the modifier (titanium).

Titanium also synergizes effectively with eutectic silicon modifiers like sodium [17], strontium [13; 14; 18], barium [19; 20], and more. Consequently, titanium-containing additives, such as the K_2TiF_6 salt and titanium dioxide TiO_2 are components of certain complex modifying fluxes for silumins, exerting influence on various structural components of the alloys [19; 20].

The objective of the research was to examine the impact of titanium on the structure and properties of eutectic silumin, contingent on the method of its introduction into the melt. Various introduction methods were studied, including the use of the Al—4%Ti alloy; titanium sponge; K_2TiF_6 salt; and titanium dioxide TiO_2. Additionally, we investigated the effects of titanium-containing additives when introduced in combination with a standard flux for modifying the eutectic in silumins with the following composition, %: 62.5NaCl + 12.5KCl + + 25NaF.

Materials and methods of research

Silumin of eutectic composition AK12, whose chemical composition was as follows (wt.%) according to the spectral analysis data, was chosen as a model alloy:

Al	Base	Ti	0.0031
Si	11.53	Zn	0.0092
Cu	0.0021	Mo	0.0026
Mg	0.0006	Fe	0.358
Mn	0.0026		

Titanium-containing additives were utilized for the modification of silumins. These additives included the following: titanium dioxide TiO_2 (TU 6-10-1356-73), the Al—4%Ti ligature (GOST 11739.20-99), the K_2TiF_6 salt (TU 20.13.62-023-69886968-2017), titanium sponge of TG-90 grade (GOST 17746-79), as well as standard flux 62.5 % NaCl (GOST 4233-77) + 12.5 % KCl (GOST 4568-95) + 25 % NaF (GOST 4463-76) [21]. The quantity of introduced Ti additives was determined based on a calculation aiming for a titanium content of 0.1 wt.%. When introducing titanium sponge, an allowance of 5 % for carbon monoxide was considered, and the standard flux was added at a rate of 1.5 % of the melting mass.

Before melting, fluoride and chloride salts (NaF, KCl, NaCl, K_2TiF_6) and titanium dioxide powder underwent a drying process at t = 150÷200 °C for 2 h to eliminate moisture.

Experimental melting was conducted within an electric resistance furnace, and each experiment was repeated 3 times.

The treatment of the melt with the Al—4%Ti ligature and titanium sponge occurred at t = 740 and 800 °C, respectively. After mixing, dissolution, and holding, the melt underwent degassing with argon.

Complex treatment involving the Al—4%Ti ligature and standard flux, as well as with titanium sponge and standard flux, was performed sequentially. Initially, the melt was treated with the ligature at t = 740 °C, and the sponge applied at t = 800 °C. Afte mixing and holding, standard flux was introduced at t = 740 °C.

When the melt was treated with the K_2TiF_6 salt, standard flux and titanium dioxide TiO_2, the melt was pre-degassed with argon, and then additives were poured on the melt surface at t = 750, 740 and 780 °C, respectively.

Complex treatment involving standard flux and the K_2TiF_6 salt was executed sequentially. First, the melt was treated with standard flux at t = 740 °C, and then, after mixing and holding, the K_2TiF_6 salt was introduced at t = 750 °C. Complex treatment with titanium dioxide and standard flux was performed concurrently at t = 780 °C.

After the treatment with additives, the melt was allowed to stand for 15 min, and slag was removed from the surface. Samples for mechanical tests were cast into a sand—clay mold at t = 710 °C.

The determination of mechanical properties (tensile strength and relative elongation) was conducted using the Instron 5982 testing system (USA).

For the evaluation of the macro- and microstructure of the AK12 alloy samples, thin sections were prepared according using established methods. Thin macro sections were etched with the 10 % copper chloride solution and clarified in concentrated nitric acid.

Micrographical investigations were carried out using an all-purpose Olympus GX51 research microscope (Olympus Corp., Japan) equipped with the Image-Pro image analyzer (Instron, USA).

