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Features of microstructure formation in the AK4-1 and AK12D aluminum alloys after their joint friction stir processing

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Abstract: Friction stir processing is one of the modern methods of local modification of the surface of aluminum alloys in the solid-phase state, which provides the dispersion of structural components. In heat-hardened aluminum alloys with a matrix type structure, heat treatment following after friction stir processing can lead to abnormal grain growth in the stir zone. However, in alloys with the structure close to microduplex type, a fine-grained structure can be formed after friction stir processing and heat treatment. This work is aimed at evaluating the possibility of increasing the microstructure thermal stability of the AK4-1 (Al–Cu–Mg–Fe–Si–Ni) matrix-type aluminum alloy. For this purpose, AK12D (Al–Si–Cu–Ni–Mg) aluminum alloy with the structure close to microduplex type was locally mixed into the studied alloy by friction stir processing. Subsequent T6 heat treatment was carried out according to the standard mode for the AK4-1 alloy. Studies showed that the stir zone had an elliptical shape with an onion-ring structure. This structure comprised alternating rings with different amounts and sizes of excess phases. At the same time, in the stir zone center, the width of rings and the average area of excess phases were larger compared to the stir zone periphery, where the width of rings and the average area of particles were smaller. The average area of excess phases in the rings with their higher content was smaller than in the rings with their lower content. This distribution of excess phases leads to the formation of a fine-grained microstructure, where the average size of grains depends on the interparticle distance in the α-Al solid solution.

Keywords: aluminum alloys; AK4-1; AK12D; friction stir processing; heat treatment; thermal stability; structure of onion rings; onion-ring structure.

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INTRODUCTION

Friction stir processing (FSP), as one of the methods of surface hardening of solid-phase aluminum alloys, has perspectives for development in various industries, since, compared to other traditional methods of surface treatment, it is free of such disadvantages as agglomeration of additive particles, formation of unwanted phases, and interphase reactions due to the high processing temperature, the necessity of using additional processing methods and complex technological equipment, low processing efficiency, etc. [1]. This method of local surface modification of alloys, based on the physical principles of friction stir welding is caused by severe plastic deformation at elevated temperatures, and provides the formation of a fine-grained structure due to the mechanisms of dynamic recrystallisation and recovery [2]. Such a structure often leads to an optimal combination of strength and ductility [3–5].

In heat-hardened aluminum alloys, the strength characteristics are largely determined by coherent dispersed particles, the formation of which occurs due to precipitation hardening during heat treatment, which includes quenching and subsequent artificial aging [6]. However, a significant temperature gradient occurs during friction stir processing/welding [7]. In this case, particles of secondary phases in the processed alloy can undergo complex transformations, which can lead to degradation of strength characteristics. For example, in the thermo-mechanically affected zone, which is subjected to a relatively low temperature effect (≈0.7–0.6 Tmelt, Tmelt is the homologous melting temperature), as a rule, coagulation of excess phases is observed, which can lead to a coherence breakdown at the interphase boundaries [8]. In the stir zone heated to high temperatures (≈0.9–0.75 Tmelt), particles of secondary phases usually dissolve in the aluminum matrix. Moreover, depending on the deformation temperature during FSP, upon subsequent cooling of the alloy to room temperature, dispersoids can partially precipitate from the α-Al solid solution [9–11], contributing to a partial restoration of the strength characteristics.

The required level of mechanical properties of aluminum alloys after friction stir processing can be achieved by subsequent re-quenching and artificial ageing. In this case, a relatively uniform release of hardening particles is provided in different areas of the treatment zone. However, high temperature treatment often leads to abnormal grain growth in the stir zone, which was observed in various aluminum alloys with a matrix type structure [12–14]. Doping of aluminum alloys with high-strength second-phase particles, such as SiC Al_2O_3, B_4C, SiO_2, TiC, fullerene, carbon nanotubes, graphene, etc., followed by the formation of aluminum-matrix composites, does not always lead to the suppression of abnormal grain growth [15]. Abnormal grain growth is usually described in terms of so-called Humphrey's "cellular model" [16; 17], according to which the anomalous nature of grain growth is associated either with an increased content of low-angle boundaries, or with a relatively low concentration of particles of secondary phases. Nevertheless, it was shown in [18] that a fine-grained structure, with an average grain size of about 3.3±0.1 μm is formed in the AK12D aluminum alloy after FSP and T6 heat treatment. This alloy is characterized by a structure close to that of the microduplex type and consisting of an α-Al matrix, and a large number of excess phases, including the eutectic silicon particles [6]. In this case, a large proportion of dispersed excess phases hindered the migration rate of grain boundaries under the action of the Zener retarding force [19].

