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Creation of aluminum matrix composites reinforced with micro- and nanoparticles of titanium carbide with nickel coating and description of their hardening mechanisms

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ABSTRACT

During the high-temperature synthesis of carbide reinforced Al matrix composites, as a result of a chemical reaction between carbide particles and a metal matrix, aluminum carbide is formed, which embrittles the system. To eliminate the negative effect of aluminum carbide in the material, the dispersed phase of TiC was pre-metallized with a thin Ni layer. Our work also evaluates the effect of Ni-plated particles of TiC and aluminum nickelide, formed as a result of the chemical reaction of Ni with molten Al, on the hardening of the Al matrix composite. The mechanisms of hardening of the aluminum matrix composites with a dispersed phase of micro- and nanometer size are proposed. The analysis of the contributions of the above mechanisms to the hardening of the TiC reinforced Al composite considered in this article, shows that the main contribution to the hardening due to the low bulk concentration of reinforcing particles is provided by the dispersion hardening Orowan-Ashby mechanism and the dislocation mechanism caused by a thermal expansion coefficients mismatch between the matrix and reinforcing particles.

KEYWORDS

composite materials • ceramic nanoparticles • aluminum • mechanical properties

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Introduction

It has been repeatedly noted that the main technological problems in the synthesis of composites containing ceramic particles and carbon additives are: the distribution of the reinforcing phase in the bulk composite; the strength of its adhesion to the matrix; chemical and structural stability of the reinforcing phase in the composite [1–5]. These tasks are solved by us first of all at the stage of the composite powder preparation.

During the high-temperature synthesis of carbide reinforced Al matrix composites, undesired aluminum carbide is formed, as a result of reaction between carbide particles and a metal matrix. Formed carbide embrittles the system and significantly reduces the plasticity of composites [6–8].

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To minimize the negative impact, we introduced TiC into the Al matrix in the form of core-shell particles. The Ni shell minimizes the possibility of chemical bonding between C and Al and improves the particle wettability in the Al melt [9–12]. In this paper, we evaluate the effect of the Ni shell on the hardening of metal composites reinforced with core-shell particles, and experimental results are compared with the theoretical assessment.

There are many factors contributing to the experimentally observed hardening of metal matrix composites by solid particles with higher rigidity than the matrix material. The hardening depends, in particular, on the physical and mechanical properties of the reinforcing particles, their shape, concentration, and distribution over the matrix, as well as the presence of a bond at the particle-matrix interface.

When reinforcing the matrix with micro- or nanoparticles, the strength increase cannot be explained by known continuous models: the mixture rule, the Halpin-Tsai, Hashin-Shtrikman, Mura and other models [13]. The hardening mechanisms based on the involvement of dislocation models make it possible not only to explain qualitatively, but also to predict an increase in the strength properties of a metal matrix composite.

The improvement in the strength characteristics of reinforced materials, compared with pure Al, is due to the structural inhomogeneities that create additional resistance to the movement of dislocations. The main reason for the hardening is the creation of obstacles to the dislocations movement in the main lattice.

The observed hardening of composites with a metal matrix is the combination of several possible mechanisms [11–24]:

1. due to the efficient load transfer between the matrix and the reinforcing elements;
2. due to the elastic modules mismatch of the matrix materials and the inclusion;
3. due to the grain grinding of the matrix (Hall-Petch law);
4. Orowan hardening caused by the dispersed phase;
5. due to internal thermal stresses caused by a mismatch in the thermal expansion coefficients between the matrix and the carbide particles;
6. hardening caused by the appearance of an interfacial layer between the reinforcing particle and the matrix.

Which of these mechanisms are implemented in each specific case is determined by both the geometry, size and concentration of particles, and the conditions for introducing reinforcing particles into the matrix. The analysis of the contributions of the above mechanisms to the hardening of the TiC reinforced Al composite considered in this article, shows that the main contribution to the hardening due to the low bulk concentration of reinforcing particles is provided by the dispersion hardening Orowan-Ashby mechanism and the dislocation mechanism caused by a thermal expansion coefficients mismatch between the matrix and reinforcing particles.

