
STRUCTURE, PHASE TRANSFORMATIONS,
AND DIFFUSION

Structure and Thermophysical Properties of Aluminum-Matrix Composites

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Abstract—The microstructure and thermophysical properties of aluminum-matrix composites have been studied, in which a granulated Al–Zn–Mg–Cu alloy has been used as the matrix, and SiC particles taken in the amounts of 10, 20, and 30 vol % have been used as the filler. It has been shown that, with an increase in the amount of the filler, the temperatures of the solidus and liquidus of the composites and the values of the thermal expansion coefficient and density increase, whereas the heat capacity, thermal conductivity, and thermal diffusivity decrease. The heat capacity of the composite depends on the amount of the filler: upon heating from 25 to 500°C, the heat capacity of the composite with 10 vol % SiC increases by only 16%, while that of the composite with 20 vol % SiC increases by 19%; and, at 39 vol % SiC, it increases by 36%.

Keywords: aluminum-matrix composite, aluminum, silicon carbide, particles, liquidus, solidus, heat capacity, thermal conductivity, thermal diffusivity, density, microhardness

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INTRODUCTION

The composite materials with an aluminum matrix find wide application in the production of articles in the automobile and transport engineering, power electrical engineering, aviation and space-rocket engineering [1–3]. A permanent interest is paid to composite materials with an aluminum matrix and silicon carbide as a filler because of their high strength properties, good technological treatability, and relatively low cost [4–9]. As the metallic matrix, commercial aluminum or aluminum alloys such as AMg6, B95, D16, etc. are usually employed. Until now, the investigations of aluminum-matrix composites (AMCs) were mainly concerned with their production methods and the estimation of their structure and properties. Now, studies devoted to the development of technological processes for producing specific details from billets are of growing interest, as well as studies that consider specific changes in the structure and properties of AMCs upon cold or hot plastic deformation [6–8, 10, 12]. A special site among these works belongs to those in which the behavior of composite materials is simulated in various methods of external action. The validity of the results of the simulation is mainly determined by the way of specifying the representative volumes of the material and the computational models employed [13]. Therefore, an important problem is the determination of the shape of the filler particles, of the properties of the AMC constituents on the microlevel and the estimation of their interaction upon heating or

upon mechanical loading on a micro-, meso-, and macrolevel, as well as the determination of the effect of the amount of the filler on the complex of thermophysical properties, which determine the methods and regimes of hot technological treatment.

This work was aimed at the determining of the structural state of the AMC with a content of the filler of 10, 20, and 30 vol %, the estimation of the effect of the amount of SiC particles on the density, heat capacity, thermal diffusivity, thermal conductivity, and the linear thermal expansion coefficient of the composite material under consideration.

EXPERIMENTAL

The investigations were carried out on samples made of composite materials with a metallic matrix and a filler in the form of silicon-carbide particles with a content of 10, 20, and 30 vol %. The samples were prepared by the method of hydrostatic pressing according to the technology developed at the VIAM (All-Russia Institute of Aviation Materials, Russia). Instead of commercial aluminum of grade A8 used as the matrix in the AMCs that were studied earlier in [10, 11], we here used granulated high-strength aluminum alloy of the Al–Zn–Mg–Cu system of the following chemical composition (wt %): 5–7 Zn, 1.8–2.8 Mg, 1.4–2 Cu, to 0.5 Fe, to 0.5 Si, 0.2–0.6 Mn, 0.1–0.25 Cr, to 0.05 Ni, to 0.05 Ti, and Al for balance. It has a high ultimate strength (600–700 MPa) and a

close yield stress. This alloy is not heat-resistant; in the case of long-term exploitation, it can be used at temperatures no higher than 100–120°C; therefore, it is used to produce heavily loaded structures that are mainly exploited under compressive conditions (details of housing, stringers, frame, spars, etc.).

The microstructure of the AMCs was studied on transverse sections using a NEOPHOT-21 optical microscope. To determine the shape of the particles of the SiC filler, the samples were dissolved electrolytically in a 45% solution of sodium hydrochloride with a subsequent addition of hydrochloric acid until the formation of soluble salts of aluminum. The deposit was filtered using a preliminarily weighed filter paper, washed by distilled water, and dried. The shape of the particles was determined using a TESCAN VEGA II XMU scanning electron microscope equipped with wave- and energy-dispersive attachments (Oxford) for electron-microprobe analysis.