The dispersion capacity of the eutectic was assessed by measuring the average length of silicon particles within ($\bar{l}_{Si}$). The average grain size ($\bar{d}$) was determined using the random secant method, calculated as the ratio of the secant length to the number of secant intersections with grain boundaries. In order to analyze the distribution of solid solution dendrites across the thin section area, the secondary dendrite arm spacing (SDAS) was determined ($\bar{\lambda}_2$) [22]. In order to ensure the accuracy of the analysis, a minimum of 50 measurements were conducted, and the resulting values were averaged. Statistical analysis of the research results for both the average length of silicon particles and the average grain size was carried out using Statistica 10 software.

The chemical (elementary) composition of the tested samples was examined utilizing a Q4 TASMAN-170 spark optical emission spectrometer (Bruker Quantron GmbH, Germany).

Results and discussion

The research results demonstrate that the introduction of titanium through titanium-containing additives significantly impacts the properties and structure of the AK12 alloy (Fig. 1).

An enhancement in the mechanical properties of the alloy and the refinement of its structural components are observed with the introduction of all compounds, except when titanium dioxide is introduced alone, and no titanium transition into the alloy occurs (Fig. 1, *e*).

For single introductions of titanium sponge, ligature, and salt, there is a noticeable increase in mechanical properties compared to the initial alloy: the relative elongation (δ) increases by 2.5, 2.2 and 3.1 times, respectively, while the ultimate strength (σ_u) increases by 24, 19, and 25 %, respectively.

Based on the spectral analysis data (Fig. 1, *c*), it is evident that when each of the considered titanium-containing additives is used, a high degree of assimilation of the modifying element by the melt occurs: the titanium yield from the ligature is 100 %, and the yield from the salt and sponge is 80 %.

The improvement in the mechanical properties of the alloy is a result of structural modification. According to qualitative (Fig. 2) and quantitative (Fig. 1, *d*—*f*) structural analysis data, the modification of the AK12 alloy through titanium-containing additives primarily leads to a reduction in the spacing between the secondary dendrite arms ($\bar{\lambda}_2$) of the aluminum-based (α-Al) solid solution (Fig. 1, *e*).

As a consequence of this modification, the dendrites acquire a more compact morphology. Compared to the original alloy, $\bar{\lambda}_2$ decreases by a factor of 1.5—2.0 (Fig. 2, *c2*, *d2*, *e2*). Titanium also promotes macro-grain refinement, resulting in a reduction of more than 5 times (Fig. 1, *d*; Fig. 2, *c3*, *d3*, *e3*).

Nonetheless, titanium introduced via the ligature, titanium sponge, and K_2TiF_6 salt not only influences the solid solution (α-Al) dendrites and macro-grain but also leads to a reduction in the average size of silicon within the eutectic (Fig. 2, *c1*, *d1*, *e1*). Compared to the original alloy, the average silicon size is reduced by a factor of 1.5, 2.5, and 3.5, respectively (Fig. 1, *f*). Modification with titanium-containing substances refines the α-Al dendrites, narrows the inter-dendrite spacing, and subdivides the eutectic into micro-volumes situated within the inter-arm spaces. The eutectic solidifies within a more confined space between dendrite arms, and the silicon refinement occurs due to the restriction of its growth within these micro-volumes. This hypothesis aligns with findings in [23; 24].

The K_2TiF_6 salt plays a significant role in refining the eutectic compared to other titanium-containing additives. It is presumed that this heightened effectiveness is due to the additional influence of potassium, which is present in the salt and acts as a eutectic surfactant [25]. The additional impact of the sponge on the refinement of eutectic silicon is likely a result of the extended holding time of the alloy (>30 min) at elevated temperatures (>780 °C) [26].