Since doping of aluminum matrix alloys, with the second-phase particles does not always help to suppress the anomalous nature of grain growth. It was suggested that a local increase in particles of excess phases due to the mixing of a microduplex-type aluminum alloy into it, can lead to the formation of a fine-grained structure after high-temperature treatment.

The purpose of this work is to evaluate the possibility of increasing the thermal stability of the AK4-1 aluminum alloy microstructure by the local mixing of the AK12D alloy into it by friction stir processing, and subsequent T6 heat treatment.

METHODS

In this work, the authors considered an commercial heat-resistant aluminum alloy AK4-1 with the following chemical composition: Al–1.97%Cu–1.73%Mg–1.01%Fe–0.98%Si–0.96%Ni–0.24%Co (wt. %). From a hot-pressed AK4-1 alloy bar Ø140 mm, plates were cut in the transverse direction, which were machined with a surface roughness of 0.6 Ra. The final thickness of the plates was 7 mm. Grooves 2 mm wide and 2 mm deep were cut out in the surfaces of these plates. As a reinforcing material, the industrial heat-resistant aluminum alloy AK12D with the following chemical composition was used: Al–12.8%Si–1.67%Cu–1.03%Ni–0.84%Mg–0.33%Mn–0.23%Co–0.24%Fe (wt. %). Inserts in the form of rectangular parallelepipeds made of AK12D alloy 2 mm wide and 2 mm high were placed in the grooves of AK4-1 alloy plates. The blank part was attached to the table of a modernized universal milling machine. In order for the inserts in the grooves not to move during the FSP process, the surface of this area was "rubbed" with hangers. In this case, the tool was advanced along the blank surface normal.

Then, a single-pass friction stir processing was performed. A processing tool with a cylindrical pin Ø6 mm and a height of 4 mm with a left-hand thread was used. The processing tool was introduced into the alloy under study at an angle of $\alpha=2°$ to the surface of the blank part, until its shoulders came into contact with the surface to be subjected to FSP. The speed modes of the processing tool were the following: rotation speed ω – 1000 rpm, feed rate ν – 30 mm/min. T6 heat treatment of all studied compositions of aluminum alloys was carried out according to the following mode: quenching at a temperature of 530±5 °C, artificial aging at 190±2 °C for 10 h.

Structural changes were evaluated in the initial heat-treated state, as well as in the state after FSP and subsequent T6 heat treatment. The cross sections of the processed blank parts were prepared for macro- and microstructural analysis. To study the macrostructure and the grain structure, the samples were etched in a solution of the following composition: H_2O (60 ml), HNO_3 (35 ml), HF (5 ml).

Macrostructural analysis of the cross sections of the samples was carried out using a ZEISS Axio Scope.A1 optical microscope (OM). Microstructural studies were carried out on a Tescan Mira 3LMH scanning electron microscope (SEM) using secondary electron (SE), and backscattered electron (BSE) detectors. Energy-dispersive spectral analysis (EDS) was performed on a Tescan Vega 3SBH SEM. Quantification of the average area (*S*) of particles of primary excess intermetallic phases (Pr) and silicon particles (Si), was carried out on the polished surface of the samples, using computer analysis techniques by graphical selection of a group of particles of each of the studied phases. For each treatment zone, quantitative measurements were carried out on the regions equal in area. The average grain size was estimated by the random linear intercept method in five fields of vision. When assessing the primary excess phases and the grain structure, at least 300 structural elements were measured. Processing of research results was carried out with a confidence level of 95 %.