In this work, we have studied the temperature dependences of thermophysical properties of the prepared AMCs. The measurements of the thermal effects and characteristic temperatures of the materials were performed using the method of differential scanning calorimetry (DSC) using an STA 409 Netzsch-Geratebau-GmbH (Germany) thermoanalyzer. The values of the heat capacity and thermal diffusivity of the samples were determined by the laser-flash method using a Netzsch LFA 457 system to characterize the thermophysical parameters of solids. Based on the results of measuring the heat capacity, thermal diffusivity, and density, the coefficient of thermal conductivity was calculated via the well-known relationship

$$\lambda = c_p \alpha \rho, \quad (1)$$

where λ is the thermal conductivity, α is the coefficient of thermal diffusivity, ρ is the density, and c_p is the heat capacity. The density was used in the calculations as a constant quantity determined at room temperature. The density was determined by the hydrostatic method according to the ASTM B311-93 standard by weighing the samples in air and in distilled water with a density of 998 kg/m³. The weighing was performed using an OHas Pioner PA 214 analytical balance. The density of AMCs was calculated via the formula

$$\rho = \frac{m_1}{m_1 - m_2} \rho_w, \quad (2)$$

where m_1 is the mass of the sample determined in air, m_2 is the mass of the sample in water, and ρ_w is the density of the distilled water. The average error of the measurements in the temperature range of 25–500°C was 2.5% for the thermal diffusivity, 5% for the heat capacity, and 1.5% for the density. The error in the measurements of the thermal conductivity was 5.7%.

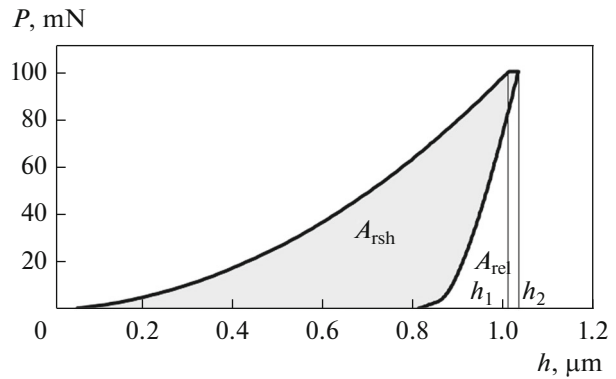


Fig. 1. Indentation curve: P is the load at the indenter; h is the depth of the penetration of the indenter; A_{rsh} is the work due to forces of residual shape change; A_{rel} is the work due to relaxation forces; the horizontal segment of the curve from h_1 to h_2 is the segment corresponding to creep.

The linear thermal expansion coefficient (LTEC) was determined using a Linsis L75VD1400C vertical dilatometer with a quartz pusher according to the ASTM E831-14 standard. A differential method of the measurement of the increment in the sample length was used, with two displacement transducers, one of which is related to the quartz frame that transfers the thermal expansion of the sample and the second detects the position of the unit of the gages relative to the quartz system. The absolute error of the device is $\pm 4 \times 10^{-8} \text{ K}^{-1}$ at a confidence level of 0.95. The microhardness of the components of composites was measured using a FISHERSCOPE 2000 XYm meter with a system of microindentation, which made also possible to measure the reduced elasticity modulus E from which the normal elasticity modulus E_{elast} was calculated via the formula

$$E_{\text{elast}} = E(1 - \mu^2), \quad (3)$$

where μ is the Poisson ratio (for SiC, $\mu = 0.187$ [14]; for the aluminum matrix, $\mu = 0.34$ [15]). The load on the indenter was varied in the range of 0.05–1.96 N.

