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Influence of hot forming modes on the structure, mechanical and tribotechnical properties of powder composites of the AL-TIC system

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In this work were investigated the structure, phase composition, mechanical and tribotechnical properties of aluminium matrix composites obtained by hot forging. The results of local phase analysis showed that the hot forgin aluminum-matrix composites consist of an aluminium matrix and dispersed titanium carbide inclusions. The HRB hardness values range from 69.1 to 80 (for samples with 45-50 Al (wt. %)) and up to 73.7-76 (55-60 Al (wt. %)) and compressive strengths are σ_b = 450 - 600 MPa; $\sigma_{0.2}$=350 - 509 MPa depending on the composition and preheating temperature. AMCs with sufficiently high strength are capable of plastic deformation ε-5.3-9.19%. The results of tribotechnical studies have shown that when testing a composite paired with copper, the samples without excess carbon wear out intensively due to the mutual adhesion of the aluminium matrix of the composite and the copper. In turn, the composites containing residual carbon showed a resistance of more than 100 times in relation to the compositions without excess carbon.

Keywords: aluminium matrix composites, hot forging, mechanical properties, tribotechnical properties, powder composites.

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Introduction

In recent decades, metal matrix composites (MMCs) have emerged as new engineering materials with superior properties compared to monolithic materials. The favourable properties that are typically exhibited by metal matrix composites include light weight, high elastic modulus, high strength, high thermal stability, resistance to elevated temperatures, improved wear resistance, a low thermal expansion coefficient, and extended fatigue life [1,2]. Due to their exceptional blend of properties, advanced matrix composites (AMCs) have risen to prominence as advanced materials, playing a crucial role in diverse industries such as aerospace, automotive [3] (i.e., pistons and connecting rod [4]), defence, and various engineering sectors [5]. Aluminium [6–8], and magnesium are the usual matrices used for lightweight MMCs [9].

Aluminium and aluminium alloy based metal matrix composites have attracted considerable interest in the field of automobile and aerospace industries by virtue of excellent properties such as high strength, specific modulus, low thermal expansion coefficient and low weight. In particular AA6061 and its composites cover a wide range of application from appliances to aerospace structures. Most widely used ceramic reinforcements are SiC, TiC, B_4C, TiB_2 and Al_2O_3 due to their low density, high hardness and chemical inertness. Conventional metal matrix composites were fabricated using liquid or powder metallurgy in which reinforcing materials were prepared separately and added to molten metal or powder form. The composites fabricated by such processes are termed as ex situ metal matrix composites. Ex situ composites suffer from several drawbacks which include poor wettability between reinforcement and matrix, uniform dispersion of reinforcement and the nature of reinforcement/matrix interfaces. Poor wettability of reinforcement with matrix material results in weak interface bonding. Weaker adhesion at the reinforcement/matrix interface can lead to decrease in strength of the composites. Likewise formation of clusters of reinforcement during solidification processing has detrimental effect on mechanical properties because these clusters act as stress concentrations. Owing to such issues the industrial development of metal matrix composites has remained limited [10]. To address the drawbacks of ex situ metal matrix composites significant efforts have been made towards the development of in situ metal matrix composites. To address the drawbacks of ex situ metal matrix composites significant efforts have been made towards the development of in situ metal matrix composites. In this type, the reinforcements are synthesized by in situ reactions of different components during composite fabrication. The in situ reaction can be between the liquid–gas, solid–solid reaction process between metal–metal particles or metal–ceramic particles or between the mixed salts to produce the required in situ reinforcements. The composites produced by in situ process have exhibited advantages like, uniform dispersion of reinforcement with fine size, excellent bonding between reinforcement and matrix with clean interface and good thermodynamical stability of reinforcement. Many metal matrix materials like aluminium, copper, titanium and nickel with in situ developed reinforcing phases like TiB_2, TiC, Al_4C_3 and Al_2O_3 have been reported so far [11–13]. The production of TiC by in situ reaction between carbonaceous gas or graphite and aluminium or Al–Ti alloy melts has been considered in many works. Li et al. [14] produced TiC particles by introducing a mixture of Ti and carbon powders in aluminium melt maintained at 900–1000 °C. Ti and carbon powders in the stoichiometric ratio of 1:1 were used for making composites to avoid the formation of Al_4C_3. Composites prepared at 1000°C with Na_3AlF_6 displayed best TiC recovery with fine size and uncontaminated macrofracture. Shyu et al. [15] reported synthesis of TiC by reacting the molten Al–Cu–Ti with carbon carrying gas at 1127 °C. The volume fraction of TiC formed was around 6%. Effect of TiC on mechanical and tribological properties was studied. The study revealed increase in tensile strength by 18% of composite suggesting good bonding between TiC and aluminium alloy matrix. The dry sliding wear test conducted showed that TiC helped in reducing the wear rate of composite by a factor of eight compared to that of the aluminium alloy. Out of all ceramic reinforcements synthesized by in situ process TiC is particularly attractive due to its high melting point (3160°C), high hardness, low coefficient of thermal expansion and excellent wear resistance. TiC also exhibits good wetting characteristics with aluminium and its alloys resulting in good adhesion at the interface and low chemical reactivity [16,17].