RESULTS

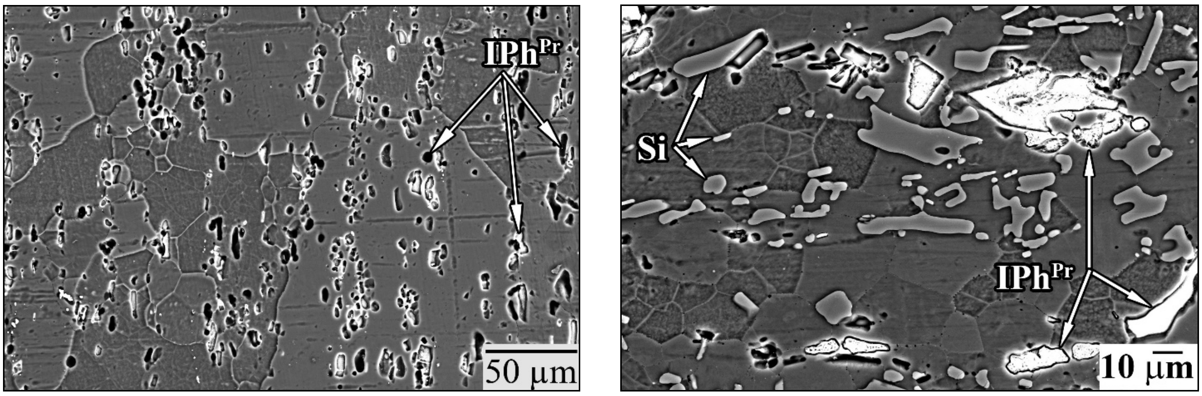
Initial microstructure

A typical microstructure of the AK4-1 aluminum alloy after T6 heat treatment consists of an α-Al solid solution, and a certain amount of excess intermetallide phases located in the direction of material flow during hot deformation (Fig. 1 a). According to [6], the following primary phases of crystallization origin can be present in the Al–Cu–Mg–Ni–Fe system alloys: Al_9FeNi, Mg_2Si, Al_7Cu_2Fe, and Al_2CuMg. After hardening heat treatment, metastable secondary hardening Al_2CuMg phases are formed in these alloys [6]. In the initial heat-treated state, a grain structure recrystallized with an average grain size of 78.6±8.0 μm is observed in the alloy.

A typical microstructure of the AK12D aluminum alloy after T6 heat treatment contains a certain amount of primary intermetallide phases and silicon, as well as a certain amount of secondary hardening phases (Fig. 1 b). In the Al–Si–Cu–Ni–Mg–Mn–Fe system alloys rich in silicon, in addition to the (Al+Si) eutectics, the presence of the following primary phases of crystallization origin can be expected: Al_5FeSi, Al_8Fe_2Si, $Al_{15}(Fe, Mn)_2Si$, $FeNiAl_9$, $Al_8FeMg_3Si_6$, Al_3Ni, Al_7Cu_4Ni, $Al_3(Ni, Cu)_2$, Al_2Cu, Mg_2Si, $Al_6Cu_2Mg_8Si_5$ [6]. When using T6 heat treatment, the formation of metastable secondary hardening phases, such as Al_2Cu, Mg_2Si, $Al_5Cu_2Mg_8Si_6$, Al_2CuMg, is possible [6]. Hardening heat treatment of the alloy leads to the formation of a grain microstructure with an average grain size of 11.5±0.4 µm.

Macrostructure after FSP

Fig. 3 shows a typical macrostructure of the AK4-1 aluminum alloy at local mixing of the AK12D alloy into it by friction stir processing. It can be observed that a defect-free processing area is formed. A stir zone consisting of a mixture of AK12D and AK4-1 alloys, a thermo-mechanical effect zone, and the base material zone corresponding to the AK4-1 alloy are visible in the structure (Fig. 2). In the near-surface region between the stir zone and the area of contact of the processing tool with a blank part, a coarse-grained microstructure of the AK4-1 alloy is observed. The stir zone has an elliptical shape with concentric circles progressively decreasing in radius, referred to as the “onion-ring” structure.