The total work due to the penetration of indenter A , the work done by relaxation forces A_{rel} , and the work of residual shape change A_{rsh} have been determined. The total work spent to penetrate the indenter is determined by the area under the loading curve; the work due to the relaxation forces (elastic after-effect) is determined by the area under the unloading curve; and the work spent for the residual shape change is determined by the area between the curves of loading and unloading (Fig. 1). The conditional index of the reserve of plasticity ϕ of the structural components of the AMC under consideration was estimated as follows:

$$\phi = \frac{A_{\text{rsh}}}{A} \times 100\%. \quad (4)$$

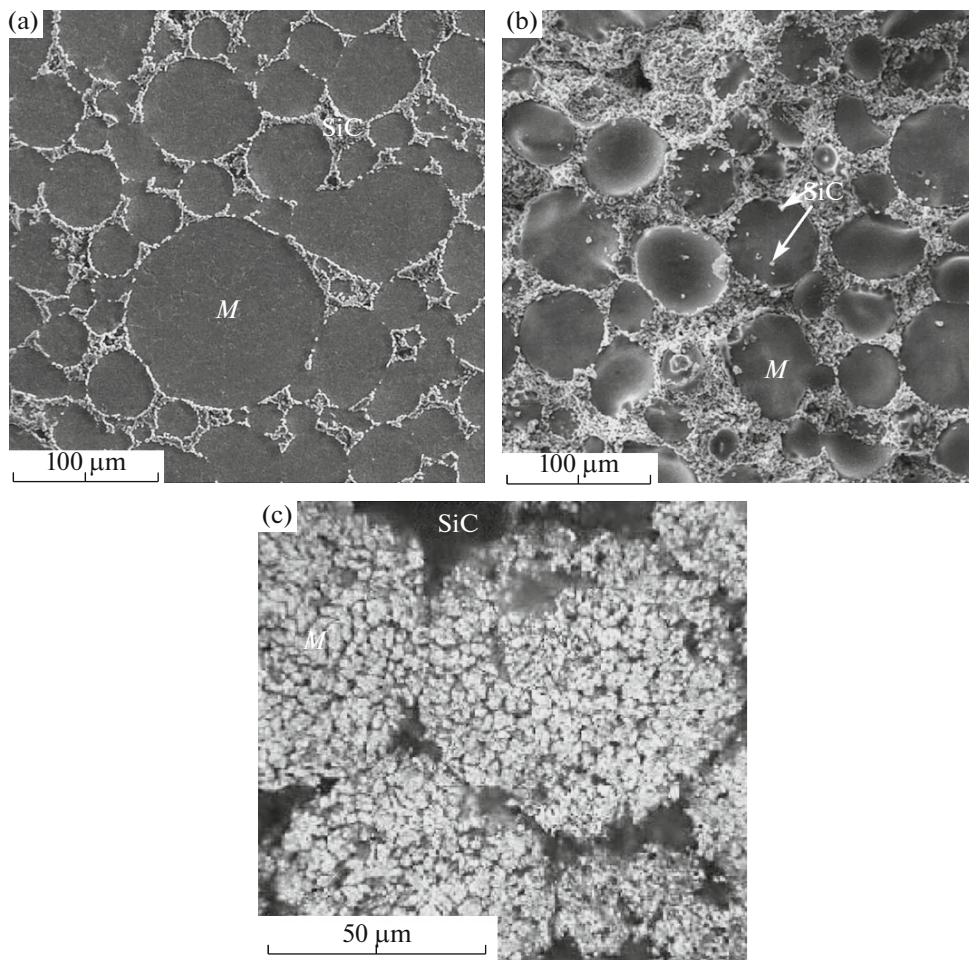


Fig. 2. Macrostructure of the composites under investigation: (a) distribution of the filler in the matrix of the composite with 10 vol % SiC; (b) distribution of the filler in the matrix of the composite with 30 vol % SiC; (c) granular structure of the composite with 10 vol % SiC; (a, b) images obtained in secondary electrons; (b) metallographic image; *M* is the matrix; and SiC is the filler.

The indentation creep [16] was calculated as follows:

$$C_{IT} = \frac{h_2 - h_1}{h_1} \times 100\%, \quad (5)$$

where C_{IT} is the creep that characterizes the ability of the material to the shape change under permanent loading; h_1 is the depth of the indenter penetration, which corresponds to the initial point of the horizontal segment in the loading curve (Fig. 1); h_2 is the depth of the indenter penetration, which corresponds to the final point of the curve. The time of holding at the maximum load is 20 s.

