Metallophysics and Advanced Technologies
Металофіз. новітні технол.
Metallofiz. Noveishie Tekhnol.
2025, vol. 47, No. 9, pp. 361–375
https://doi.org/10.15407/mfint.47.04.0361
Reprints available directly from the publisher

PACS numbers: 62.20.Qp, 64.70.kd, 64.75.Nx, 64.75.Op, 81.05.Bx, 81.20.Ev, 81.40.Cd

Structure and Phase Composition of Sintered Alloys of the Al–Fe–Ga System

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Peculiarities of phase transformations in the Al–Fe–Ga system at temperatures of 800, 1000, and 1200°C are investigated. The objects of research are samples obtained from mixtures of Al powders and Fe–Ga ligatures of equiatomic composition. The proportion of Al in the mixture is of 90, 70, and 50% by mass. Sintering of the compressed samples is carried out in an Ar environment under a pressure of 0.2 MPa with isothermal holding at the sintering temperature for 1 hour. The structure of the samples is studied depending on the material composition and sintering temperature by x-ray phase analysis and x-ray spectral microanalysis and microdurometry. As established, the base of the structure of all samples is an Al-based solid solution and ternary intermetallic inclusions mainly of the $Al_x(Fe,Ga)_{(100-x)}$ type ($x = 2...3.6$). As found, the structure of the material with 90% mass. Al changes little with temperature. Phase composition becomes increasingly dependent on the temperature with increasing amounts of Al in the samples.

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Citation: D. A. Honcharuk, O. V. Khomenko, G. A. Bahliuk, O. M. Hripachevskyi, and V. M. Novychenko, Structure and Phase Composition of Sintered Alloys of the Al–Fe–Ga System, *Metallofiz. Noveishie Tekhnol.*, **47**, No. 4: 361–375 (2025). DOI: 10.15407/mfint.47.04.0361

The Al-based solution contains Ga atoms at the level of solubility of this metal in Al on the same level as in binary system Al–Ga (to 8–9% at.); at the same time, very small amount of Fe is detected in the solution. A practically constant ratio of Al atoms to the total number of other two components is found in the composition of intermetallics. In addition, in the structure of samples with 70% and 50% mass. Al, the residual Ga-based phase with a small amount of Al and Fe is observed. The microhardness of the Al-based solution depends slightly on both the composition of material and the sintering temperature and is equal to 0.41…0.45 GPa. Microhardness of intermetallics varies from 1.3…4.3 GPa (800°C) to 5.3 and 6.3 GPa (1000°C and 1200°C, respectively) for samples with a content of 70 and 50% mass. Al. Variations in hardness values are conveniently explained by variable composition of intermetallic inclusions. This indicator is maximal in the material with 90% mass. Al and corresponds to the level of hardness of the intermetallide Al_3Fe.

Key words: Al–Fe–Ga system, powder metallurgy, intermetallics, microhardness, microstructure.

Досліджено особливості фазових перетворень у системі Al–Fe–Ga за температур у 800, 1000 і 1200°C. Об'єктами досліджень були матеріяли, одержані із сумішей порошків Al та ліґатури Fe–Ga еквіатомного складу, з часткою Al в суміші у 90, 70 і 50% мас. Спікання спресованих зразків проводили в Ar під тиском у 0,2 МПа з витримкою за температури спікання впродовж 1 год. Основою структури всіх зразків є твердий розчин на основі Al й потрійні інтерметалідні включення переважно типу $Al_x(Fe,Ga)_{(100-x)}$ ($x = 2...3,6$). Структура матеріялу, що містить 90% мас. Al, мало змінюється з температурою спікання зразків. Зі зменшенням кількости Al в матеріялі його фазовий склад стає більш залежним від температури спікання. В розчині на основі Al виявлено Ga на рівні розчинности цього металу в подвійній системі Al–Ga (8–9% ат.); водночас, у розчині Fe практично відсутній. У складі інтерметалідів виявлено всі три компонента зі сталим співвідношенням атомів Al до загальної кількости атомів двох інших металів. Крім того, в структурі зразків із 70 та 50% мас. Al присутня окрема фаза на основі Ga з малою кількістю Al та Fe. Мікротвердість Al-розчину слабо залежить від складу матеріялу й температури спікання та дорівнює 0,41…0,45 ГПа. Для зразків із вмістом 70 та 50% мас. Al мікротвердість інтерметалідів змінюється від 1,3..4,3 ГПа (800°C) до 5,3..6,3 ГПа (1000°C і 1200°C). Варіяція твердости, певно, пояснюється змінним складом інтерметалідних включень. Цей показник є максимальним у матеріялі з 90% мас. Al, що відповідає рівню твердости подвійного інтерметаліду $FeAl_3$.

Ключові слова: система Al–Fe–Ga, порошкова металурґія, інтерметаліди, мікротвердість, мікроструктура.

(Received 13 June, 2024; in final version, 10 October, 2024)

1. INTRODUCTION

In recent years, materials scientists have been increasingly interested

in gallium (Ga) as an alloying element for obtaining a number of functional materials; this element, despite its own low melting point and low hardness, can form fairly hard and substitutional solid solution and compounds with metals [1–3], creating a synergy effect.

