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Structure and tensile fracture mechanism of aluminum matrix composites produced by internal oxidation

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Abstract: This article presents experimental results of resistance against fracture upon static tension of cast aluminum matrix composites based on aluminum with various content of Al_2O_3 strengthening phase. The cast aluminum matrix composite materials were produced by the technology based on burnout of aluminum melt upon interaction with oxygen. Two batches of ingots with various content of solid phase were smelted for tests of static strength. The average particle size of strengthening phase of predominantly prismatic morphology was 60–80 μm, and their content varied from 15 to 25 %. The fracture surfaces obtained upon static uniaxial tension of the considered samples were studied on the samples destroyed at maximum stress. The fracture surfaces were analyzed using an optical microscope with expanded options due to improved long-focus system and digital processing of images based on unique procedure of 3D structure analysis. For in-depth analysis of characteristic fracture region a scanning electron microscope was used equipped with energy and wavelength dispersive elemental analyzers. It was established in the studies that in the samples with lower content of dispersed phase, the fracture is characterized by mixed heterogeneous in terms of macrogeometry pattern. This can be interpreted as dry fibrous fracture with visible crystalline pimples and breakaways. With an increase in the solid phase, a mixed, sufficiently homogenous in terms of macrogeometry, fracture pattern of fanlike fibrous structure can be observed. Crystalline pimples were also detected of a different fracture surface area, as well as breakaways of other geometrical sizes. The features of changes in the relief of fracture surface and the fracture mechanisms of the obtained composites have been detected and described.

Keywords: cast aluminum matrix dispersion strengthened composite material (DSCM), corundum, tension, transcrystalline fracture, intercrystallite fracture, macrostructure, microstructure, deformation

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Структура и механизм разрушения алюмоматричных композитов, полученных методом внутреннего окисления, при растяжении

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Аннотация: Представлены результаты исследований сопротивления разрушению при статическом растяжении литых алюмоматричных композитов на основе алюминия с различным содержанием упрочняющей фазы Al_2O_3. Литые алюмоматричные композиционные материалы были изготовлены по технологии, которая основана на процессе выгорания расплава алюминия при взаимодействии с кислородом. Для проведения исследований на статическую прочность были отлиты две партии слитков с различным содержанием твердой фазы. Средний размер частиц упрочняющей фазы, преимущественно призматической морфологии, составлял 60–80 мкм, а их количество изменяли от 15 до 25 %. Поверхности разрушения, полученные при статическом одноосном растяжении исследованных образцов материала, изучались на образцах, разрушившихся при максимальном значении напряжения. Исследования поверхности разрушения проводились с помощью оптического микроскопа с расширенными возможностями за счет усовершенствованной длиннофокусной оптической системы и цифровой обработки изображения с применением оригинальной методики изучения 3D-структур. Для углубленного анализа характерных областей излома использовался растровый электронный микроскоп с энерго- и волнодисперсионным элементным анализаторами. В ходе проведенных исследований было установлено, что у образцов с меньшим содержанием дисперсной фазы излом носит смешанный неоднородный по макрогеометрии характер, который можно интерпретировать как сухой волокнистый излом с видимыми кристаллической сыпью и вырывами. С увеличением количества твердой фазы наблюдается смешанный, достаточно однородный по макрогеометрии характер излома с веерообразно-волокнистым строением, в котором также зафиксированы кристаллическая сыпь, отличающаяся распределением по площади излома, и вырывы других геометрических размеров. Выявлены и описаны особенности изменения рельефа поверхности разрушения и механизмы разрушения полученных композитов.

Ключевые слова: литой алюмоматричный дисперсно-упроченный композиционный материал (ДУКМ), корунд, растяжение, транскристаллитное разрушение, интеркристаллитное разрушение, макроструктура, микроструктура, деформация

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Introduction

Cast composite materials are promising in terms of their wide scale implementation in various fields of aircraft and mechanical engineering as anti-friction structural and other materials. They provide for decrease in the item weight, improvement of their specifications and especially development of new machines and structures [1—8]. Serial production of dispersion strengthened composite materials (DSCM) with solid phase in the form of SiC and Al2O3 was pioneered by “Duralcan” (Canada), “Alcan” and “Alcoa” (Canada, USA). However, full scale use of DSCM is restricted, especially in Russia, and does not comply with potential technical

capabilities of these materials. This is primarily attributed to insufficiently developed scientific and engineering basis for their creation, which would guarantee the selection and forecasting of their structure, composition and production technology. This would also achieve the required strength and operational properties of machine parts and structures from DSCM, including those with nanosized strengthening elements at acceptable costs [9—11].

Leading positions in terms of application volumes in various fields of mechanical engineering and aircraft are occupied by composite materials based on aluminum matrix. At present numerous technologies of strengthening by dispersed phase are available, including strengthening by own oxides (Al_2O_3) and carbides (Al_4C_3) or mixing of various strengthening dispersed phases (for instance, TiC—Al_2O_3—Al) or aluminides (for instance, Fe_3Al—TiC) [12—21]. In each case with an increase in the volumetric fraction of solid phases, the strength increases and the plasticity of ready composites decreases.