RESULTS AND DISCUSSION

The structure of the AMCs investigated here differs from that considered in [9, 10]. AMCs with 50 and 70 vol % SiC were characterized by a relatively uniform distribution of SiC particles over the bulk of the

composites; in this work, AMCs represented granules of the aluminum alloy with the particles of the filler localized at their boundaries (Fig. 2a). With an increase in the volume fraction of the filler in the composite, the thickness of the zones of pileups of the filler particles increases (Fig. 2b). Separate particles of the filler introduced into the granules of the aluminum matrix were observed, which can be clearly seen after deep electrolytic etching (protruding particles in Fig. 3a). Each matrix granule is surrounded by the filler particles, which form a continuous network.

High-strength Al–Zn–Mg–Cu alloys are characterized by the formation of the strengthening intermetallic phases η (MgZn_2), T ($\text{Mg}_3\text{Zn}_3\text{Al}_2$), and S (Al_2CuMg) in α solid solution [17]. The S phase is usually precipitated at grain boundaries, whereas the η and T phases are distributed uniformly over the entire volume of the alloy, have dimensions of 20–50 nm, and can be observed only with the aid of transmission electron microscopy [3, 17]. The aluminum matrix in

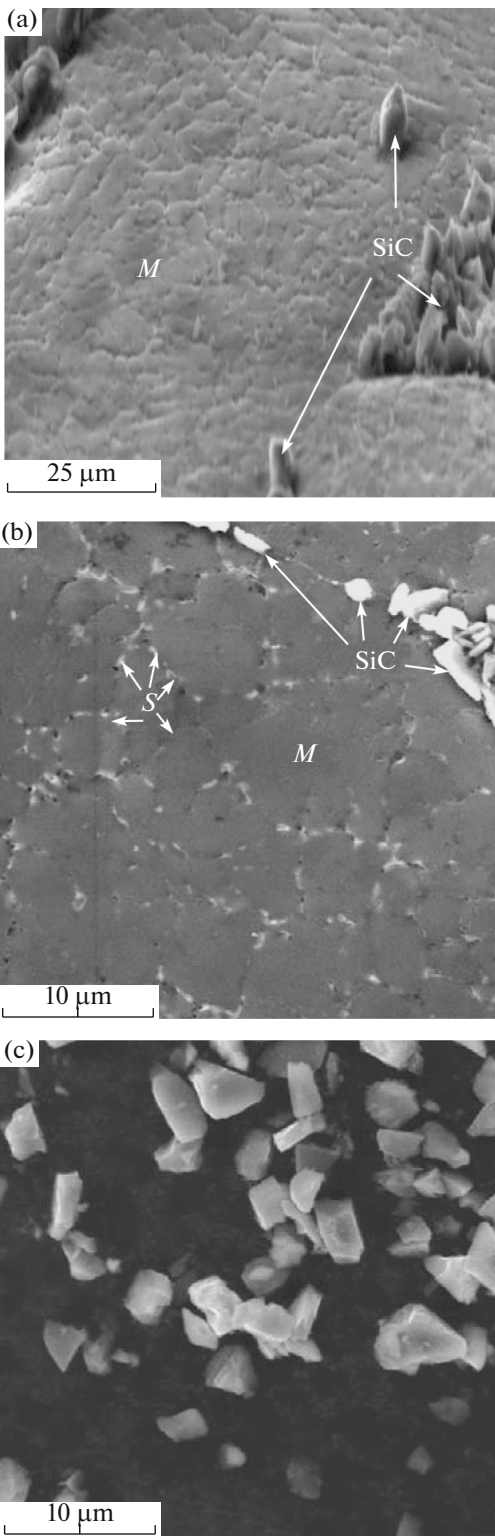


Fig. 3. Shape of the filler particles in the composites under investigation: (a) the surface relief of a sample with 10 vol % SiC after deep etching; (b) distribution of the filler over the grain boundaries and particles of the *S*-type intermetallic compound inside the matrix; (c) shape of the filler particles separated after electrolytic etching of the matrix (SEM images taken in secondary electrons); *M* is the matrix; SiC is the filler; *S* is the intermetallic compound Al_2CuMg .