In order to investigate the combined effect of titanium addition, it was introduced simultaneously with a sodium and potassium salt-based flux. The standard Na-containing flux is commonly used for eutectic modification in silumins [1; 2]. According to the adsorption theory of modification, sodium, as a surfactant, adsorbs onto the surface of growing silicon crystals, slowing their growth. This process leads to the formation of a finely modified structure within the alloy under supercooled conditions.

The relative elongation of the AK12 alloy, modified with the Na-containing flux, exceeds that of all previously studied titanium-containing additives, reaching a value of 8.1 %, which is 3 times higher than without modification. The flux has a modifying effect on eutectic silicon (Fig. 3, *a1*) but does not influence the SDAS and macro grain diameter (Fig. 3, *a2*, *a3*).

The concurrent introduction of flux and titanium-containing additives, such as titanium dioxide TiO_2, ligature, and K_2TiF_6 salt, results in an even more significant improvement in properties. In comparison to treatment with sodium-containing flux, δ was found to be higher by 36 %, 37 %, and 20 %, and σ_u by 10 %, 7 %, and 21 %, respectively. The combination of titanium sponge with flux also enhances properties but to a lesser extent, with δ being higher by 17 % and σ_u by 7.5 %.

The increased mechanical properties of the alloy are attributed to the structural refinement, especially of the dendrites of the solid solution (α-Al) (Fig. 3, *b2*, *c2*, *d2*, *e2*), and the macrostructure (Fig. 3, *b3*, *c3*, *d3*, *e3*). Consequently, the addition of titanium-containing additives to the flux has minimal impact on the degree of eutectic silicon refinement (Fig. 3, *b1*, *c1*, *d1*, *e1*). When modified with sodium and titanium, as well as sodium alone, the eutectic silicon takes on a globular, highly refined form (compared to the original alloy, the average length of silicon particles decreases by 10—20 times). The inclusion of Ti-containing