So, in particular, Fe–Ga alloys are in the centre of attention due to their giant magnetostriction in low saturation magnetic fields and high mechanical properties (with the possible exception of low plasticity) [2]. In recent years, special attention has also been paid to the inelastic properties of these alloys [3].

Based on the results of the study of martensitic transformations and magnetic properties of Heusler alloys (Ni–Fe–Ga systems), the authors [4] note that the martensitic structure formed in them has a low twinning stress and high magnetocrystalline anisotropic properties. They consider it expedient to use such alloys as efficient materials with shape memory.

Materials based on non-stoichiometric Ni–Mn–Ga alloys have also attracted considerable interest during the last decade due to their high damping properties. The authors note that their relative damping capacity is better than that of any other known material. In addition, these alloys demonstrated the presence of a shape memory effect, high mechanical superelasticity and superelasticity under the influence of magnetic field, and magnetocaloric properties [5].

Four-component alloys of the Ni–Fe–Mn–Ga system, obtained by vacuum-arc melting and subsequent heat treatment, are also characterized by the presence of ferromagnetic shape memory effect [6]. The results of the study of the influence of Fe content in the alloys of this system on the magnetic characteristics of the alloys showed that with increase in the Fe content, the saturation magnetization and the Curie temperature increase.

At the same time, the Al–Fe–Ga ternary system is currently practically unexplored, although dual-composition alloys based on the components of this system are widely researched and, obviously, have significant potential for the development of new functional materials with specific characteristics. Thus, in particular, alloys based on the Fe–Al double system (iron aluminides) are distinguished by a unique combination of mechanical properties (in particular, at elevated temperatures) in combination with resistance to corrosion and wear [7–12], which made it possible to use these intermetallics in aggressive environments in a wide temperature range.

The other side of the concentration triangle of the Al–Fe–Ga phase diagram is the double Fe–Ga system, which has become the subject of active research in the last three decades. The impetus for intensive scientific research was unexpectedly high values of magnetostriction, which Fe–Ga alloys demonstrate [1]. In this regard, studies of the Al–Fe–Ga ternary system were mainly focused on the region of the iron

corner of the diagram [13]. The concentration ranges enriched with aluminium and the conditions for the formation of ternary compounds based on it were neglected.

Taking into account the propensity of Al and Fe to form intermetallics of different composition and the similarity of the physical and chemical properties of Al and Ga, one can expect the formation of intermetallics of ternary composition in the Al–Fe–Ga system. Doping solutions of double intermetallics with a third component provides opportunities to optimize their properties. Considering the fact that the Al–Fe–Ga system combines in its composition components with a significant difference in melting temperatures, the study of the phase transformation of intermetallics in this system in the conditions of the interaction of liquid and solid phases in a wide range of concentrations seems to be particularly interesting and promising.

In this regard, the purpose of this work is to study the peculiarities of structure formation and phase composition of powder alloys of the Al–Fe–Ga system obtained by sintering in the temperature range of 800–1200°C.

2. EXPERIMENTAL DETAILS

Aluminium powder (PA-4, 98.99% mass. Al, GOST 6058-73), pulverized iron powder (PZHR GOST 9849-86, 99.9% mass. Fe) and Ga (Gl-1, GOST 12797, 99.99% mass. Ga) were used as starting components for obtaining alloys of various component compositions.

Since Ga is a low-melting metal with a melting point of 29.8°C, it was introduced into the initial charge in the form of a ligature alloy, namely, ferrogallium (Fe–Ga system), which was obtained with a composition close to equiatomic one by melting a mixture of iron powder and crushed Ga in bulk the ratio of the components as 55% mass. Fe–45% mass. Ga (50% at. Ga) [14]. The melting of the ligature alloy was carried out in Ar at a temperature of 1300°C.

The mode of the mixture components fusion was chosen according to the state diagram of the Fe–Ga system [15], according to which the melt of equiatomic composition crystallizes at temperatures lower than 1037°C, and taking into account the required degree of overheating and time for the melt homogenization. Before mixing with aluminium powder, the melt master alloy was crushed and the ≤ 100-μm fraction was sieved.

Aluminium and iron powders and ligature alloy were mixed in appropriate proportions to obtain mixtures of different component composition (Table 1). The resulting batch mixtures were dried in vacuum at a temperature of 200°C for 2 hours. The composition of the mixtures was selected in such a way as to investigate the competing effects of Fe and Ga on phase transformations in the material in a range in a fairly

TABLE 1. Component compositions of the original powder mixtures.

Composition of the mixture, % mass.	Atomic concentrations of metals in the mixture, % (at.)		
	Al	Fe	Ga
90 Al–10 ligature alloy	95	2.5	2.5
70 Al–30 ligature alloy	84	8	8
50 Al–50 ligature alloy	70	15	15

wide range of Al atomic concentrations.

The powder mixtures were consolidated into briquettes with a diameter and height of 20 mm under the pressure of 400 MPa. The mixtures had good compressibility, held their shape after consolidation and had no visible cracks.