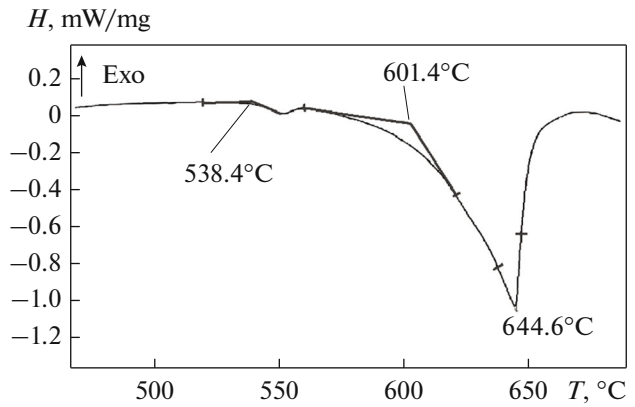


Fig. 4. DSC curve and curve used to determine critical temperatures of composites with 20 vol % SiC (H is the specific heat flux).

the composites under investigation has a granular structure (Fig. 2c) and the greater the size of a granule, the greater the grain size in it. The size of the granules in the matrix is 10–150 μm ; the grain size in the aluminum matrix is 0.05–5 μm . At the grain boundaries, particles of the intermetallic compound *S* (Al_2CuMg) with dimensions of up to 2 μm have been detected (Fig. 3b).

The particles of the filler SiC in the process of the preparation of the composites become incorporated into the surface layers of the original granules of the Al–Zn–Mg–Cu alloy and are located at the boundaries of the sintered granules of the matrix. To determine the true shape of the filler particles, the matrix was dissolved electrolytically. The washed and dried particles were glued onto Scotch cellulose tape and analyzed using a scanning electron microscope (Fig. 3c). The SiC particles had the shape of irregular prisms and plates with an average size of 4 μm .

The investigations by the DSC method have shown that in the metallic matrix of the materials under study there takes place a phase transformation, which is accompanied by the appearance of endothermic peaks at temperatures of 534, 538, and 542°C for the composites with the content of the filler of 10, 20, and 30 vol %, respectively (Fig. 4; Table 1). As is known [3, 17], a eutectic transformation $L \rightarrow \alpha + T$ ($\text{Mg}_3\text{Zn}_3\text{Al}_2$) or $L \rightarrow \alpha + \eta$ (MgZn_2) can take place in the Al–Zn–Mg–Cu alloys at temperatures of about 480°C; or, at a higher temperature, an $L \rightarrow \alpha + S$ (Al_2CuMg) transformation, which occurs at the boundary between the solid solution and the corresponding intermetallic compound. In the temperature range of 480–500°C, the particles of the η and *T* phases become completely dissolved in α solid solution. The endothermic peaks appear to be related to the processes of local melting at grain boundaries according to the reaction $L \rightarrow \alpha + S$ (Al_2CuMg). An increase in the volume fraction of the filler in the com-

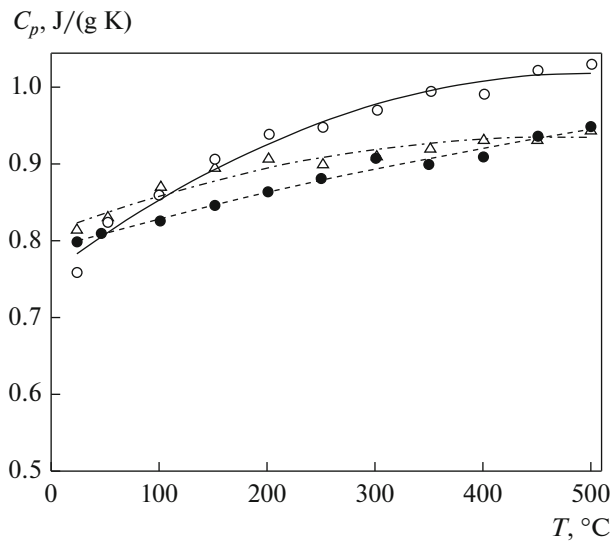


Fig. 5. Temperature dependences of the heat capacity of the composite under consideration: (\blacktriangle) with 10 vol % SiC; (\bullet) with 20 vol % SiC; (\circ) with 30 vol % SiC.

posite leads to an increase in the temperatures of the phase transformations, which is explained by the strengthening of the effect of the filler on the general properties of the composites (Table 1).