As can be seen from the literature, a comprehensive study of various metal-matrix composites reinforced with titanium carbide has been carried out, mainly from aluminum alloys such as: AA6061 [18], AA7075 [19] and AA2024 [20,21]; which were obtained by casting methods. However, there is a limited number of studies devoted to their microstructural analysis, analysis of hardness and wear characteristics. In turn, casting methods have characteristic disadvantages associated with the uneven distribution of hard inclusions in the composite, but studies have shown that the use of powder metallurgy and thermal synthesis of titanium carbide allows for a uniform distribution of titanium carbide in the matrix. As noted in the studies, both methods also have a common disadvantage in the form of shells (porosity) in the resulting composite, which negatively affects the mechanical properties of the resulting composites.

To obtain a compact and reduce the number of structural defects, hot plastic deformation methods are traditionally used. To date, very little information is available on the hot deformation of titanium carbide-reinforced aluminum matrix composites. Therefore, this work presents data on the influence of hot deformation modes on the structure and functional properties of a pre-synthesized aluminum matrix composite of the Al–TiC system.

There is a need to develop lightweight composite powder materials that would have high performance properties, especially high wear resistance. This paper proposes to resolve this issue by using aluminium matrix composite materials dispersion-hardened with titanium carbide. The aim of this work is to study the structure, phase composition, mechanical and tribotechnical properties of aluminium matrix composites obtained by hot forgin. The objective will be achieved by exploiting the effects of obtaining the desired dispersed phases during thermal synthesis, which will make it possible to control the grain growth rate and artificially create heterogeneity in the composite, thereby forming a fine-grained microheterogeneous structure with high physical, mechanical and tribotechnical properties. A new approach to the creation of such powder materials is that dispersion-modifying additives (in particular, TiC) are formed by thermal synthesis from elemental powders.

I. Materials and methods

Aluminium (PA-4), titanium (PTC-80) and carbon powders were used as starting components to prepare the charge for further thermal synthesis. To evaluate the effect of the ratio of the mixture components on the structure of the synthesised ligatures, four compositions of the initial charge were selected, including different aluminium contents (from 45 to 60 wt. %), as well as two compositions with an excess of carbon in the initial charge. The ratio of carbon to titanium corresponded to the stoichiometric composition (Table 1).

Table 1.

Component content of the initial mixtures of Al-Ti-C composites

No. of the mixture	Elements content, wt. %		
	Al	C	Ti
1	45	11	44
2	50	10	40
3	55	9	36
4	60	8	32
5	50	15	40
6	55	14	36

The starting mixtures were compacted at a pressure of 300 MPa, and the thermal synthesis was carried out in a vacuum induction heating furnace at a temperature of

950 °C and a holding time of 1 hour at a heating rate of 5-10 deg/min. The studies of the structure and phase composition of the synthesised aluminium matrix composites are given here [22]. The billets obtained after thermal synthesis were heated in a laboratory electric furnace to a temperature of 520°C and 630°C for 20 min, a protective mixture of silicate glue and graphite was applied to the heat (for protection), and then subjected to hot forging (HS) on a PG-60 hydraulic press, in a semi-closed die, the scheme of which is shown in Fig. 1.

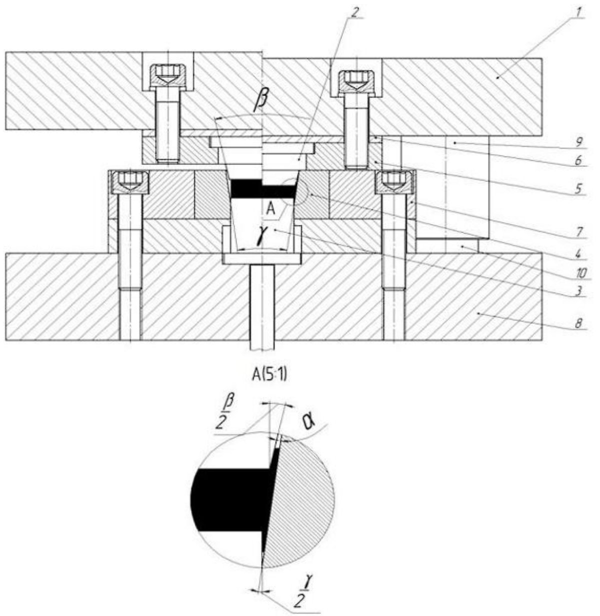

Fig. 1. Semi-closed die for hot forging.
1 - upper plate; 2 - upper punch; 3 - lower punch;
4 - matrix; 5 - punch holder; 6 - backing plate; 7 - bandage;
8 - lower plate; 9 - bushing; 10 – columns.