a *b*

Fig. 1. *Typical microstructure of the AK4-1 (**a**) and AK12D (**b**) alloys in the initial heat-treated state.*
IPh^{Pr} – primary intermetallic phases, Si – silicon particles. BSE mode SEM images
Рис. 1. *Типичная микроструктура сплавов АК4-1 (**а**) и АК12Д (**b**) в исходном термообработанном состоянии.*
IPh^{Pr} – первичные интерметаллидные фазы, Si – частицы кремния. BSE-режим съемки

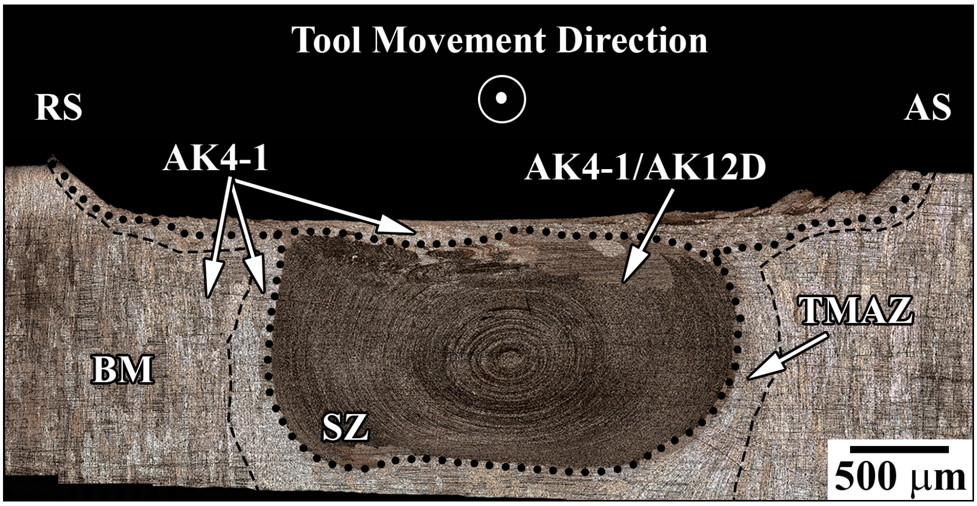


Fig. 2. *Macrostructure of the AK4-1 aluminum alloy after mixing into it the AK12D alloy via friction stir processing at the ω=1000 rpm and v=30 mm/min.*
AS – advancing side, RS – retreating side, SZ – stir zone, TMAZ —thermo-mechanical affected zone, BM – base metal (initial alloy). Optical metallography
Рис. 2. *Макростроение алюминиевого сплава АК4-1 после замешивания в него сплава АК12Д обработкой трением с перемешиванием при скоростях деформации ω=1000 об/мин и v=30 мм/мин.*
AS – наступающая сторона, RS – отступающая сторона, SZ – зона перемешивания, TMAZ – зона термомеханического влияния, BM – основной металл (исходный сплав). Оптическая металлография

Microstructure after FSP

The "onion rings" structure is heterogeneous over the cross section of the stir zone, and represents alternating rings with different amounts and sizes of excess phases (Fig. 3). The width of the rings decreases from the stir zone center to the periphery. Friction stir processing leads to intense crushing of particles of excess phases. A quantitative assessment of the alloy microstructure is given in Table 1. The average area of particles in rings with a higher content of excess phases is smaller than in rings where the number of phases is lower. It should be noted that eutectic silicon particles are concentrated in rings with a higher content of primary phases (Fig. 4). This is also evidenced by the EDS analysis results. The corresponding distribution maps for the main alloying elements are shown in Fig. 5.

The study of the grain structure showed that after FSP, and T6 heat treatment, a fine-grained microstructure is formed in the stir zone (Fig. 6), the average grain size (Table 1) of which depends on the amount of excess phases located in different parts of the stir zone.