Samples of each composition, in turn, were divided into three groups, which were sintered at temperatures of 800°C, 1000°C, and 1200°C, respectively, with isothermal exposure for 1 hour in a graphite crucible in the chamber of an induction furnace. In order to degas the chamber, 5 cycles of rarefaction to a vacuum of 10^{-1} MPa were carried out, followed by filling the chamber with Ar. The choice of temperature range is determined by the presence of phase transitions in this range and taking into account the need for overheating. The rate of heating the samples to the sintering temperature was 40°/min on average, after isothermal exposure, cooling to room temperature was carried out together with the furnace.

The phase composition of the obtained alloys was determined by x-ray phase analysis and local x-ray microspectral analysis. Diffractograms were taken on a DRON-3M spectrometer in point mode using the powder method ($U = 35$ kV, $I = 35$ mA in CoK_{α_1} radiation). A graphite single crystal was used as a monochromator. The shooting was carried out in the range of angles $2\theta = 20–100°$ with a scan step of 0.05° and an exposure time of 2 sec. The x-ray spectrum analysis program MATCH v.1.9 was used with the help of a JEOL microscope model JSM-46 CAMECA (France). Quantitative analysis was performed using the program ZOND, which calculated corrections for absorbance, fluorescence, and atomic number.

For the metallographic analysis of the alloys' structure, metallographic samples were made in a standard way, which were studied by the methods of optical and scanning electron microscopy. To determine the structural components, etching of the surface of the sections with reagents of the composition (HF–HCl–HNO_3–H_2O and $FeCl_2$–HCl–C_2H_5OH), which are used to detect the structures of Al-containing and Fe-containing alloys, respectively. Microdurometric measurements were performed on a PMT-3 microhardness tester at a load of 0.49 N.

3. RESULTS AND DISCUSSION

The appearance of the compacts after sintering showed that while the samples sintered at 800°C practically kept the shape of the initial preforms (Fig. 1, *a*), the samples sintered at 1000°C and 1200°C lost their initial shape as a result of melting (Fig. 1, *b*).

The porosity of samples with 90% mass. Al, regardless of the sintering temperature, does not exceed 1%. Reducing the concentration of Al in the alloy to 50–70% mass. leads to a significant increase in the porosity of sintered materials. Thus, the samples obtained by sintering at 800°C are characterized by porosity at the level of 40–50%, while with an increase in the sintering temperature to 1000–1200°C, their porosity decreases to the level of $\leq 10\%$.

On the surface of the samples, there are separate places of sweating of the liquid phase, caused by the low ability to wet the remains of the oxide phases with liquid aluminium. Individual shrinkage shells are found in fractures, stepped intercrystalline chips appear on the surface of the fracture in places of accumulation of small and large crystals, which alternate with regions of a viscous matrix.

The microstructure of the sintered samples is presented in Figs. 2 and 3. In the structure of the samples with the highest content Al (90% mass.), the volume concentration of the Al-based matrix phase is about 70–75%, everything else is mainly needle-shaped intermetallic inclusions (see Fig. 2, *a*).

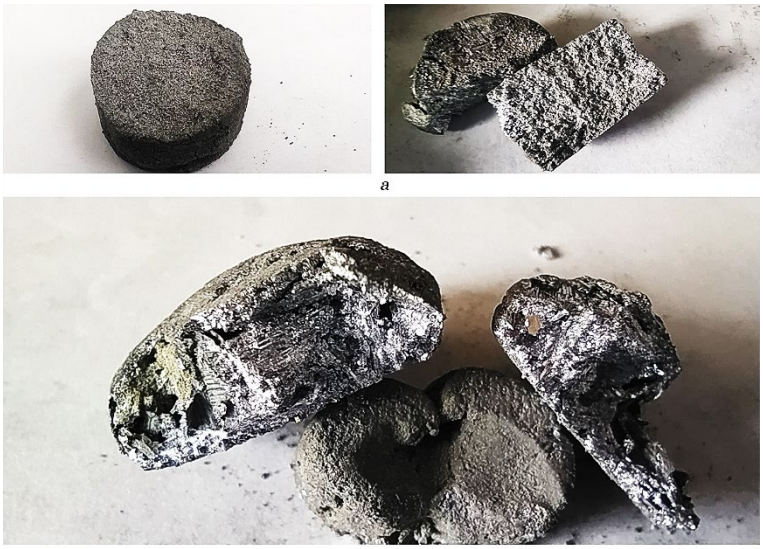

Fig. 1. The appearance and fracture surfaces of samples with composition 70% mass. Al–30(Fe–Ga), sintered at 800°C (*a*) and at 1200°C (*b*).