The microstructure of the obtained synthesised composites was investigated using a Tescan Vega 3 LMU scanning electron microscope manufactured by the Czech company Tescan Brno s.r.o. The Tescan Vega 3 LMU provides images of the structure with high spatial resolution and depth of field in reflected (BSE) and secondary (SE) electrons, as well as the information on the chemical composition and structure. The samples were etched in a 40% NaOH solution.

The hardness of the AMCM was determined using a Novotest TS-BRV device according to the Rockwell method (HRB scale).

The uniaxial compression tests were carried out on a CERAMTEST universal testing machine. For this purpose, the specimens with dimensions of BxHxL=5×5×8 mm were made. Special programs for calculating compression load curves allow us to calculate the basic mechanical characteristics of materials for this type of test. Compression tests helped determine the proportionality limit $\sigma_{0.01}$, yield strength $\sigma_{0.2}$, and tensile strength $\sigma_{в}$.

The tribotechnical characteristics of the composites were tested in conjunction with a copper wheel (hardness H_v - 113 kg/mm^2) on an end friction machine, according to the shaft-plane scheme. The prismatic samples were tested in the dry friction mode under a load of 30 N, at a speed of 2 m/s and 2 km. The copper counterbody was a washer with an outer diameter of 40 mm and a width of 12 mm. The sliding speed was 2 m/s at a load of 30 N and a distance of 2 km.

II. Results and discussion

Table 2 shows the values of the theoretical density of the mixture and the density of the AMCM after hot forging at different preheating temperatures. The density of the synthesised material was determined by the pycnometric method, while the density of the compacted composite was determined by the hydrostatic weighing method.

Table 2.

Values of the theoretical density of the mixture and the density of AMCM after hot forging depending on the preheating temperature

No.	Composition, wt. %	γ, g/cm^3 theoretical	530 °C	620 °C
			γ, g/cm^3 after HS	γ, g/cm^3 after HS
1	45Al-11C-44Ti	3.5	3.1	3.3
2	50Al-10C-40Ti	3.4	3.22	3.27
3	55Al-9C-36Ti	3.27	3.04	3.19
4	60Al-8C-32Ti	3.33	3.02	3.05
5	50Al-15C-40Ti	3.38	3.12	2.96
6	55Al-14C-36Ti	3.36	3.26	2.95

The study of samples obtained after hot forging of various compositions showed that the microstructure of the composites is characterised by the presence of clearly defined two phases: the basis is an aluminium-based matrix in which titanium carbide inclusions of various sizes are distributed, depending on the composition of the composite, from 0.1 to 0.5 µm (Fig. 2).

In the course of the work, we studied the effect of preheating temperature on the structure of aluminium matrix composites after HS. Table 2 shows that the density values for materials of different compositions differ depending on the preheating temperature.

For the composite with the lowest amount of aluminium 45 (wt. %), the best compaction is achieved at a heating temperature of 620 °C. At this temperature, the composite matrix is in a viscous-fluid state, which contributes to the compaction process by intensifying the plastic flow of the material during hot forming and moving solid inclusions in the direction of intensive flow of the material of the composite matrix. For composites with an aluminium content of 50-60 (wt. %), the preheating temperature has almost no effect on the density value, but the results of electron microscopic studies indicate that with an increase in temperature to 630 °C, compaction is much more intense due to the viscous flow state of the composite matrix (Fig. 2, b - d).

In turn, for materials with an excess of carbon, compositions No. 5 and 6 (Table 2), the intensification of compaction processes is observed to a lesser extent when the heating temperature is increased to 620 °C.This effect can be explained by the presence of free graphite inclusions that impede the viscous flow of the material, while the movement of carbide particles occurs mainly in the direction of the tool stroke. This, in turn, leads to the formation of a hard-to-deform carbide frame and prevents compaction of the composite during deformation. Furthermore, the analysis of the SEM image of sample No. 6 - 55Al-14C-36Ti at different heating temperatures shows that as the temperature increases from 530 °C to 620 °C, the compaction of the composite increases similarly to that of composites without excess carbon. As