DISCUSSION

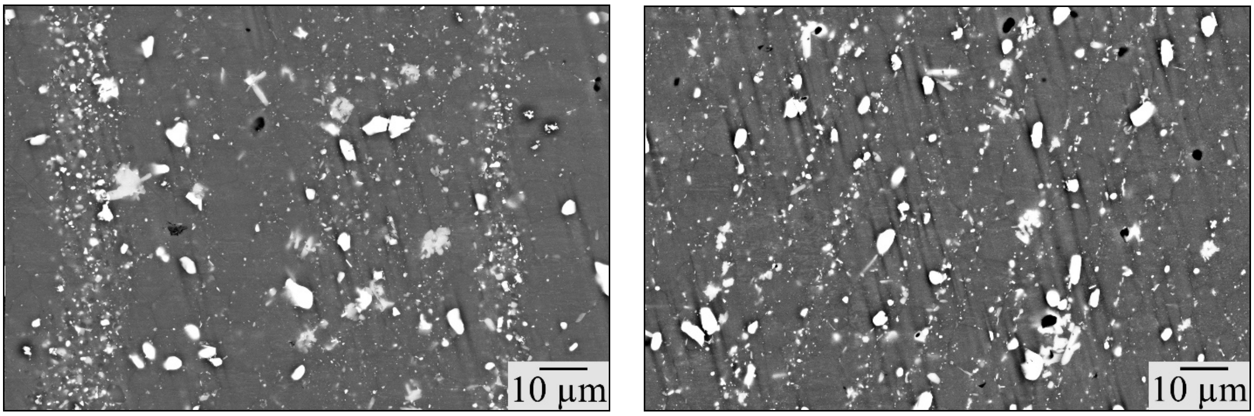
As a rule, the stir zone shape depends on the processing tool geometry, the technological parameters of processing, the thermal conductivity of the material, and the temperature of the blank part [20; 21]. The stir zone shape is much determined by the temperature of the heated alloy in the near-surface regions, during friction between the processing tool shoulders and the blank part. During friction stir processing, a stir zone typically assumes a basin-like shape with a widening at the blank part surface or an elliptical shape [20; 21].

According to findings presented in [21], a stir zone with a basin-like shape is created during low-speed tool rotation. In this case, the alloy yield strength is higher due to the low temperature of the heated material, and consequently, its volume subjected to deformation (caused by the pin motion) is smaller. With an increase in the rotation velocity of the tool, the temperature of the heated alloy increases, contributing to a decrease in the yield strength. An increase in the volume of the material that is involved in the deformation process, and the formation of an elliptical stir zone (Fig. 3). The formation of the "onion rings" structure occurs due to a periodic change in the stress state in the three-dimensional flow of a plastically deformable alloy, which is caused by the movement of the processing tool (shoulders and pins) [22–24]. As a result, the stir zone structure exhibits a periodically changing average grain size [25], alternating bands (rings) enriched in excess phases [26], different grain orientations [27], and texture changes [28]. Moreover, the temperature of deformation during the FSP process is non-uniform over the cross section of the stir zone [29–31]. Therefore, in the structure of the treatment zone, rings are observed, the width of which decreases from its center to the periphery.

As noted earlier, subsequent T6 heat treatment (including solution heat treatment and artificial aging) can lead to abnormal grain growth in the treatment area, which presumably indicates a low thermal stability of the microstructure. The AK12D alloy, in contrast to the AK4-1 alloy, is characterized by a structure close to that of the microduplex type [6]. The formation of a fine-grained microstructure during mixing of the AK12D alloy into the AK4-1 alloy by friction stir processing and subsequent T6 heat treatment occurs due to the fact that a large number of excess phase particles of both alloys, has a retarding effect on the migration of grain boundaries. In areas where the number of excess phases is greater, the average grain size is smaller. This is related to the smaller distance between the excess phase particles in the α-Al solid solution.

CONCLUSIONS

The authors studied the structure of the AK4-1 aluminum alloy, in which the AK12D alloy was locally mixed by friction stir processing, and subsequent T6 heat treatment carried out according to the standard mode for the AK4-1 alloy.