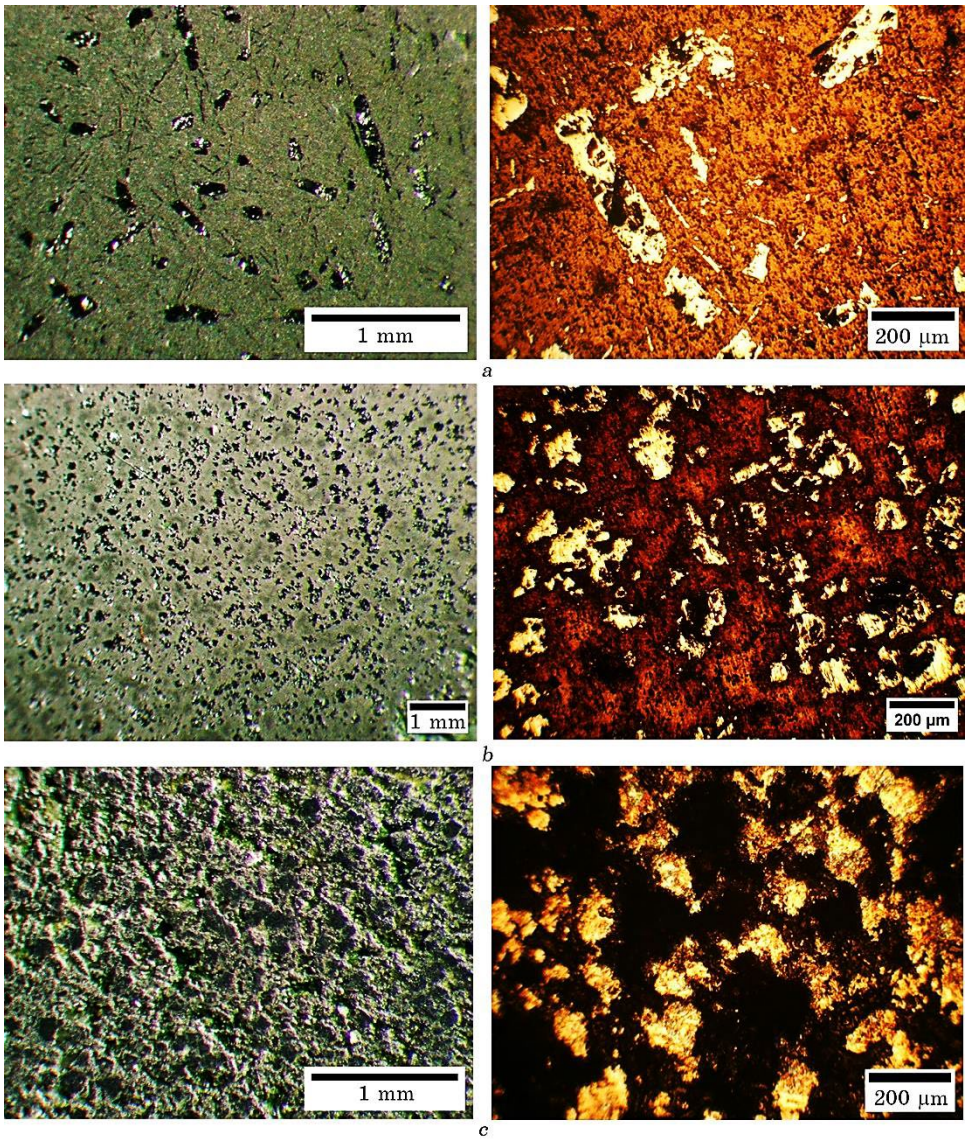

Fig. 2. Structure of samples sintered at 800°C depending on the material composition (% mass.): 90Al–10(Fe–Ga) (*a*); 70Al–30(Fe–Ga) (*b*), 50Al–50(Fe–Ga) (*c*).

A similar shape of crystals, determined by separate directions of rapid crystal growth, is quite typical for intermetallic inclusions in the structures of materials of the Al–Fe system [12].