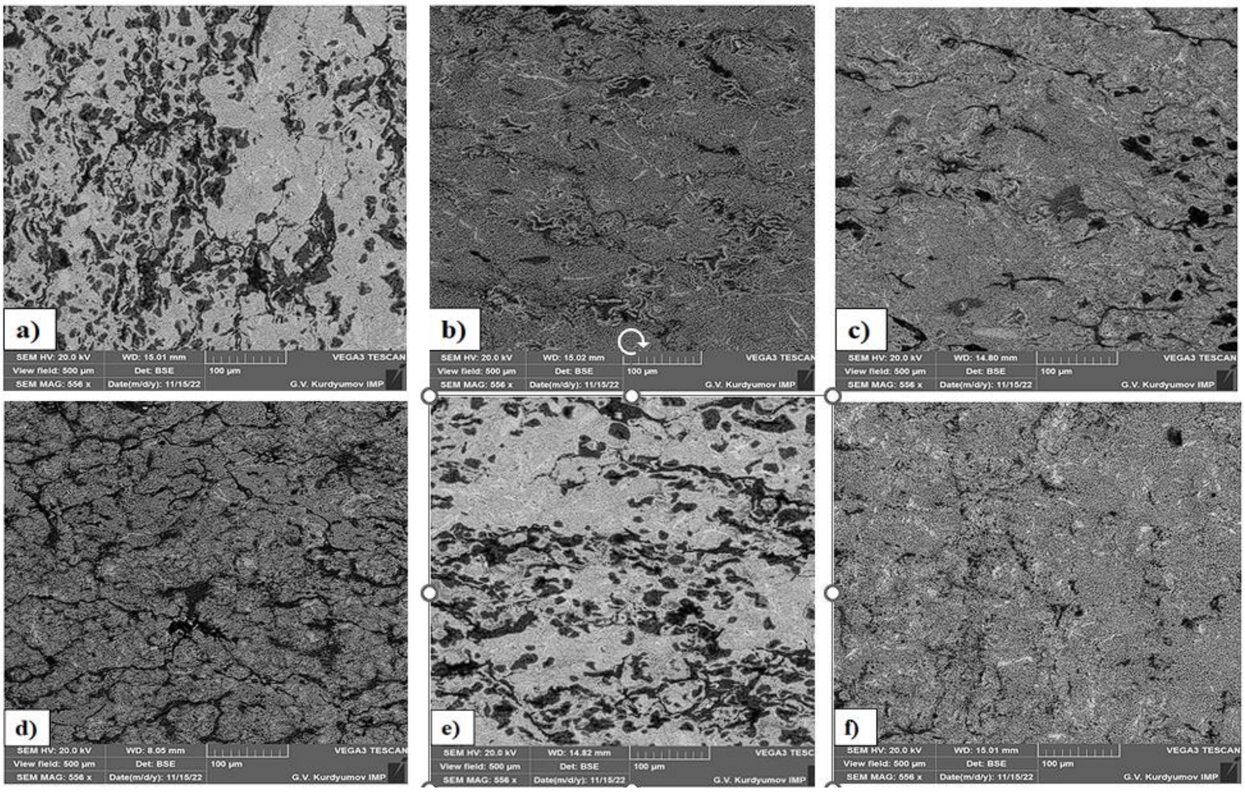

Fig. 2. SEM image of aluminium matrix composites obtained by hot forging at a preheating temperature of 520 °C. a) - 45Al-11C-44Ti; b) - 50Al-10C-40Ti; c) - 55Al-9C-36Ti; d) - 60Al-8C-32Ti; e) - 50Al-15C-40Ti; f) - 55Al-14C-36Ti.