Materials and Methods

Production of a bulk composite based on Al reinforced with nano-TiC

The of Al samples were cast on an induction vacuum compression casting machine UVLK–30A (Spark–Don LTD, Russia), equipped with an autonomous water cooling module MAVO–3. During the casting, we used tablets of material, consisting of dispersed Al matrix and a reinforcing TiC phase. Composite tablets were obtained by mixing Al matrix particles with a reinforcing phase in dry mixing mode at room temperature, followed by pressing. Al was mixed with a dispersed reinforcing phase in ratios 99:1, 99:3, 95:5 wt. %.

Aluminum ASP-50 with a size of 50–80 μm was used as a matrix. As a reinforcing phase, core-shell TiC/Ni particles with a diameter of 20 μm and 200 nm were synthesized [14–28]. The difference in the method of Ni plating of TiC particles was to increase the thickness of the Ni coating on TiC surface by increasing the deposition cycles number to 3 [12].

The samples were melted in a vacuum melting mode with a pressure of less than 250 mbar under constant inert gas flow (argon). The flow of Ar throughout the entire melting and casting process provides minimized oxidation of the melt and its gas saturation. The minimum weight of the workpiece was chosen at 3.2 g. The casting temperature for Al raw materials was selected experimentally as 700 °C. Plates were made from the obtained samples on a spark-cutting machine to evaluate mechanical properties.

Characterization

The phase composition of the samples was monitored using XRD analysis on a Bruker "D2 Phaser" powder diffractometer. The surface defects of the samples were examined by optical microscopy. The structure of the composite material and elemental composition were studied using a scanning electron microscope Zeiss Merlin.

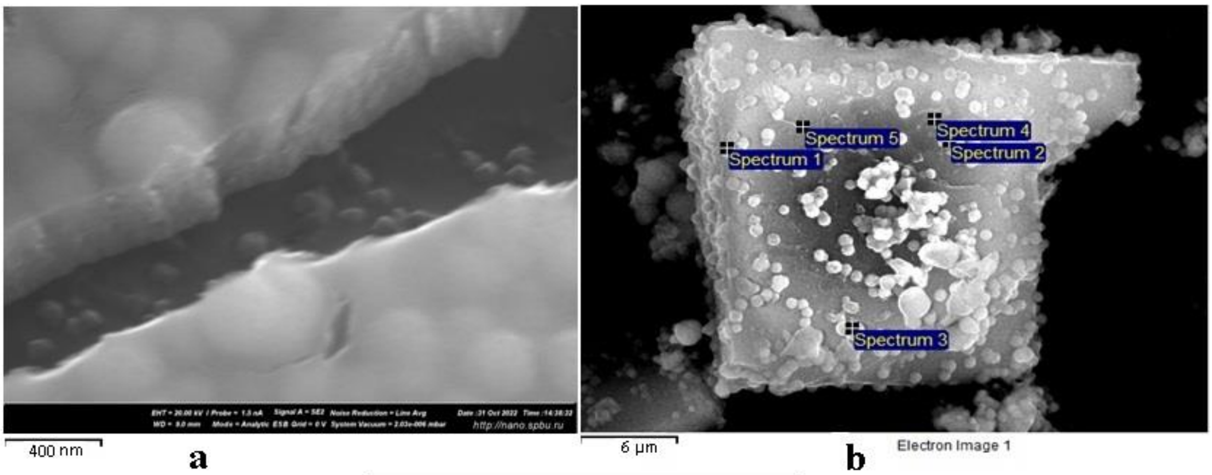
Mechanical tests, conducting tests for uniaxial tension

After a comprehensive study, samples were cut from the plates for uniaxial loading tests. Uniaxial stretching was performed on a Shimadzu AG–50kNX testing machine at room temperature, the deformation rate was $5 \cdot 10^{-4}$ s^{-1}. The deformation of the samples was controlled by a TRViewX 55S video extensometer. Bending tests were carried out in accordance with GOST 14019–80 "Metals and alloys. Methods of testing for bending".