As the sintering temperature of samples of this composition in-

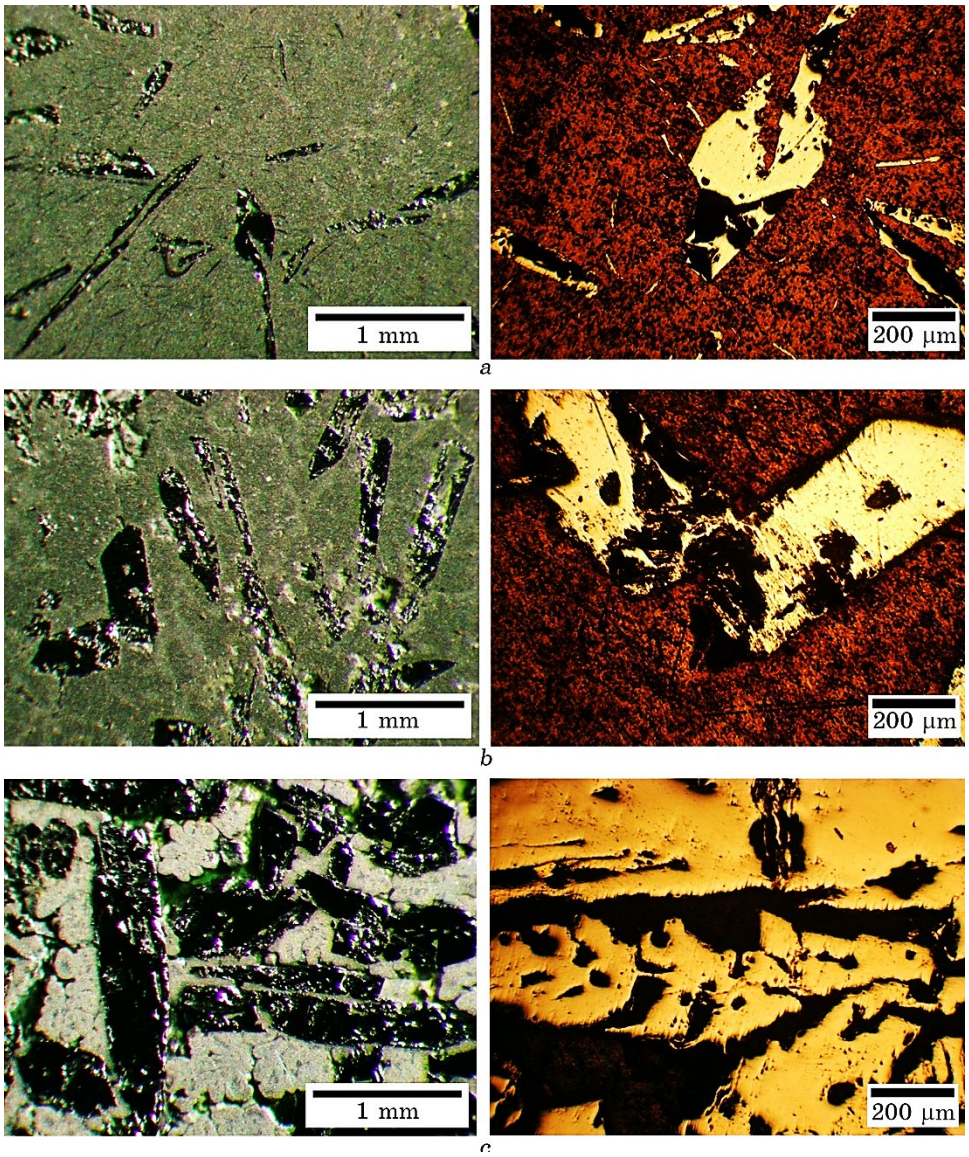

Fig. 3. Structure of samples sintered at 1200°C depending on the material composition (% mass.): 90Al–10(Fe–Ga) (*a*); 70Al–30(Fe–Ga) (*b*); 50Al–50(Fe–Ga) (*c*).

creases, the volume ratio of the matrix and intermetallics in the structure of the material is changed little (Fig. 3, *a*).

At the same time, an increase in sintering temperature contributes to a noticeable increase in the size of intermetallic grains. In general,

with the increase in the sintering temperature to 1200°C, the crystalline discharges can reach a length of 600–800 microns while maintaining the cross-sectional dimensions of the latter at the level of 10–40 microns. At the same time, neither coalescence of contacting crystals nor accommodation of their shape are observed.

As the concentration of Al in the alloy decreases, volume fraction of intermetallics increases to 50–65 and 70–85% (for materials with 70 and 50% mass. Al, respectively). At the same time, multiple separations of a separate light phase are observed in the matrix, the volume fraction of which varies depending on the sintering temperature within 15–25% (see Fig. 2, *b*, *c*).

The results of the x-ray phase analysis of the obtained materials show that on the diffractograms of alloys with a content of 90% mass. Al, the most intense are the lines that coincide with the lines of the spectrum of Al from the planes (111), (200), (211), (311) and (222) (Fig. 4, *a*). In addition, along with the Al(100) and Al(200) lines, peaks of low intensity were detected, located at the angles of 43...45 and 50...53°, which coincide with the spectrum lines of some intermetallics of Fe–Ga and Al–Fe binary systems.

In particular, the spectrum of Fe_3Ga_4 intermetallide is characterized by lines of high intensity in the above-mentioned regions. The base of the ligature alloy, which is included in the original composition of the samples, is precisely this intermetallic [14]; therefore, its partial preservation is quite likely, at a relatively low sintering temperature of 800°C. The interaction of aluminium with the components of the ligature alloy during sintering leads to the formation of aluminides of various compositions. Thus, the most intense peaks in the spectra of double compounds $Al_{3.2}Fe$, $Al_{13}Fe_4$ and Al_2Fe_5 are also located in the regions bordering the Al(111) and Al(200) lines. The lattices of these compounds belong to syngony of low symmetry, and their spectra partially overlap each other. Based on the data of micro-x-ray spectral analysis (see below), it is possible to conclude that the undefined peaks belong to iron aluminides. With increasing of sintering temperature, no significant changes were found in the profile of these spectra, which indicates the constancy of the phase composition of the material.

The diffraction patterns of the samples with content of 70 and 50% mass. Al (Fig. 4, *b*, *c*) are significantly different from the spectra of the material with 90% mass. Al, which indicates significant differences in structural transformations when the composition of the material changes.