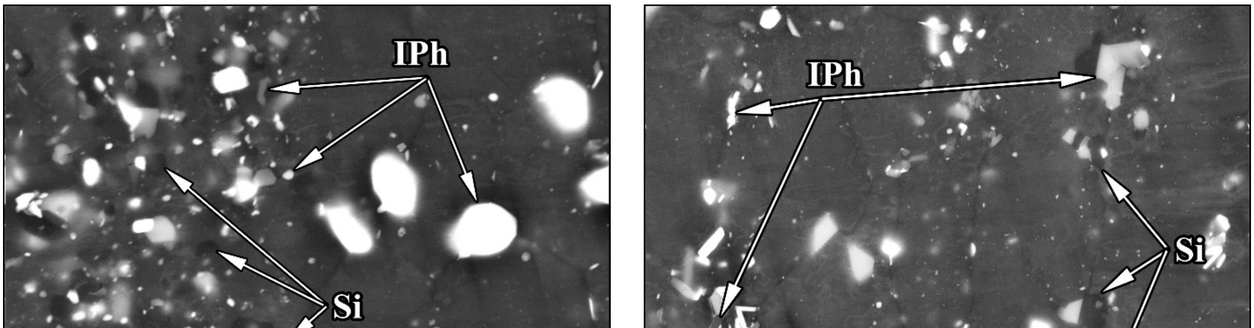
a *b*

Fig. 3. *Typical microstructure of the polished stir zone surface.*
*Ring fragments in the center (**a**) and on the periphery of stir zone (**b**) are shown. BSE mode SEM images*
Рис. 3. *Типичная микроструктура полированной поверхности зоны перемешивания.*
*Приведены фрагменты колец в центре (**a**) и на периферии зоны перемешивания (**b**). BSE-режим съемки*

Table 1. *Quantitative estimation of the AK4-1 aluminum alloy microstructure after mixing the AK12D alloy into it via friction stir processing*
Таблица 1. *Количественная оценка микроструктуры алюминиевого сплава АК4-1 после замешивания в него сплава АК12Д обработкой трением с перемешиванием*

Microstructural elements of alloys			State					
			Initial state		Friction stir treatment			
					Stir zone AK4-1/AK12D			
			AK4-1	AK12D	Center		Periphery	
					SZ	SZ-1	SZ	SZ-1
S	IPh	μm^2	12.8±1.0	39.9±4.0	13.5±0.4	0.17±0.01	10.3±0.3	0.13±0.01
	Si	μm^2	–	45.9±5.7	–	2.2±0.1	–	1.2±0.1
d		μm	78.6±8.0	11.5±0.4	7.1±0.2	3.0±0.1	4.0±0.1	

Note. SZ – rings with a low content of excess phases;
SZ-1 – rings with a higher content of excess phases;
S – average area of primary intermetallic phases (IPh) and Si particles;
d – average grain size. For comparison, a quantitative estimation of the structure in the initial heat-treated state of the AK4-1 and AK12D alloys is given.
Примечание. SZ – кольца с малым содержанием избыточных фаз;
SZ-1 – кольца с бóльшим содержанием избыточных фаз;
S – средняя площадь первичных интерметаллидных фаз IPh и частиц Si;
d – средний размер зерен. Для сравнения приведена количественная оценка структуры в исходном термообработанном состоянии сплавов АК4-1 и АК12Д.



a *b*

Fig. 4. *Typical microstructure of the polished stir zone surface.*
*Fragments of the boundaries between the rings with varying degrees of excess phases in the center (**a**) and on the periphery (**b**) of the stir zone are shown. IPh – intermetallic phases, Si – silicon particles. BSE mode SEM images*
Рис. 4. *Типичная микроструктура полированной поверхности зоны перемешивания.*
*Приведены фрагменты границ между кольцами с бóльшим и меньшим количеством избыточных фаз в центре (**a**) и на периферии (**b**) зоны перемешивания. IPh – интерметаллидные фазы, Si – частицы кремния. BSE-режим съемки*