Results and Discussion

To study the effect of the dispersed TiC phase with Ni nanostructures on the mechanical properties of an Al matrix composite, samples of the reinforcing phase with a core-shell structure were obtained after 3 cycles of Ni deposition. It should be borne in mind that the carbide phase is used for reinforcing an Al matrix composite at Al melting temperatures. So, for better TiC particles shielding from the Al matrix, the Ni shell must be thicker.

The morphology of the reinforcing phase particles and the surface of bulk sample obtained by casting was studied by a scanning electron microscopy (SEM) (Figs. 1 and 2). Figure 1 shows a micrograph of a 20 μm TiC particle with Ni. A mixed type of Ni coating growth is observed. On the TiC surface, both Ni shell coating (Fig. 1(a)) and Ni agglomerates (Fig. 1(b)) form. Elemental analysis of the modified particles shows the presence of Ti, C and Ni. The carbon content is due to carbon tape that was used as a substrate for the samples analysis (Fig. 1(c)).

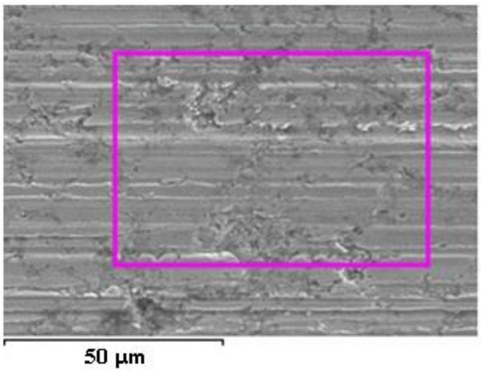


Spectrum	In stats.	C	Ti	Ni
Spectrum 1	Yes	67.91	13.29	18.74
Spectrum 2	Yes	65.91	25.06	9.03
Spectrum 3	Yes	68.88	18.98	12.14
Spectrum 4	Yes	67.29	23.74	8.97
Spectrum 5	Yes	66.69	25.37	7.94
Max.		67.88	25.37	21.37

c

Fig. 1. Micrograph of TiC particles with Ni on the surface: (a) a section of the Ni shell on the TiC surface, (b) a TiC particle with Ni on the surface, (c) the elemental composition of the TiC surface

Figure 2 shows a micrograph of the composite sample surface with 5 % added reinforcing phase. The sample is nonporous. For a detailed study of the samples surfaces, the elemental composition at several surface points was evaluated simultaneously with microscopy. It can be seen from the microphotographs that the elemental sample composition corresponds to the expected one and consists of Al, Ti, C and Ni.



element	concentration weight %
Ti	4.40
Ni	2.45
C	5.05
Al	88.10

Fig. 2. Micrograph of a section of a composite Al sample with a 5 wt. % TiC/Ni

When obtaining bulk materials, the high temperature promotes the chemical reaction between carbide particles and the Al matrix, and undesired aluminum carbide can be obtained, which embrittles the system and reduces the strength characteristics and significantly reduces the plasticity of composites. XRD studies of samples using of a TiC-reinforced Al matrix composite with a Ni shell showed the absence of an aluminum carbide phase. The Al_3Ni phase was detected instead (Fig. 3) that, in opposite, leads to additional hardening [29,30].