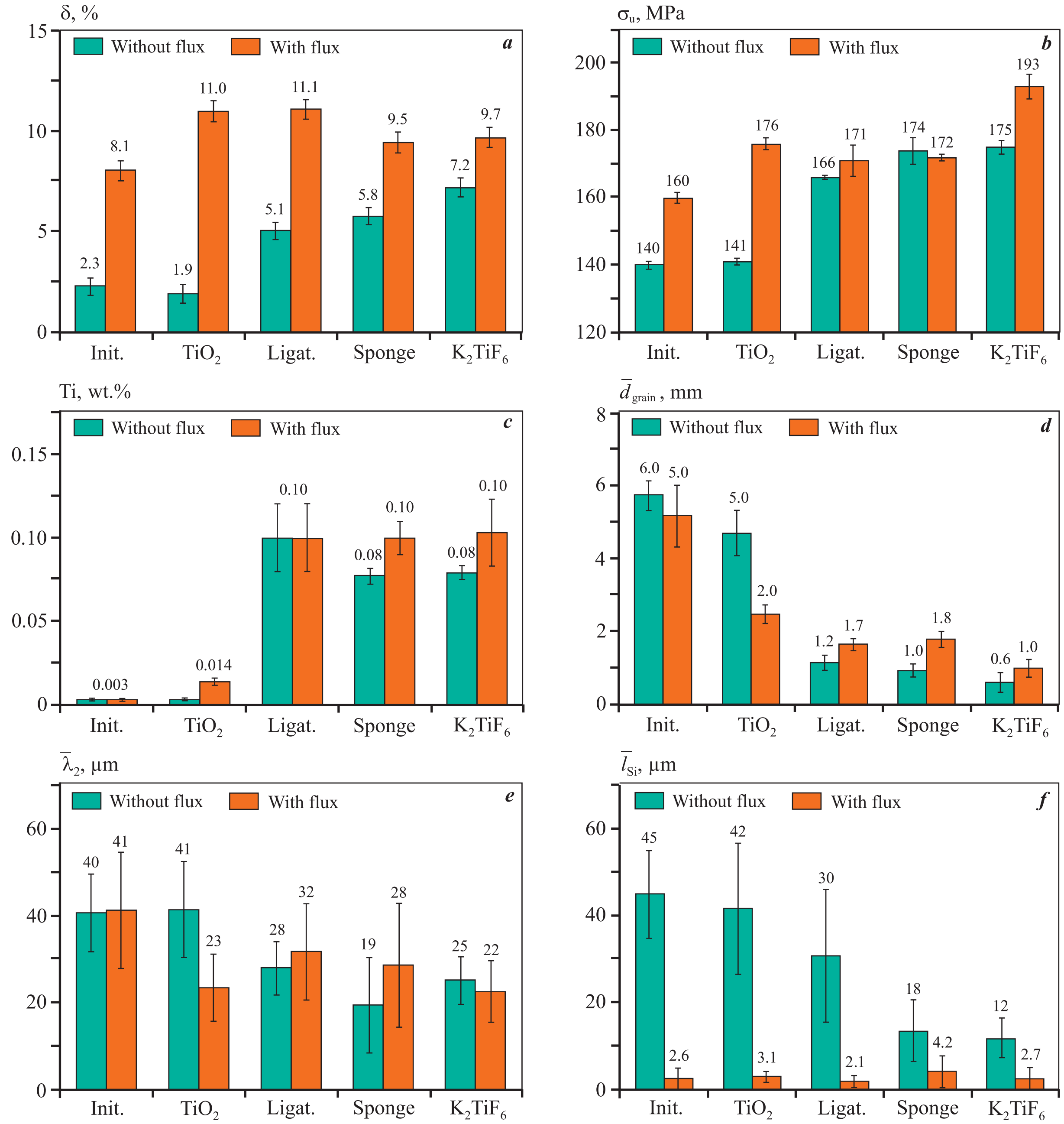
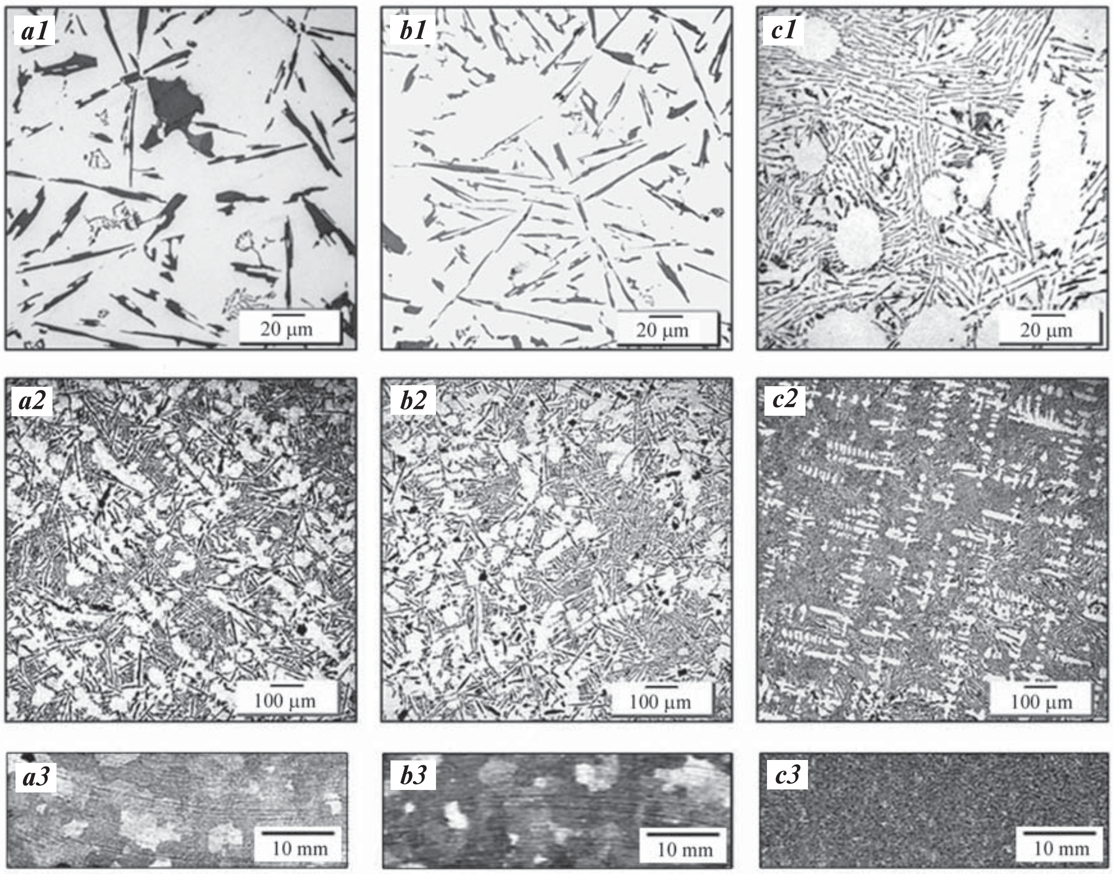