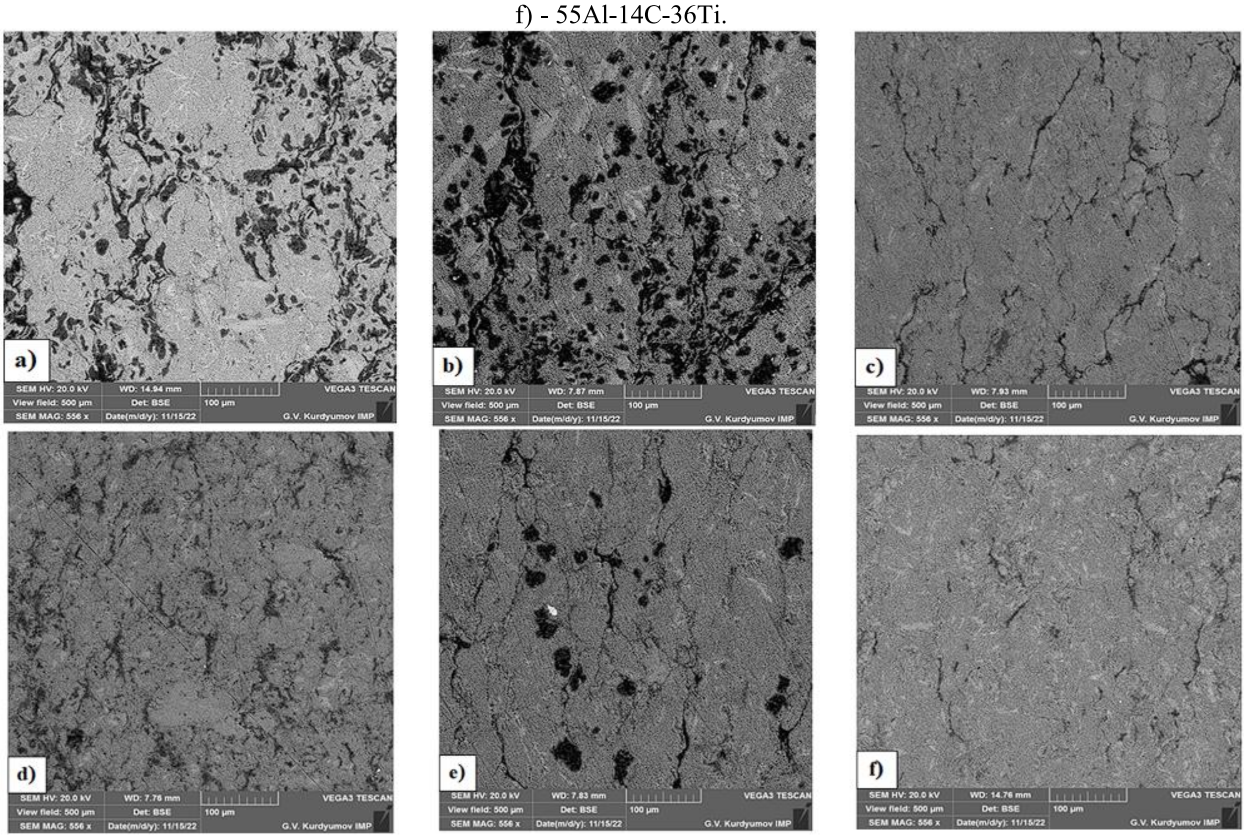

Fig. 3. SEM image of aluminium matrix composites obtained by hot forging at a preheating temperature of 630 °C. a) - 45Al-11C-44Ti; b) - 50Al-10C-40Ti; c) - 55Al-9C-36Ti; d) - 60Al-8C-32Ti; e) - 50Al-15C-40Ti; f) - 55Al-14C-36Ti.