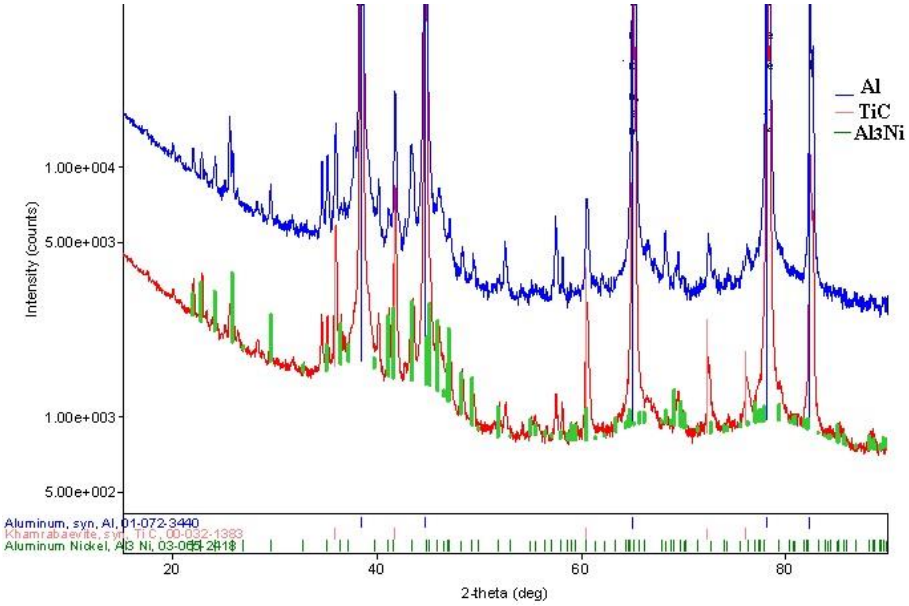


Fig. 3. Diffractogram of a bulk Al–TiC/Ni sample

The mechanical properties of Al matrix composites have been studied by uniaxial stretching. Mechanical tests were carried out at room temperature at the deformation rate $5 \cdot 10^{-4}$ s^{-1}. From the samples obtained, double-sided blades with a working part size of 6 × 2 mm^2 were made on the ART 123 PRO electroerosion machine of NPK Delta–Test LLC. The ends of the blades were examined using a side-illuminated Micmed-6 microscope in order to control the samples integrity. All Al matrix composite samples obtained by injection molding show increased strength properties when tested for uniaxial tension (Fig. 4).

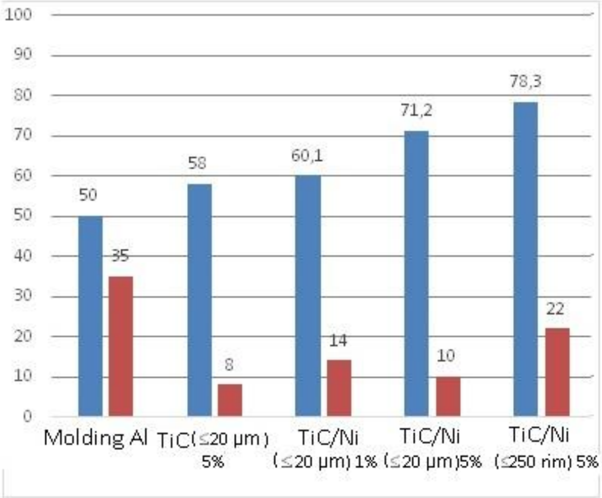


Fig. 4. Mechanical characteristics of Al matrix composites. Blue bars indicate the strength of the samples, red bars indicate the relative elongation of the samples

Al-based composites strengthened by Ni-plated TiC particles of both micron and nanometer sizes demonstrate higher strength characteristics compared with pure Al. This strength improvement is due to structural inhomogeneities that create additional resistance to the dislocations movement. The increased effect of this mechanism can be observed during the transition from micron reinforcement particles to nanoscale ones.

Note that when cooling an Al composite reinforced with Ni-plated TiC particles from 600 to 20 °C, the reinforcing particles are in compression conditions, and the matrix material near the interface is under strong stretching conditions, which has a strong effect on the crack resistance of the composite and on plastic deformation. When the mixture melts, an aluminum nickelide layer forms at the TiC particles boundary. Upon cooling, microcracks arise in this layer, which cause a decrease in the plastic deformation area [27].