Fig. 1. The influence of different titanium introduction methods on the mechanical properties and structural parameters of the AK12 alloy

a – relative elongation; ***b*** – tensile strength; ***c*** – titanium content in the alloy according to spectral analysis data;
d – average diameter of the macro grain; ***e*** – average secondary dendrite arm spacing; ***f*** – average length of eutectic silicon particles

Рис. 1. Влияние способов ввода титана на механические свойства и параметры структуры сплава АК12

a – относительное удлинение; ***b*** – предел прочности на разрыв; ***c*** – содержание титана в сплаве, по данным спектрального анализа;
d – средний диаметр макрозерна; ***e*** – среднее расстояние между дендритными осями второго порядка; ***f*** – средняя длина частиц эвтектического кремния



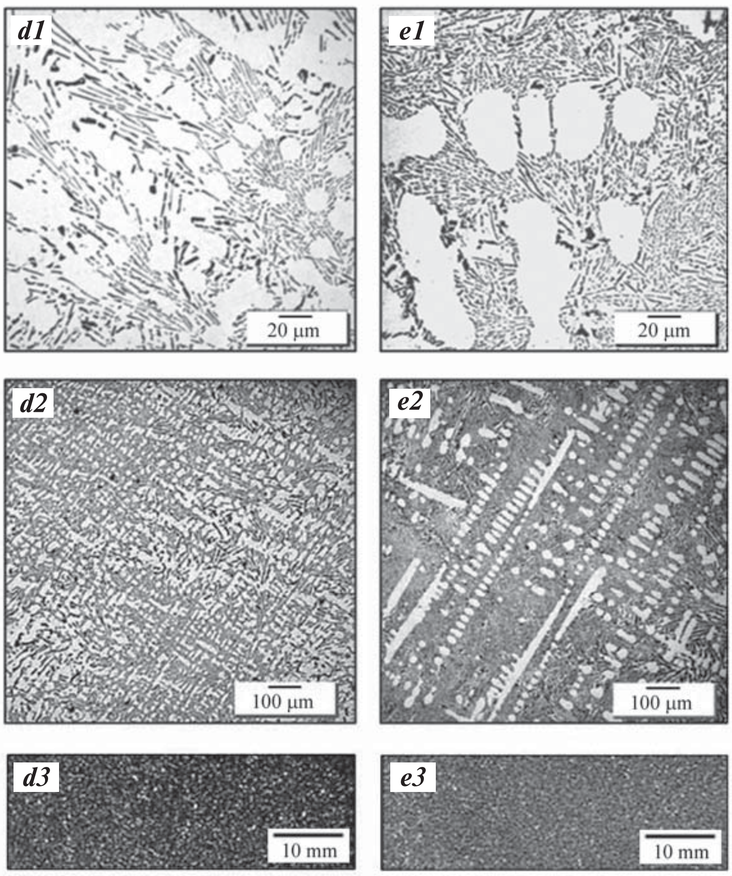


Fig. 2. The influence of different titanium introduction methods on the micro- and macrostructure of the AK12 alloy

a1–a3 – initial alloy;
b1–b3 – treated with titanium dioxide TiO_2;
c1–c3 – treated with ligature;
d1–d3 – treated with sponge;
e1–e3 – treated with K_2TiF_6 salt