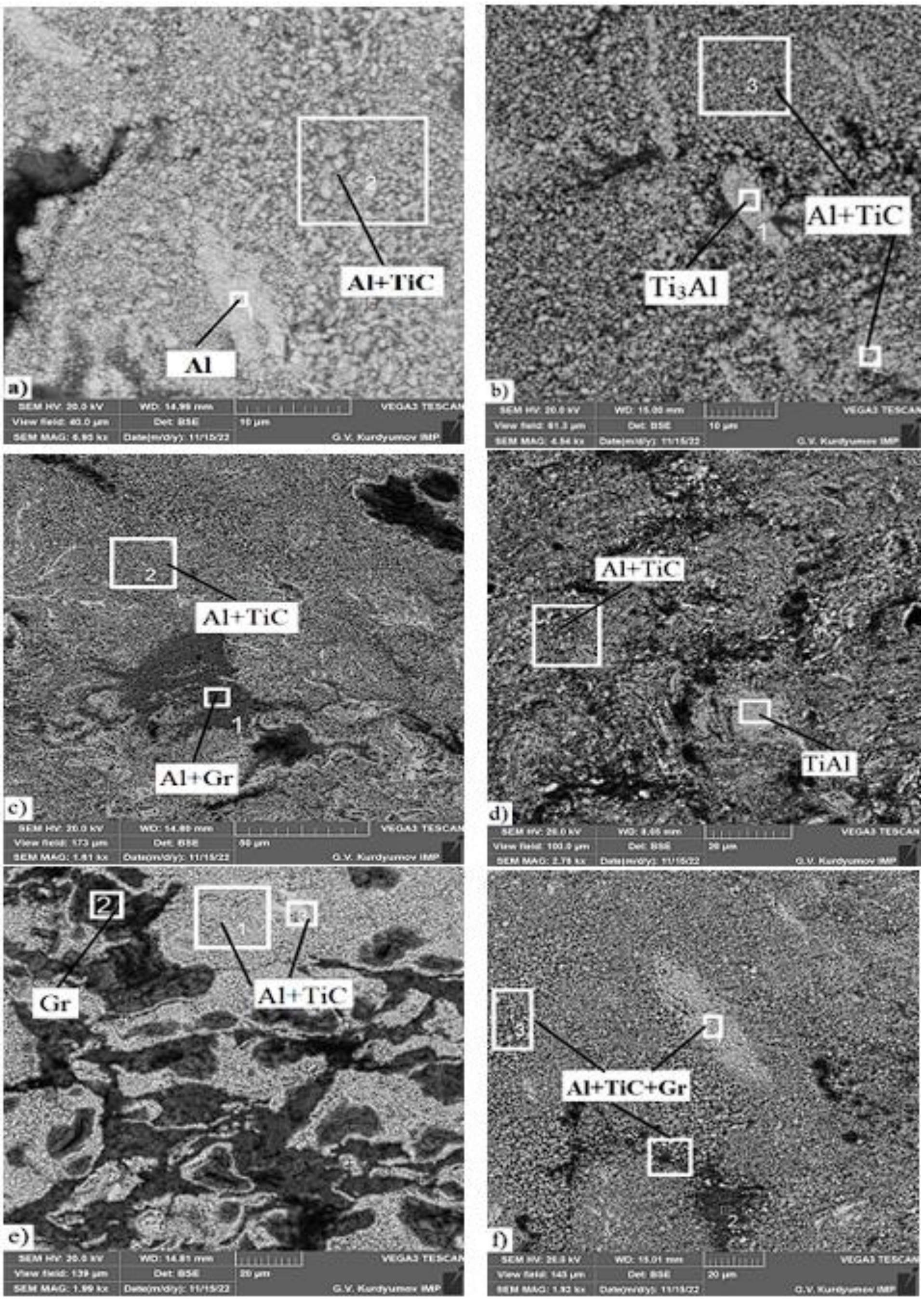

Fig. 4. Local X-ray spectral analysis of aluminium matrix composites obtained by hot forging. a) - 45Al-11C-44Ti; b) - 50Al-10C-40Ti; c) - 55Al-9C-36Ti; d) - 60Al-8C-32Ti; e) - 50Al-15C-40Ti; f) - 55Al-14C-36Ti.

a result, the compaction of the composites in this system is directly dependent on the amount of plastic matrix that facilitates the movement of the carbide inclusions in space.

Further analysis of the microstructure images (Fig. 3) showed that an increase in the plasticity of the matrix phase leads to a change in the nature of the distribution of free carbon in composites No. 5 - 50Al-15C-40Ti and No. 6 - 55Al-14C-36Ti. Contrary to the composite with a lower aluminium content (No. 5, Fig. 3, e), in which, upon compaction, the carbon inclusions completely fill the pore cavity and acquire a rounded shape of 10-20 μm, in the composite with a higher aluminium content and a lower content of the carbide component No. 6 (Fig. 3, f), it is observed that the pore cavity and the free carbon inclusions expand in the direction of deformation. This leads to a decrease in the anisotropy of the composite and increases the uniformity of the distribution of carbon inclusions up to 5 μm in size.

The phase composition of the aluminium matrix composites (Figs. 4 and 5) after compaction was also investigated, depending on the material composition, by micro-X-ray spectral and X-ray phase analyses. The microstructures clearly show that with an increase in aluminium content from 55 to 60 (wt. %), the size of titanium carbides decreases compared to composites with a lower aluminium content (45-50 Al wt. %) and is on average 0.2 μm. In the 45Al-11C-44Ti and 50Al-10C-40Ti composites, the size of the carbide component ranges from 0.4 to 0.6 μm.

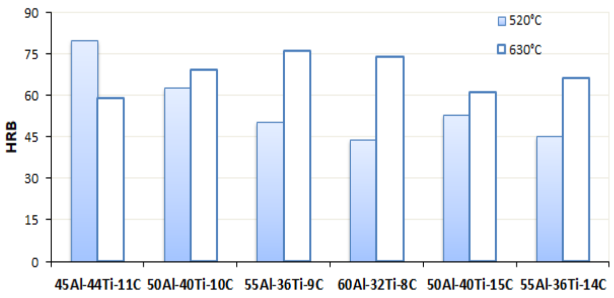

Fig. 5. Influence of heating temperature and composition of the initial charge on the hardness of aluminium matrix composites.

The SEM microstructure images (Fig. 4, e, f) clearly show the difference between the structures of composites with excess carbon after hot forging of 50Al-15C-40Ti and 55Al-14C-36Ti compositions. The structure of the samples consists of an aluminum matrix, titanium carbide, and evenly distributed free carbon throughout the composites.

However, in the sample of 50Al-15C-40Ti composition, free carbon is recorded on the structure in the form of conglomerates (dark grey specks), and in the sample of 55Al-14C-36Ti in the form of finely dispersed inclusions. The microstructural analysis also shows that the size of carbide inclusions for composites with excess carbon is slightly larger than for composites without excess carbon, ranging from 0.4 to 1 µm.

Fig. 5 shows the hardness values of aluminium matrix composites of different compositions depending on the change in preheating temperature for hot forming. As can be seen from the figure, an increase in the heating temperature from 520 °C to 630 °C leads to an increase in hardness from 44 to 76 HRB, depending on the composition. However, the hardness of the composite with the lowest aluminium content, 45Al-11C-44Ti, decreases from 80 HRB (520 °C) to 59 HRB (630 °C) with increasing heating temperature, since this sample does not have the required amount of plastic component to move the solid inclusions in space, resulting in the formation of a hardly deformable carbide-frame structure and large residual porosity, which leads to a decrease in the hardness of the composite as a whole.