Mechanisms of hardening of Al matrix composite

Dispersion hardening mechanism

The presence of small-scale reinforcing particles in the metal matrix creates additional resistance to the dislocations movement through the matrix along its sliding planes. When colliding with a particle, moving dislocations first bend and then form a dislocation loop. This leads to an increase in the stress required for further dislocation movement, which increases the yield strength. The interaction between stress fields created by the looping of dislocations makes it difficult to further bend dislocations around particles. Therefore, a higher voltage is required to facilitate the dislocations movement, thereby increasing the composite strength.

Dispersion hardening by particles can be quantified using the Orowan–Ashby equation [17–21]:

$$\Delta\sigma_{Orow} = \frac{0.13 G_m\, b}{\lambda}\ln\left(\frac{d_p}{2b}\right), \quad (1)$$

$$\lambda = d_p\left[\left(\frac{1}{2V_p}\right)^{1/3} - 1\right], \quad (2)$$

where λ is the average distance between the particles, G_m is the shear modulus of the matrix material, for aluminum $G_m = 25.4 \cdot 10^9$ MPa, $b = 0.286 \cdot 10^{-9}$ MPa is the Burgers vector, d_p is a diameter of the reinforcing particle, V_p is a volumetric concentration of reinforcing particles.

The mechanism of deformation hardening caused by the discrepancy between the linear expansion coefficients of matrix materials and reinforcing particles

The strength increase of a metal matrix composite can be explained by matrix strengthening by creating a high dislocations density. While producing a metal matrix composite, thermal mismatch deformations are induced during cooling in the immediate vicinity of reinforcing particles due to the linear expansion coefficients difference between the matrix and the reinforcement. These mismatch thermal deformations change the internal stress state at the matrix-particle interface. To reduce the thermal effect and remove residual thermal stresses at the interface, the dislocations density near the interface of the particle matrix increases, which reduces the stored energy. As a result, the matrix undergoes deformation hardening near the interface and its strength increases. This hardening is quantified using the following equations [21–23]:

$$\Delta\sigma_{CTE} = M\beta G_m b\sqrt{\rho_{CTE}}, \quad (3)$$

$$\rho_{CTE} = \frac{A\Delta\alpha\Delta T V_p}{b d_p (1-V_p)}, \tag{4}$$

where ρ_{CTE} is the dislocations density generated by the linear expansion coefficients mismatch, M is the Taylor coefficient $M \approx 1$, β is a geometric constant, $\beta \approx 1.25$ for Al, A is a geometric constant (4 for equiaxed particles and 12 for extended ones), $\Delta\alpha$ is the linear expansion coefficients difference between reinforcing particle and matrix materials, ΔT is the difference between the synthesis temperature (in our case, the Al melting point) and room temperature.

These two mechanisms are used to evaluate the Al matrix hardening by TiC particles with a 20 μm diameter, TiC particles with a 20 μm diameter with a deposited 50 nm Ni layer and TiC particles with a diameter of 200 nm with a deposited 50 nm Ni layer.

Modeling of hardening by TiC and TiC/Ni particles

In the injection molding metal matrix composite obtaining approach, a reinforced with carbide particles, after cooling from the Al melting point to room temperature in the vicinity of the reinforcing particle-matrix boundary, the dislocation density increases significantly due to a linear expansion coefficients mismatch Eq. (4), which results in the composite hardening.

The main contribution to the hardening of a metal matrix composite reinforced with 20 μm TiC particles is provided by the dislocation hardening mechanism ($\Delta\sigma_{CTE} \approx 4.5$ MPa); the dispersion mechanism contribution is insignificant. When 20 μm TiC particles with a deposited 50 nm Ni layer, interact with molten Al, aluminum nickelide particles are formed, diffusing into molten Al. This assumption is confirmed by the diffractogram of a bulk composite Al – TiC/Ni (Fig. 3).