This work investigates aluminum matrix composites obtained by liquid phase method [22], which is based on burnout of aluminum melt upon interaction with oxygen. In the course of its development the positive features of well-known technologies were taken into account: basic oxygen-converter process, casting of aluminum alloys in oxygen environment and development of air independent energy facility on the basis of high metallized fuel [23, 24]. During the interaction of aluminum melt with oxygen, the presence of solid interface between matrix and filler is provided. In addition, this method allows composites to be fabricated in one stage and to provide uniform particle distribution in the melt. Thus, it allows to more complete implementation of DSCM potential. Our comparative uniaxial tension tests demonstrated that the ultimate strength is in the range σ_u = 180÷205 MPa, which in comparison with the results in [25] (σ_u = 100÷÷150 MPa upon variation of Al_2O_3 in alloy from 5 to 20 %) is higher by about 25 %.

The aim of this work is to study the fracture surface of cast aluminum matrix composites after uniaxial tension to fracture with accounting for structure modification.

Experimental

In order to implement the developed method of internal oxidation, a unique test rig was created consisting of a high temperature induction furnace (Fig. 1, position *I*) and system of oxygen storage and supply.

The test rig is comprised of a high accuracy gas panel, equipped at the output with needle valve for accurate adjustment of gas supply, connected with a pressure meter, and after the needle transforming into a regular ball valve and rotameter. Aluminum billets were loaded into the furnace *4* (Fig. 1, position *I*) and smelted. Then, the silicon carbide tube *3* was inserted into the aluminum melt with control of the vertical supply (*1*) and in the corner (*2*), which was hermetically connected with the steel tube fixed with the rotameter. Using this system, oxygen was fed into the aluminum melt. In order to prevent undesirable surface oxidation of aluminum, the protective inert gas environment was formed which was fed via the tube *5*. As a consequence of high temperature chemical reaction $4Al + 3O_2 \rightarrow 2Al_2O_3$, the ceramic phase was obtained directly in the melt in one stage process. In order to eliminate cast defects and to degas, the synthesized material was blown with argon directly before casting into molds of moderate size.

From the fabricated castings, the samples for tensile tests (Fig. 2) were fabricated according to State Standard GOST 1497-84. The ingots were mechanically cut in the transverse direction into preliminary workpieces (Fig. 2, *a*). Then the samples were cut out (Fig. 2, *b*) and ground (Fig. 2, *c*) using the rotary horizontally located wheel of a SShPM-1 machine (Russia) with the ability to vary the number of rotations. In this case, for ease of work, the sample was installed in a special device.

After grinding, the samples were mechanically polished. In this case the wheel was covered with cloth or felt, wetted with chromium oxide slurry during polishing. The surface of polished samples was washed, degreased, and dried.

Aluminum, Grade A6 (Fig. 3, *a*), was used as a matrix material for fabrication of dispersion strengthened composite, with the following chemical composition according to State Standard GOST 11069-2001, wt.%: 99.6 Al; 0.25 Fe; 0.2 Si; 0.03 Ti; 0.01 Cu; 0.06 Zn. The alloy was strengthened with solid oxide phase Al_2O_3 (Fig. 3, *b*), in most cases with a 4- and 6-face shape. In order to analyze the static strength, two batches of ingots were smelted with various content of solid phase.

Three samples for various ingot regions were fabricated from the obtained castings. The average particle size of strengthening phase, mainly of prismatic morphology, was 60—80 μm (Fig. 4), and their content was varied from 15 to 25 vol.%.

The uniaxial tensile tests of flat samples were carried out using an AG-Xplus-0.5 universal testing machine ("Shimadzu", Japan) at ambient temperature, the loading rate was 5 N/(mm^2·s).