The character of the temperature dependences of the heat capacity C_p , thermal diffusivity α , thermal conductivity λ , and thermal expansion coefficient (TEC) indicates that the values of these thermophysical characteristics of the AMCs studied are determined by the content of the filler. The values of the TEC increase with an increase in the amount of SiC; the values of the heat capacity decrease (Table 2). With an increase in the amount of SiC, the dependence of

Table 1. Values of the solidus (T_S) and liquidus (T_L) temperatures and of the temperature of the phase transformation (T_I) of the composite materials investigated

Content of filler, vol %	T_S , °C	T_L , °C	T_I , °C
10	593.6	635.9	534
20	601.4	644.6	538
30	615.3	654.7	542

Table 2. Thermophysical properties of the Al–SiC composite materials at a temperature of 25°C

Property	10 vol % SiC	20 vol % SiC	30 vol % SiC
Thermal expansion coefficient, 10^{-6} K^{-1}	9	16.5	18
Heat capacity, J/(g K)	0.81	0.79	0.76
Thermal conductivity, W/(m K)	106	72	76
Thermal diffusivity, mm^2/s	45.5	33	36
Density, g/cm^3	2.74	2.82	2.90

the heat capacity on temperature increases; upon heating from 25 to 500°C, the heat capacity of the composite with 10 vol % SiC increased by only 16%, that of the AMC with 20 vol % SiC increased by 19%, and that of the AMC with 30 vol % SiC increased by 36% (Fig. 5).

The values of the thermal diffusivity of composite materials upon heating from 25 to 500°C on the whole decrease (Fig. 6a), although for the composite with 10 vol % of the filler their increase was observed in the range of 25–250°C to 49 mm^2/s with the subsequent decrease upon the heating to 500°C to 44 mm^2/s . With allowance for the error of the measurement of the thermal diffusivity, it can be assumed that for the AMC with 10 vol % SiC, the values of this characteristic hardly change in this temperature range.

As is known, the thermal diffusivity or the coefficient of thermal diffusivity is not a physical property of a substance or material. This parameter characterizes the propagation of the temperature in a material in nonstationary thermal processes. In fact, it is a measure of thermo-inertial properties of the substance. The greater the content of the filler in the composite, the lower its thermoinertial properties in the temperature range of 25–500°C; the tendency to the decrease in thermal diffusivity upon heating is most significantly pronounced for the AMC with 30 vol % SiC (Fig. 6a).

The temperature dependences of the thermal conductivity calculated via formula (1) had a tendency to grow with increasing temperature (Fig. 6b). This is explained by the fact that the values of the heat capacity and density that enter into formula (1) increase with increasing amount of the filler in the composite. Note that an increase in the values of the heat capacity for the material with 30 vol % SiC is more substantial than the decrease in the values of the thermal diffusivity.

To estimate the mechanical properties of the investigated composite materials, we used the results of microindentation at various loads. As was shown earlier [18, 19], the results of kinetic microindentation give an estimate on the strength characteristics of the structural constituents of different materials. It is important to correctly choose the load on the indenter in order to estimate the micromechanical properties of each structural constituent or the properties of the