In the spectra of samples with 70% mass. Al, the lines belonging to the Al spectrum at the angles of reflection from the (111), (200), (220), (311), and (222) planes are clearly visible, but these lines become less intense. In addition, the detected lines coincide with the lines of the spectrum of the α-Ga lattice.

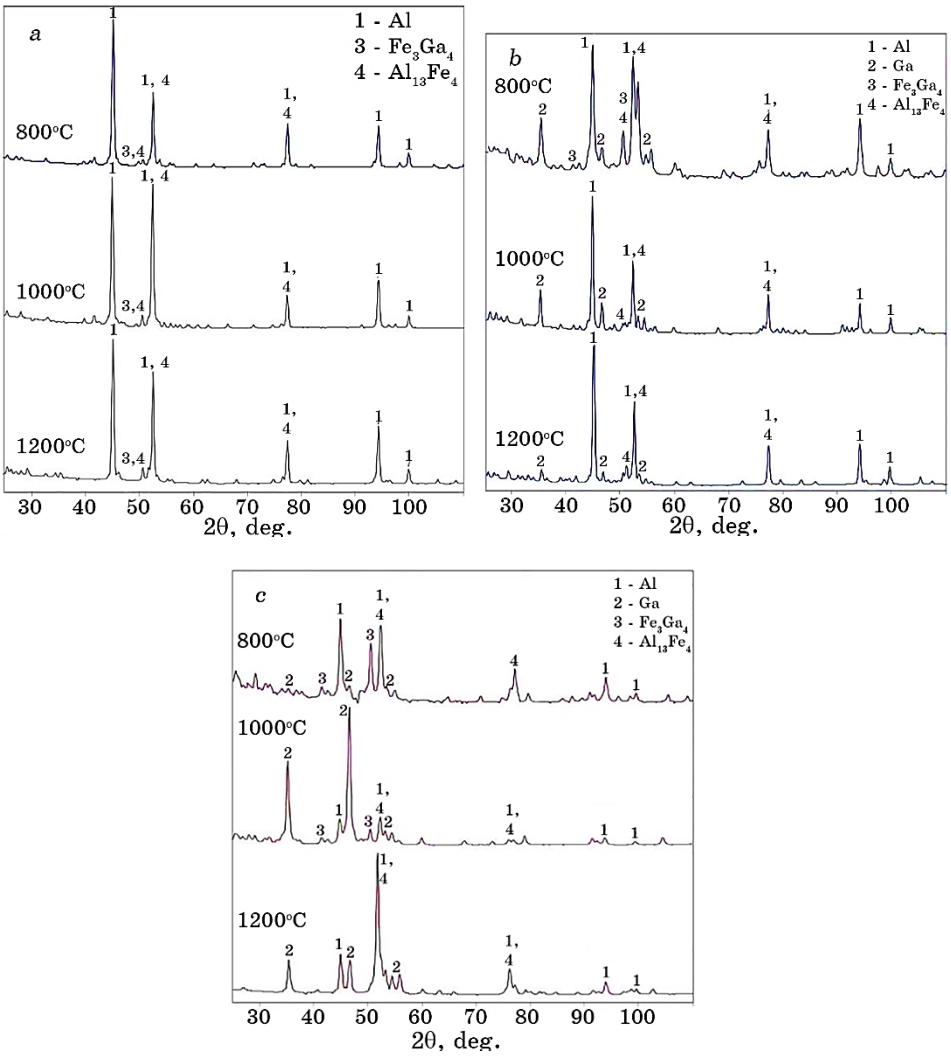

Fig. 4. Diffraction patterns of samples with compositions (% mass.): 90Al–10(Fe–Ga) (*a*); 70 Al–30(Fe–Ga) (*b*); 50Al–50(Fe–Ga) (*c*) depending on sintering temperature.

At the same time, the peaks in the regions of the lines of double intermetallic compounds become more intense. Fe_3Ga_4 peaks are probably present in the spectrum of the sample sintered at 800°C, which practically disappear, when the sintering temperature is increased. On the other hand, the temperature factor affects the intensity of the intermetallic peaks formed during the interaction of aluminium with the components of the ligature alloy (Fig. 4, *b*).

In the spectra of samples containing 50% mass. Al, the intensity of intermetallic lines is higher than the intensity of Al lines, which indicates their predominant number in the sample structure (Fig. 4, *c*).

In particular, the spectrum of the sample sintered at 800°C probably contains lines of Fe_3Ga_4 intermetallide, which is partially preserved in the composition of the material. In the spectra of samples sintered at higher temperatures, individual lines of intermetallics are inferior in intensity to others, which is a sign of a change in their volume in the material structure (Fig. 4, *c*).

Local x-ray microspectral analysis of sintered alloys provides an opportunity to clarify the information obtained by the XRA method. To establish the nature of the distribution of components in the structure of the samples, 7–10 'points' in each of the phases were analysed on different planes of the section. When the concentration of components varied, the number of 'points' was increased to determine the range of fluctuations.