Compressive mechanical properties of compacted aluminium matrix composites were tested, during which the tensile strength (σ_b), yield strength ($\sigma_{0.2}$), and relative strain (ε) of the materials were determined (Fig. 6). All the studied composites demonstrated high compressive properties, namely σ_b = 450-610 MPa; $\sigma_{0.2}$ = 350-500 MPa. Fig. 6 (d) shows that the composites with 55-60 Al (wt. %) at a sufficiently high strength are capable of plastic deformation ε - 5.3-9.19%. However, materials with an excess of carbon are characterised by low ductility (ε - 0.6-1.6%) due to the inclusion of free carbon, which acts as stress concentrators and contributes to the fracture of the composite.

The study of antifriction properties was carried out on the M-22M friction machine using the shaft-plane scheme. The tests were carried out in conjunction with copper. The copper counterbody was machined from a copper bar hardened by precipitation on flat strikes (with a deformation of 70 %) and was a washer with an outer diameter of 40 mm and a width of 12 mm. The sliding speed was 2 m/s at a load of 30 N and 2 km. The results of tribotechnical studies of aluminium matrix composites on a copper counterbody showed that materials with an aluminium content of 45 to 60 (wt. %) exhibit significant linear wear of the composites from 1032 to 1044 mg/km. As can be seen from Table 3, the friction path for the above materials is only 0.3 km, as further critical wear occurred.

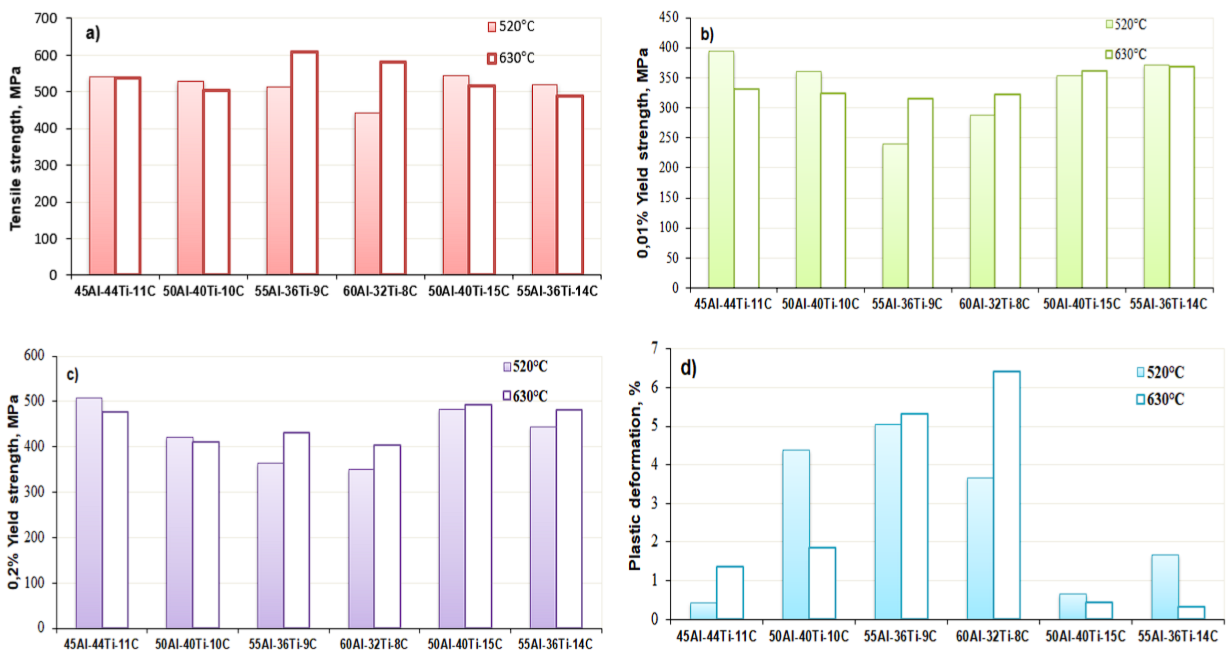

Fig. 6. Mechanical properties at temperatures 520 °C and 630 °C of different hot-stamped aluminium matrix composites, based on the results of a compression test.

Table 3.

Tribotechnical studies of the copper-composite friction pair of aluminum matrix composites

No.	Sample composition, wt. %	Friction path, km	I_m, mg/km	I_m counterbody, mg/km	I_l, µm/km	Friction coefficient, µ
1	45Al-44Ti-11C	0.3	52.6	85.5	1032	0.73
2	50Al-10C-40Ti	0.3	64.2	76.1	1039	0.7
3	55Al-9C-36Ti	0.3	76.3	65.4	1044	0.71
4	60Al-32Ti-8C	0.3	81.7	57.3	1039	0.7
5	50Al-40Ti-15C	2	+0.3	1.05	19.4	0.24
6	55Al-36Ti-14C	0.8	62.6	50.3	204.6	0.37

Table 3 shows that with an increase in the amount of aluminium, the mass wear of the counterbody decreases, as the aluminium matrix sticks to the copper counterbody, after which the friction pair changes from ‘copper-composite’ to ‘composite-composite’, accompanied by active wear. In composites with the lowest aluminium content (45Al-44Ti-11C), on the contrary, the composite begins to wear the counterbody, which leads to catastrophic wear of the counterbody.