Рис. 2. Влияние способов ввода титана на микро- и макроструктуру сплава АК12

a1–a3 – исходный сплав;
b1–b3 – обработанный диоксидом титана TiO_2;
c1–c3 – лигатурой;
d1–d3 – губкой;
e1–e3 – солью K_2TiF_6

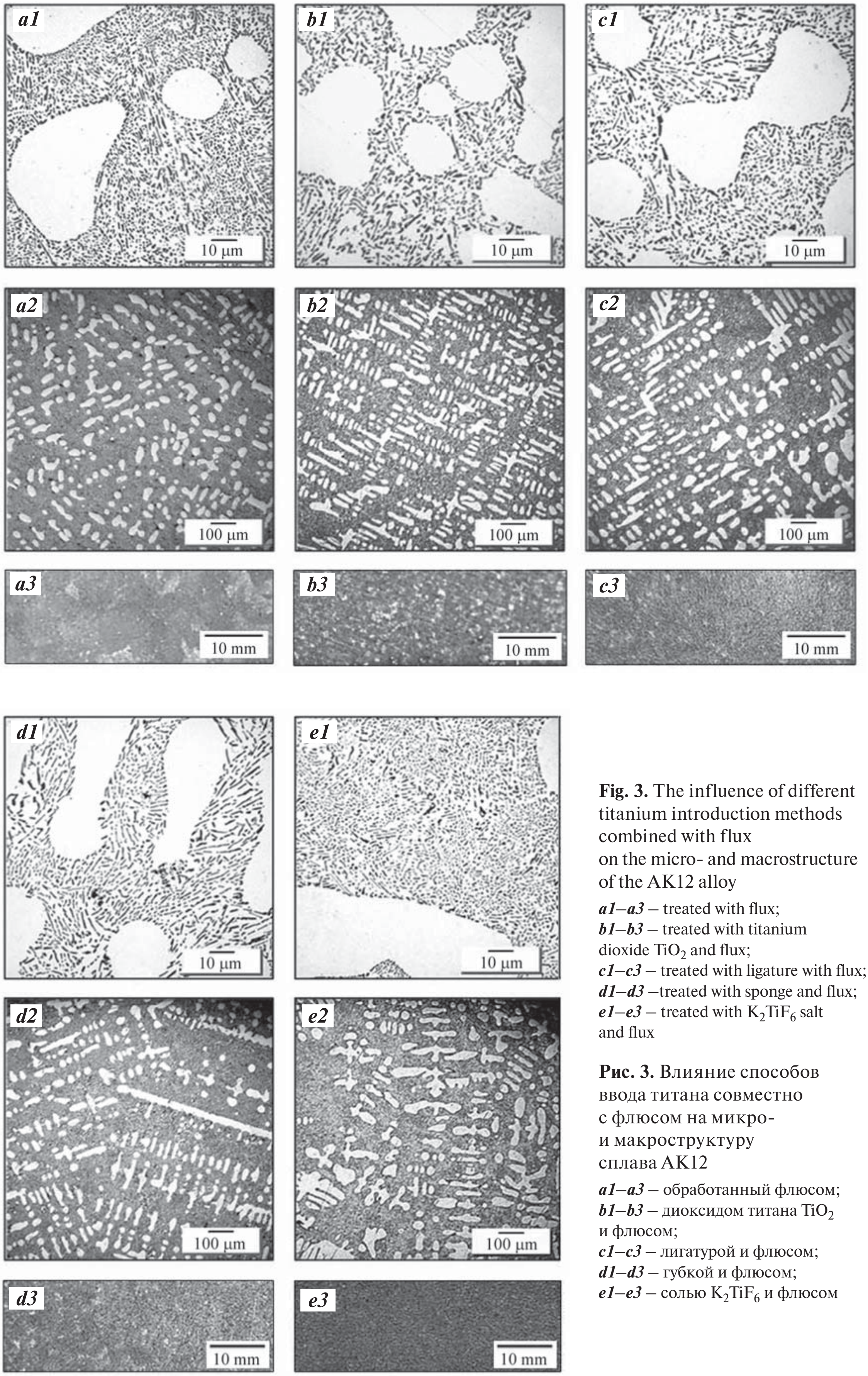


Fig. 3. The influence of different titanium introduction methods combined with flux on the micro- and macrostructure of the AK12 alloy

a1–a3 – treated with flux;
b1–b3 – treated with titanium dioxide TiO_2 and flux;
c1–c3 – treated with ligature with flux;
d1–d3 –treated with sponge and flux;
e1–e3 – treated with K_2TiF_6 salt and flux

Рис. 3. Влияние способов ввода титана совместно с флюсом на микро- и макроструктуру сплава АК12

a1–a3 – обработанный флюсом;
b1–b3 – диоксидом титана TiO_2 и флюсом;
c1–c3 – лигатурой и флюсом;
d1–d3 – губкой и флюсом;
e1–e3 – солью K_2TiF_6 и флюсом

additives in the sodium flux reduces the dendrite arm spacing by 1.3—1.9 times (Fig. 1, *e*), the macro grain diameter by 2—4 times (Fig. 1, *d*), and subsequently improves the alloy's properties.

The standard flux enhances the yield of titanium from titanium dioxide, increasing it from 0 to 14 % in the melt. This effect can be explained by the fact that the fluoride—chloride flux reduces interfacial tension and improves the wetting of solid oxide particles by the melt [27; 28]. Additionally, titanium dioxide dissolves in fluoride salts, leading to aluminothermal reduction of titanium dioxide to titanium in the silumin melt and, consequently, enhanced assimilation of titanium by the melt [17].