Spectral microanalysis of the phase composition of the sintered samples showed that the basis of their structure is Al-based solid solution, and intermetallics of a ternary composition with a constant ratio of Al to the total amount of the other two components (Fig. 5).

For samples obtained from mixtures containing 70 and 50% mass. Al in a solid solution Ga was detected at the level of its maximum solubility in the binary system Al–Ga (8–9% at. [15]) (see Table 2), while in the material with 90% mass. Al, the solubility of this component in solid solution does not exceed 1% at., which is probably due to a decrease in its total amount in the alloy structure. Another component, namely, Fe, dissolves very weakly in Al-based solution (0.2–0.8 and to 2% at. in materials with 70 and 50% mass. Al respectively) and is practically absent in solid solution in the structure of the material with 90% mass. Al.

Compositions of intermetallic inclusions include Al (70–78% at.), Fe (25...27% at.) and Ga (up 3 to 8% at.) is found in some crystals. The ratio of metal atoms at the points of determining the composition is relatively constant and approaches Al_3(Fe,Ga). In particular, the ratio of Al atoms to the total amount of other metal atoms varies from $Al_{2.3}$(Fe,Ga) to $Al_{3.2}$(Fe,Ga), and changes in the amount of Ga are inversely correlated with changes in the concentration of Fe. It can be assumed that such phase changes occur due to the partial replacement of iron atoms by gallium atoms in the crystal lattice nodes of double intermetallics.

As the sintering temperature increases, in the structure of the samples, the remains of the ligature alloy disappear, while the amount of Ga-based phase allocations increases. Intermetallic inclusions contain all three main components of the system and differ slightly in composition.

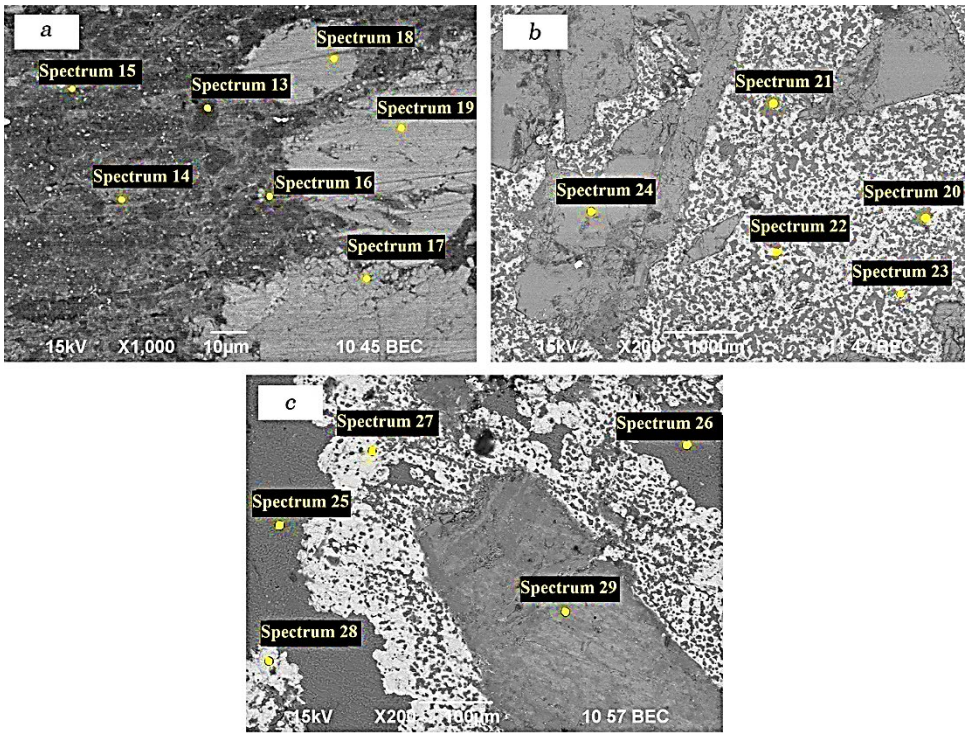

Fig. 5. SEM image of microstructure of samples produced from powder mixture 70 % mass. Al-30 (Fe–Ga), sintered at 800°C (*a*), 1000°C (*b*) and 1200°C (*c*) (regime back scattered electrons).

The obtained results give grounds for assumptions about the mechanisms of mass transfer in the sintering process, which include the interaction of aluminium with the components of the ligature alloy, their dissolution in the melt, and the chemisorption of aluminium atoms on their surface. This creates conditions for further chemical reactions with the formation of solutions based on Al and Ga and iron intermetallics doped with gallium.

The influence of the sintering temperature on the phase changes in the structure of samples containing 90% mass. Al is hardly noticeable, and this indicates a certain thermodynamic stability of the composition formed by various mechanisms in the studied temperature range, while the structures of the samples with 70 and 50% mass. Al show a greater dependence on the temperature factor, which is due to a decrease in the amount of the liquid phase, which is the main accelerator of diffusion processes in the system.

This leads to the fact that the sintering temperature of 800°C does not ensure the completeness of the phase transformations and the

structure of the alloys records the transitional stages of the diffusional redistribution of the components.