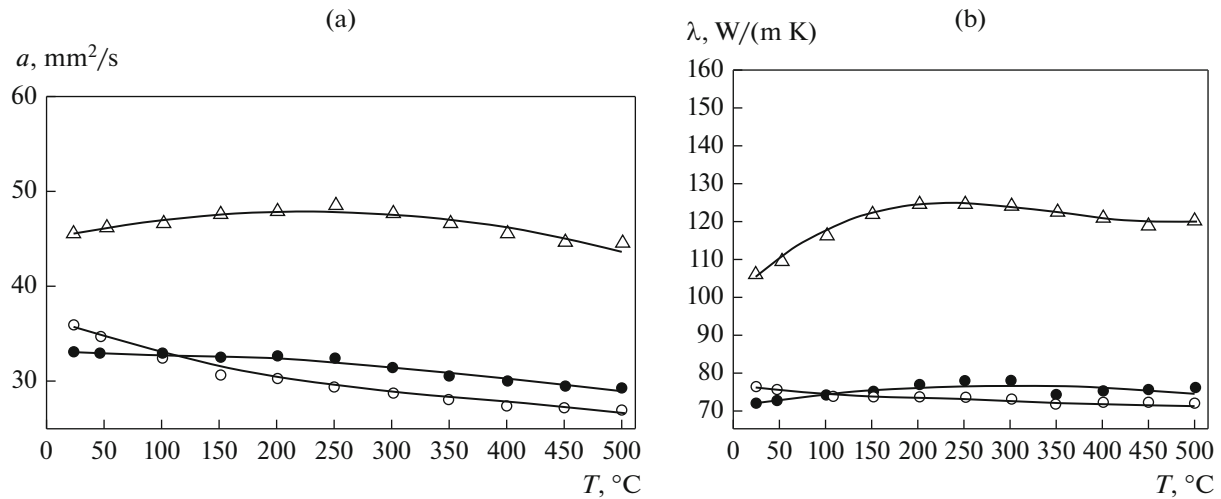


Fig. 6. Temperature dependences of (a) the thermal diffusivity and (b) the thermal conductivity of the composites under consideration: (Δ) with 10 vol % SiC; (\bullet) with 20 vol % SiC; (\circ) with 30 vol % SiC.

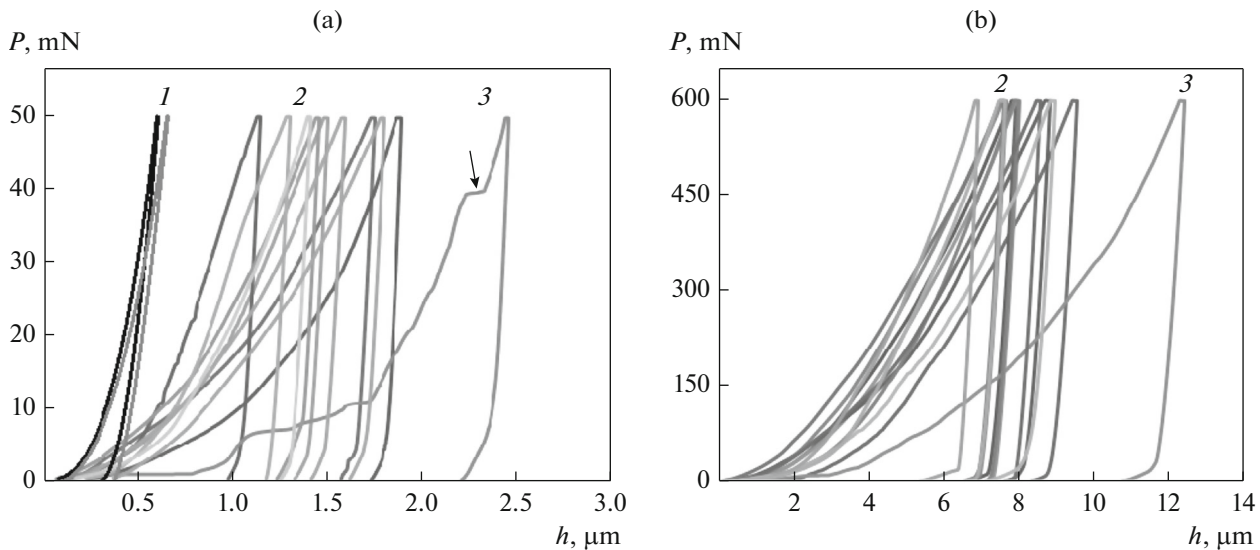


Fig. 7. Microindentation curves for the AMC with 30 vol % SiC: (a) maximum load 0.05 N; (b) maximum load 1.96 N; (1) filler (SiC); (2) zone of interactions of constituents of composite; and (3) matrix.

material on the mesolevel, where the mutual effects of the structural constituents manifest themselves. To estimate the loading conditions under which the true properties of the matrix and the filler are revealed, we performed the microindentation under loads of 0.05–1.96 N.