After cooling to room temperature, Ni_3Al forms an additional reinforcing phase along with TiC, which, despite its low concentration, makes a significant contribution to the metal composite hardening. At 1 wt. % of TiC/Ni, hardening due to Ni_3Al particles at their concentration of less than 0.01 vol. % TiC ~ 8.5 MPa, and at 5 wt. % of TiC/Ni, hardening due to Ni_3Al particles at their volume concentration in an Al matrix of less than 0.03 % $\Delta\sigma_{Ni3Al} \approx 18.5$ MPa.

Table 1. Composition of aluminum matrix based composites

Matrix	Reinforcing phase	Reinforcing phase, %	σ_V, MPa exp.	σ_V, MPa calc.	Increment σ_V, %	Relative elongation
Cast Al 99.9%	-Cast Al		50	–	–	35
Cast Al 95.5%	20 μm TiC 5 wt. %	5	58	54.	+15	8
Cast Al 99.0%	20 μm TiC/Ni 1 wt. %	1	60.1	60.5	+20	14
Cast Al 95.5%	20 μm TiC/Ni 5 wt. %	5	71.2	80.3	+42	10
Cast Al 95.5%	TiC/Ni ($d \leqslant 250$ nm) 5 wt. %	5	78.3	88.2	+47	22

Considering that the hardening by carbide and aluminum nickelide particles are independent, the total hardening is determined by their sum [4]: $\Delta\sigma = \Delta\sigma_{TiC} + \Delta\sigma_{Ni3Al}$. At 1 wt. % of TiC/Ni, the total hardening is $\Delta\sigma \approx 10.5$ MPa, at 5 wt. % of TiC/Ni $\Delta\sigma \approx 30.4\ \mathrm{MPa}$. The theoretical hardening model correlates with the experimental results (Table 1).

When reinforced with 200 nm TiC particles coated with a 50 nm Ni layer, the Ni_3Al layer formed on the carbide particle surface does not diffuse into molten Al. These particles with a diameter of 250 nm are barriers to the dislocations movement, generating the metal composite hardening. Taking into account the mechanical properties of aluminum nickelide, this composite particle can be considered as homogeneous, attributing to it the Ni_3Al properties.

Estimating dispersion hardening Eq. (1) and dislocation hardening Eq. (3), we obtain $\Delta\sigma_{Orow} = 13.6\ \mathrm{MPa}$ and $\Delta\sigma_{CTE} = 24.6\ \mathrm{MPa}$. The total hardening due to these two mechanisms $\Delta\sigma = \Delta\sigma_{Orow} + \Delta\sigma_{CTE} = 38.2\ \mathrm{MPa}$ corresponds to the experimental hardening (Table 1).

Conclusions

In conclusion, the use of core-shell TiC particles with a Ni shell in the production of Al matrix composites not only minimizes the possibility of chemical bonding between C and Al and improves the particle wettability in the Al melt, but also increases strength properties.

When reinforcing an Al matrix with micro-sized TiC particles with a nanoscale Ni layer at 3 vol. %, a strength increase of > 40 % is observed.

The analysis shows that for small concentrations of reinforcing carbide particles with a Ni shell on their surface, the main hardening mechanism involves the dislocation hardening caused by thermal expansion coefficients mismatch between the matrix and the carbide particles, and the dispersion mechanism.

CRediT authorship contribution statement

Elena G. Zemtsova: writing – review & editing, writing – original draft; **Boris N. Semenov**: writing – original draft; **Nikita F. Morozov**: data curation; **Yuri V. Sidorov**: writing – review & editing; **Vladimir K. Kudymov**: data curation; **Vladimir M. Smirnov**: investigation, writing – original draft.

Conflict of interest

The authors declare that they have no conflict of interest.

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