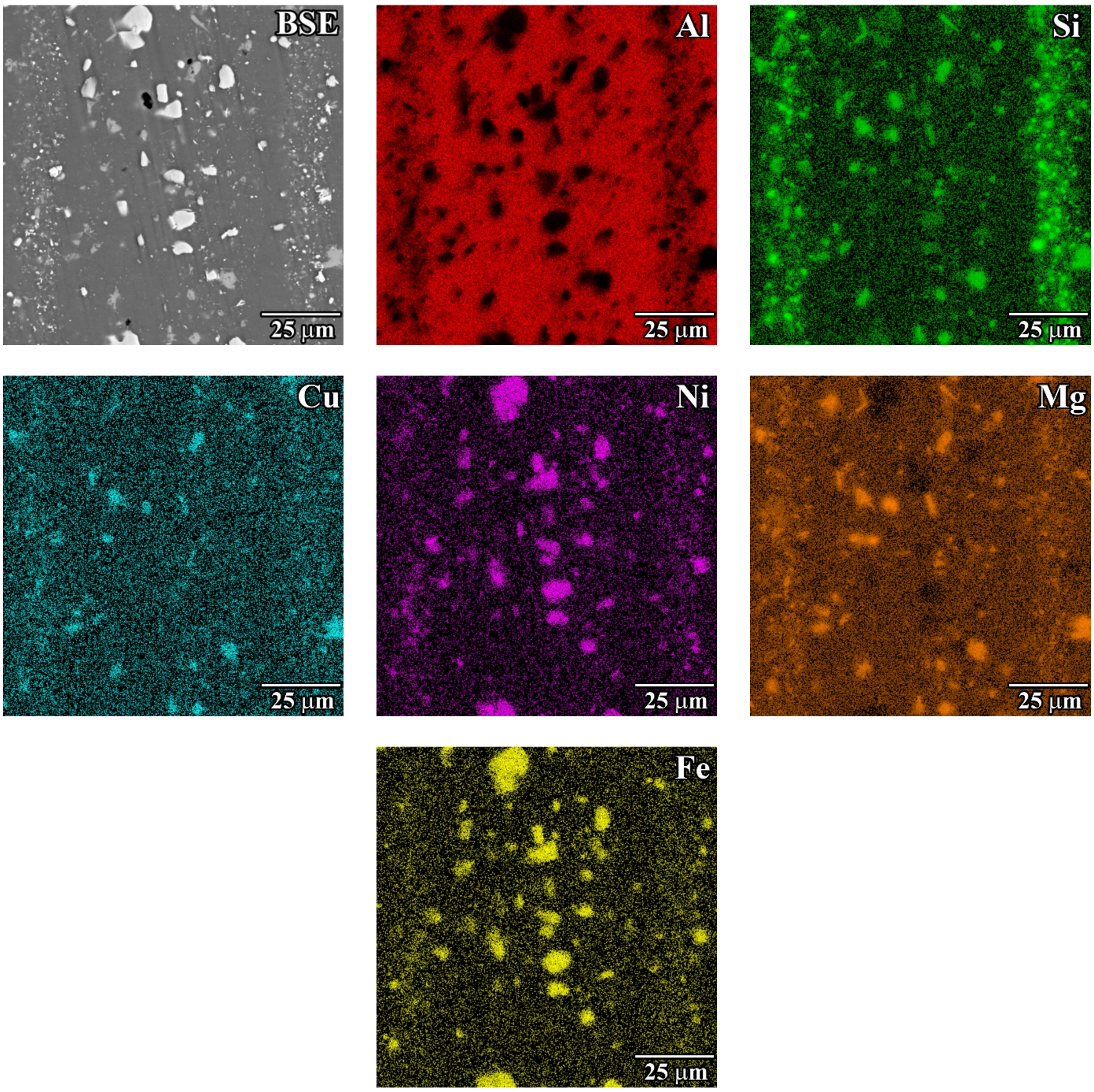
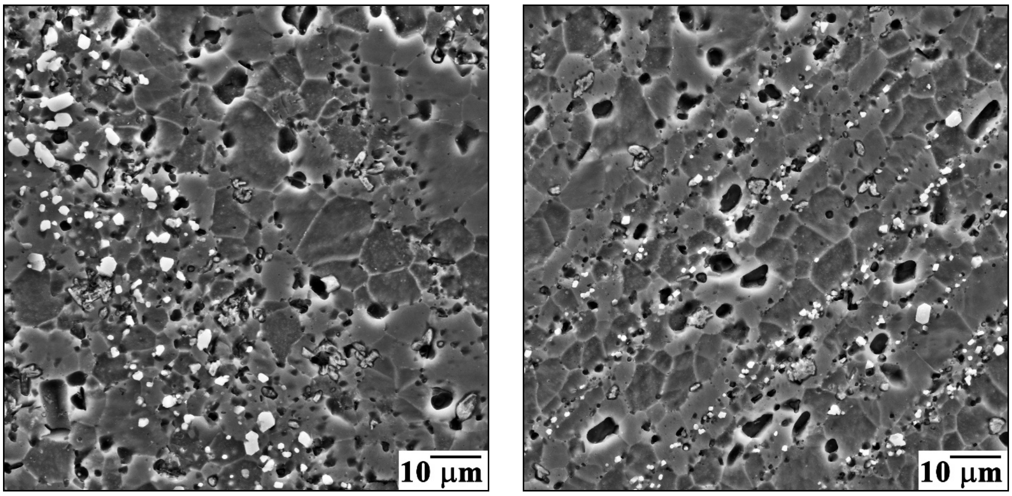