The study of the tribotechnical properties of samples with excess carbon (No. 5 and No. 6) in a copper-composite friction pair showed quite different wear results. Composite No. 5 showed the best result among all the above compositions, with a resistance increase of almost 100 times. This can be explained by the presence of free carbon in the material (in the form of conglomerates), which, after thermal synthesis and subsequent hot forging, was evenly distributed over the structure of the composite (Fig. 4, e) and acts as a solid lubricant, reducing the coefficient of friction and increasing the antifriction properties. Table 3 shows that the 50Al-40Ti-15C composite wears the copper counterbody by only 1.05 mg/km, while abrasive wear of the counterbody surface by titanium carbide and subsequent adhesion of the chips, formed on the composite surface, occur, which in turn leads to an increase in the composite weight by +0.3 mg. As for composite No. 6, the linear wear of the material is significantly lower than that of the other samples without excess carbon, but higher than that of the similar sample with excess carbon No. 5. This dependence can be explained by the fact that with an increase in the aluminium content by 5%, free carbon (in the amount of 5 wt. %) is not enough to form a sufficient lubricating film on the composite-counterbody contact surface.

Conclusion

1. The structure and phase composition of the composites after hot forging were investigated. The results of local phase analysis showed that the composites consist of an aluminium matrix and dispersed titanium carbide inclusions. However, in the samples of 50Al-10C-40Ti and 60Al-32Ti-8C compositions, in addition to aluminium and titanium carbide.
2. The effect of hot forging temperature on the compaction of aluminium matrix composites was determined. The studies have shown that heating the samples to 630 °C has a positive effect on the compaction of the synthesised sinter. The compaction of the composites of this system is directly dependent on the amount of plastic matrix that contributes to the movement of the carbide inclusions during deformation. According to the SEM results, it was found that when the samples are heated to 520 °C, the shape of residual pores is almost spherical, and at 630 °C they are volumetrically elongated in the direction of visco-fluid matrix flow.
3. The studies have shown that the samples of all compositions have compacted after hot forming. The HRB hardness values range from 69.1 to 80 (for samples with 45-50 Al (wt. %)) and up to 73.7-76 (55-60 Al (wt. %)), depending on the composition and preheating temperature. The composites with excess carbon have hardness values of 61-66 HRB. The compressive mechanical properties of hot stamped aluminium matrix composites have been tested. All composites that have been tested showed high compressive properties, namely σ_b = 450 - 600 MPa; $\sigma_{0.2}$=350 - 509 MPa. The plastic deformation ε - 5.3 - 9.19% is possible for composites with 55-60 Al (%, wt) at a sufficiently high strength. However, materials with an excess of carbon are characterised by low ductility due to the inclusion of free carbon, acting as stress concentrators ε - 0.6 - 1.6%.
4. The results of tribotechnical studies have shown that when testing a composite paired with copper, the samples without excess carbon wear out intensively due to the mutual adhesion of the aluminium matrix of the composite and the copper. In turn, the composites containing residual carbon showed a resistance of more than 100 times in relation to the compositions without excess carbon.

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Вплив режимів гарячого деформування на структуру, механічні та триботехнічні властивості порошкових композитів системи AL-TIC

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У роботі досліджено структуру, фазовий склад, механічні та триботехнічні властивості алюмінієвих матричних композитів, отриманих методом гарячого кування. Результати локального фазового аналізу показали, що гарячештамповані алюмоматричні композити складаються з алюмінієвої матриці та дисперсних включеннями карбіду титану. Значення твердості за школою HRb коливаються від 69,1-80 (для зразків із 45-50%, (мас.) Al) та до 73,7-76 (55-60 %, (мас.) Al) та міцності при стисненні становлять $\sigma_в$=450-600 МПа; $\sigma_{0,2}$=350-509 МПа в залежності від складу та температури попереднього нагріву. AMCs при достатньо високій міцності здатні до пластичного деформування ε-5,3-9,19%. Результати триботехнічних досліджень показали, що при випробуванні композиту в парі з міддю зразки без надлишкового вуглецю інтенсивно зношуються через взаємну адгезію алюмінієвої матриці композиту та міді. В свою чергу композити які містять залишковий вуглець показали стійкість в більше чим в 100 раз по відношенню до складів без надлишкового вуглецю.

Ключові слова: алюмоматричні композити, гаряче штампування, механічні властивості, триботехнічні властивості, порошкові композити.