At a load of 0.05 N, the indentation curves can be divided into three groups. The first group corresponds to the minimum values of the depths of penetration of the indenter and determines the properties of the filler; the second group characterizes the mutual influence of the matrix and the filler; the third group characterizes only the matrix (Fig. 7a). At a load of 1.96 N, the loading curves are located close to each other, the values of the microhardness and elasticity

modulus at the majority of the points analyzed are approximately equal, which indicates the influence of the filler on the properties of the matrix not only in the plane of the section, but also in the underlying layers (Fig. 7b). The filler is not separated into a special series, although regions were observed that corresponded only to the matrix (when coarse matrix grains were indented). The loading curves of the matrix have often an unstable character (marked by an arrow in Fig. 7a), which is related to the enhanced tendency of aluminum alloys to creep.

With an increasing amount of filler in the composites studied, the averaged values of the micromechanical pressures change (Table 3). The properties of the filler and matrix could only be determined separately

Table 3. Results of the microindentation of the Al–SiC composites

Amount of the filler, vol %	<i>HV</i> 0.2	<i>E</i> , GPa	<i>E</i> _{elast} , GPa	φ, %	Creep, %
10	90	76	67	89	0.8
20	124	88.7	74	88	0.7
30	135	95	84	86	0.6
Filler SiC	170	150	132	86	0.6
Matrix	55	67	60	92	1.2

under the load of 0.05 N on the samples with 20 and 30 vol % SiC, since in these cases, the dimensions of the regions occupied by the filler on the surface of the sections proved to be sufficient to exclude the effect of the matrix in the maximum extent. The values of the microhardness and the elasticity modulus for the filler proved to be much lower than the known values for the sintered SiC (216 *HV*0.002 and *E* = 312 GPa [20]), since in the case of the kinetic microindentation, the effect of the matrix cannot be completely excluded. Furthermore, it should be taken into account that the indenter is intruded into the region of the filler, which represents an agglomerate of dispersed particles, each surrounded by the aluminum matrix (Figs. 3a, 3b).

CONCLUSIONS

(1) Structural features of the investigated aluminum-matrix composites have been established. The particles of the filler (SiC), which have the shape of irregular prisms or plates with an average size of 4 μm, are located at the boundaries of matrix granules. Each granule is surrounded by the particles of the filler that form a continuous network. The matrix granules have a structure characteristic of the alloys of the Al–Zn–Mg–Cu system: α solid solution of the alloying elements in aluminum with particles of the intermetallic compound Al₂CuMg (*S* phase) with dimensions of 2 μm located at grain boundaries.

(2) The thickness of the interlayers between the matrix granules increases with an increase in the volume fraction of the filler from 10 to 30 vol %. Separate particles of the filler incorporated into granules of the aluminum matrix have been detected; they are clearly visible after deep electrolytic etching of the surface of the polished section. The greater the amount of filler in the composite, the greater the number of regions with SiC particles incorporated into the central parts of the matrix granules.

(3) It has been established that, in the metallic matrix of the studied materials, a phase transformation occurs that is accompanied by the appearance of endothermic peaks at temperatures of 534°C (in the composite with 10 vol % SiC), 538°C (20% SiC), and 542°C (30% SiC). These endothermic peaks can be related to the processes of local melting at grain boundaries according to the reaction $L \rightarrow \alpha + S$

(Al₂CuMg). An increase in the volume fraction of the filler in the composite leads to an increase in the temperatures of liquidus and solidus.

(4) The temperature dependences of the thermo-physical characteristics of the composites investigated have been determined. It has been shown that the TEC and the density increase with increasing amount of SiC, whereas the thermal diffusivity and the thermal conductivity decrease. With an increase in the fraction of the filler, the dependence of the heat capacity on the heating temperature increases, i.e., upon heating from 25 to 500°C, the heat capacity of the composite with 10 vol % SiC increased only by 16%, while that of the AMC with 20 vol % SiC increased by 19% and that of the AMC with 30 vol % SiC increased by 36%.

(5) It has been shown that the greater the amount of the filler in the investigated composites, the lower its thermoinertial properties in the temperature range of 25–500°C. The tendency of the thermal diffusivity and thermal conductivity to decrease upon heating has also been presented.

(6) The investigations of the micromechanical properties of the composite materials have shown that the most reliable results upon the determination of the properties of each of the structural constituent separately can be obtained upon indentation at a load of no more than 0.05 N. At greater loads, the neighboring structural constituents influence one another and the measured values of the properties characterize the composite on a mesolevel.

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