Fig. 5. *Distribution maps of the primary alloying elements in the center part of the polished stir zone surface. EDS analysis*
Рис. 5. *Карты распределения основных легирующих элементов в центральной части полированной поверхности зоны перемешивания. ЭДС-анализ*



a ***b***

Fig. 6. *Typical grain microstructure in the center (**a**) and on the periphery (**b**) of the stir zone. SE mode SEM images*
Рис. 6. *Типичная зеренная микроструктура в центре (**a**) и на периферии (**b**) зоны перемешивания. SE-режим съемки*

It is shown that after FSP, a defect-free treatment area was formed, the stir zone of which had an elliptical shape with the "onion rings" structure. The width of these rings decreased from the stir zone center to the periphery. The onion structure consisted of alternating rings with different amounts and sizes of excess phases.

It was established that the friction stir processing led to intense crushing of the primary excess phases of both alloys. At the same time, the average area of particles in rings with a higher content of them is smaller than in rings where their number is lower. Moreover, eutectic silicon particles are concentrated in rings with a higher content of primary phases.

It was found that the nonuniform distribution of particles of excess phases led to the formation of a fine-grained microstructure, the average grain size of which depends on the interparticle distance in the α-Al solid solution. The minimum average grain size was observed in the stir zone center in rings with a higher content of excess phases and was 3.0±0.1 μm. The largest average grain size reached 7.1±0.2 μm and was formed in the stir zone center in rings with a low content of excess phases.

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Особенности формирования микроструктуры алюминиевых сплавов АК4-1 и АК12Д после их совместной обработки трением с перемешиванием

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Аннотация: Обработка трением с перемешиванием – один из современных методов локального модифицирования поверхности алюминиевых сплавов в твердофазном состоянии, обеспечивающий диспергирование структурных составляющих. В термически упрочняемых алюминиевых сплавах со структурой матричного типа последующая после обработки трением с перемешиванием термообработка может приводить к аномальному росту зерен в зоне перемешивания. Однако в сплавах, структура которых близка к микродуплексному типу, после обработки трением с перемешиванием и термообработки может сформироваться мелкозернистая структура. Работа направлена на оценку возможности повышения термической стабильности микроструктуры алюминиевого сплава АК4-1 (Al–Cu–Mg–Fe–Si–Ni) матричного типа. Для этого в исследуемый сплав обработкой трением с перемешиванием локально замешивался алюминиевый сплав АК12Д (Al–Si–Cu–Ni–Mg) со структурой, близкой к микродуплексному типу. Последующая упрочняющая термообработка проводилась по стандартному режиму для сплава АК4-1. Исследования показали, что зона перемешивания имеет эллиптическую форму со структурой «луковичных колец». Такая структура представляет собой чередующиеся кольца с разным количеством и размером избыточных фаз. При этом в центре зоны перемешивания ширина колец и средняя площадь избыточных фаз больше по сравнению с периферией зоны перемешивания, где ширина колец и средняя площадь частиц меньше. Средняя площадь частиц избыточных фаз в кольцах с бóльшим их содержанием меньше по сравнению с кольцами, где их количество ниже. Такое распределение избыточных фаз приводит к формированию мелкозернистой микроструктуры, средний размер которой зависит от межчастичного расстояния в α-Al твердом растворе.

Ключевые слова: алюминиевые сплавы; АК4-1; АК12Д; обработка трением с перемешиванием; термообработка; термическая стабильность; структура луковичных колец; луковично-кольцевая структура

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