Instead, the achievement of a liquid state by the system with an increase in the sintering temperature eliminates all factors affecting the diffusion activity of the components. In this case, the formation of the structure is determined by the phase equilibria of the ternary system

TABLE 2. Local micro-x-ray spectral analysis of structural components of samples with composition (% mass.) 70Al–30(Fe–Ga) sintered at different temperatures.

Spectrum No.	O		Al		Fe		Ga		Local phase composition
	wt.%	at.%	wt.%	at.%	wt.%	at.%	wt.%	at.%	
800°C									
13	17.36	29.67	60.64	61.45	2.65	1.30	19.35	7.59	Al-based solid solution, the remains of the ligature alloy and Ga-based phase
14	1.15	2.20	78.23	88.71	0.36	0.20	20.26	8.89	
15	18.86	41.01	22.05	28.42	8.89	5.54	50.19	25.04	
16	19.86	43.07	20.40	26.24	7.80	4.85	51.93	25.84	
17	7.89	25.48	5.09	9.75	1.56	1.44	85.46	63.33	
18	2.29	4.85	56.41	70.82	35.38	21.46	5.92	2.88	Intermetallic inclusions $Al_{2.3...3}$(Fe,Ga)
19	1.77	3.76	56.61	71.56	35.5	21.68	6.13	3.00	
1000°C									
20	1.53	2.87	80.24	89.28	–	–	18.23	7.85	Al-based solid solution, separation of the Ga-based phase
21	1.58	2.99	78.99	88.58	–	–	19.44	8.43	
22	0.98	4.01	2.21	5.35	–	–	96.80	90.64	
23	1.67	6.43	4.88	11.12	–	–	93.45	82.45	
24	–	–	59.66	75.55	38.39	23.49	1.95	0.96	Intermetallic inclusions $Al_{3...3.2}$(Fe,Ga)
1200°C									
25	1.86	3.52	77.96	87.69	–	–	20.18	8.78	Al-based solid solution, separation of the Ga-based phase
26	1.09	2.08	78.26	88.82	0.23	0.12	20.43	8.97	
27	0.91	3.72	2.01	4.88	–	–	97.09	91.40	
28	0.73	3.05	0.84	2.08	0.44	0.53	98.00	94.34	
29	10.28	20.97	45.62	55.21	27.18	15.89	16.92	7.93	Intermetallic inclusions $Al_{2.3}$(Fe,Ga)

and the conditions of crystallization of the melt during cooling. An increase in the concentration of Fe and Ga in the composition of the material contributes to the formation of intermetallics of variable composition, which are probably solid solutions based on Al_3Fe and Al_5Fe_2 aluminides doped with Ga. At the same time, saturation of the Al solution with Ga is observed, and the excess of this component forms a solid solution, which obviously crystallizes last.

The microhardness of intermetallic inclusions in the structure of samples with 90% mass. Al content is of 6.1…6.5 GPa. This indicator is weakly dependent on the sintering temperature and corresponds to the average level of $FeAl_3$ microhardness (5…8 GPa). The microhardness of the solid Al solution, which is the basis of this material, is also practically unchanged and equal to 0.41…0.45 GPa. For the samples with 70% and 50% mass. Al content, the microhardness of intermetallics varies from 1.3…4.3 (after sintering at 800°C) to 5.3…6.3 GPa (for 1000°C and 1200°C, respectively) depending on the sintering temperature. Fluctuations in microhardness values are obviously explained by the variable composition of intermetallics.

4. CONCLUSIONS

It was established that the samples with the maximum Al content (90% mass.) regardless of the sintering temperature are characterized by the highest density (porosity $\leq$ 1%), due to the presence of a significant amount of liquid phase, which increases the contact area of diffusion interaction. As the concentration of Al in the alloy decreases, the porosity of sintered alloys increases significantly. The samples with 50 and 70% mass. Al, sintered at 800°C, are characterized by significant porosity (40–50%), however, as the sintering temperature increases, their porosity decreases to the level of $\leq$10%.

The base of the phase composition of an alloy with 90% mass. Al is an Al-based solid solution, which contains a certain amount of Ga (up to 3% at.) and intermetallic inclusions of the type $Al_{13}Fe_4$. The influence of the sintering temperature on the phase changes in the structure of the samples is insignificant, which indicates the completion of diffusion redistribution of the components already after sintering at 800°C.

As the concentration of Al in materials decreases, phase transformations in the alloy are more dependent on the sintering temperature. At the same time, the sintering temperature of 800°C does not ensure the completeness of the phase transformations and the structure of the alloys records the transitional stages of diffusion redistribution of the components: in particular, Fe_3Ga_4 peaks, which practically disappear when the sintering temperature is increased. After raising the sintering temperature to 1000°C and 1200°C, intermetallic phases of variable composition are formed in the composition of materials, namely,

Al_3Fe- and Al_5Fe_2-based solid solutions doped with Ga (up to 8% at.). At the same time, the saturation Ga (up to 9% at.) in the Al-based solid solution with is observed, and the excess of this component forms a Ga-based solid solution, which obviously crystallizes last.

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