Undoubtedly, a significant benefit of modifying silumins with titanium is the sustained duration of the modifying effect. Previous studies have shown that titanium introduced with K_2TiF_6 [20] and TiO_2 [19], at a content of 0.05—0.15 % depending on the composition of complex fluxes, maintains its modifying effect for up to 5 hours and through several remeltings.

Conclusion

1. The positive impact of using titanium for the modification of eutectic silumin has been conclusively demonstrated. Titanium, at levels of 0.05—0.1 wt.%, has a favorable effect on the structure and mechanical properties of the AK12 alloy. However, the extent of these properties and the degree of structural modification depend on the method of titanium introduction. When introduced without flux, the use of the K_2TiF_6 salt for introduction is the most effective.

2. The positive influence of titanium, introduced with the K_2TiF_6 salt, Al—4%Ti ligature, and Ti sponge, has been observed in terms of macro grain refinement, the reduction of spacing between secondary dendritic arms in the α-solid solution, and the dispersion of eutectic silicon within the AK12 alloy.

3. The most effective method of introducing titanium into the silumin melt is through joint introduction with titanium-containing substances and a sodium salt-based flux, at a rate of 0.1 % Ti and 1.5 % flux relative to the melting mass. This combination has a complex effect on the silumin structure, involving simultaneous macro grain refinement, the reduction of spacing between secondary dendritic arms of the solid solution (α-Al) facilitated by titanium, and the refinement and enrichment of silicon in the eutectic with sodium. As a result, the modified AK12 alloy, when cast into a sand—clay mold, achieves a relative elongation of 9.5—11.1 % and an ultimate strength is 171—193 MPa.

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