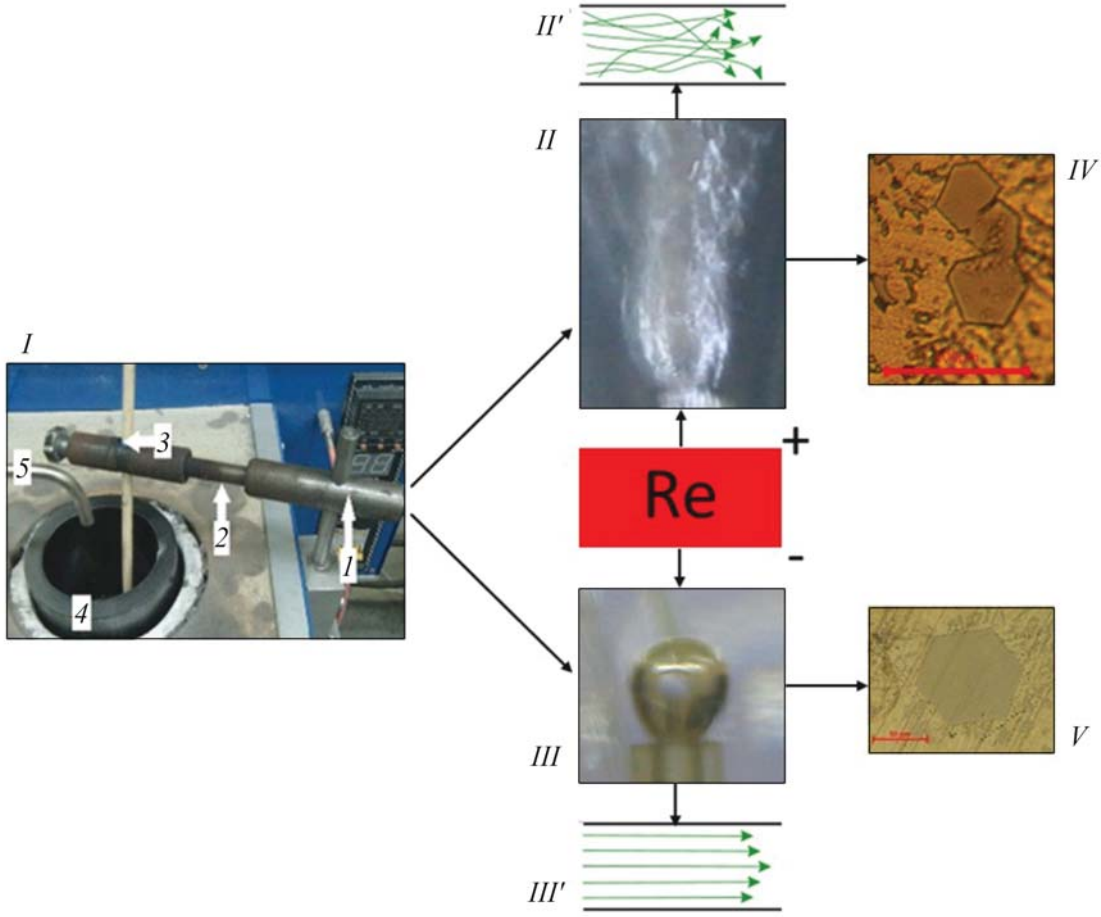

Fig. 1. Layout of ceramic phase fabrication

I – furnace working part with dismounted heat insulation (***1***, ***2*** – control system of oxidizer vertical supply (***1***) and in the corner (***2***), ***3*** – silicon carbide tube, ***4*** – crucible, ***5*** – supply tube of protective inert gas); ***II*** – jet of supplied oxidizing gas with characteristic turbulent motion regime ***II′***; ***III*** – single bubble of oxidizing gas with laminar motion regime ***III′***; ***IV*** and ***V*** – microstructures obtained in the course of processes ***II*** and ***III***

Рис. 1. Схема получения керамической фазы

I – рабочая часть печи с демонтированной теплоизоляцией (***1***, ***2*** – система регулировки подачи окислителя по вертикали (***1***) и углу (***2***), ***3*** – карбидокремниевая трубка, ***4*** – тигель, ***5*** – трубка подачи защитного инертного газа); ***II*** – струя подаваемого газа-окислителя с характерным турбулентным режимом движения ***II′***; ***III*** – единичный пузырек газа-окислителя с ламинарным режимом движения ***III′***; ***IV*** и ***V*** – микроструктуры, получаемые при реализации процессов ***II*** и ***III***

The fracture surface was analyzed using a VHX-1000 optical microscope (“Keyence”, Japan) with expanded options due to improved long-focus system and digital processing of images. The 3D structures illustrated in Figs. 5, *e* and 6, *e*, were analyzed using the procedure for studying fracture surface in 3D image by means of the “e-Preview Optimal Image” regime [26], on the basis of which the most heterogeneous morphologically sites of fracture surface profile. The characteristic features of these fracture regions were analyzed in details using a JSM-IT300LV scanning electron microscope (“JEOL”, Japan) with energy and wavelength dispersive elemental analyzers.

Results and discussion

The fracture surfaces obtained upon static loading of the analyzed samples from dispersion strengthened composite materials were analyzed on samples destroyed at the maximum stress.

Figure 5, *a* illustrates macroscopic image of the fracture surface of flat DSCM sample 1 with 15 % of inclusions of solid phase. Mixed relief, heterogeneous in terms of macrogeometry is observed with crystalline pimples and breakaways. The fracture center and crack nucleation area are distinctly observed at the point of conglomeration of dispersed phase inclusions

(see Fig. 5, *b*, *c*). Analysis of the macrostructure in polarized light in Optimal Image regime (Fig. 5, *b*) and microstructure (Fig. 5, *d*) revealed secondary cracking. This can be caused by high normal tensile stresses as a consequence of the separation of matrix—solid phase interface along the plane of maxi-

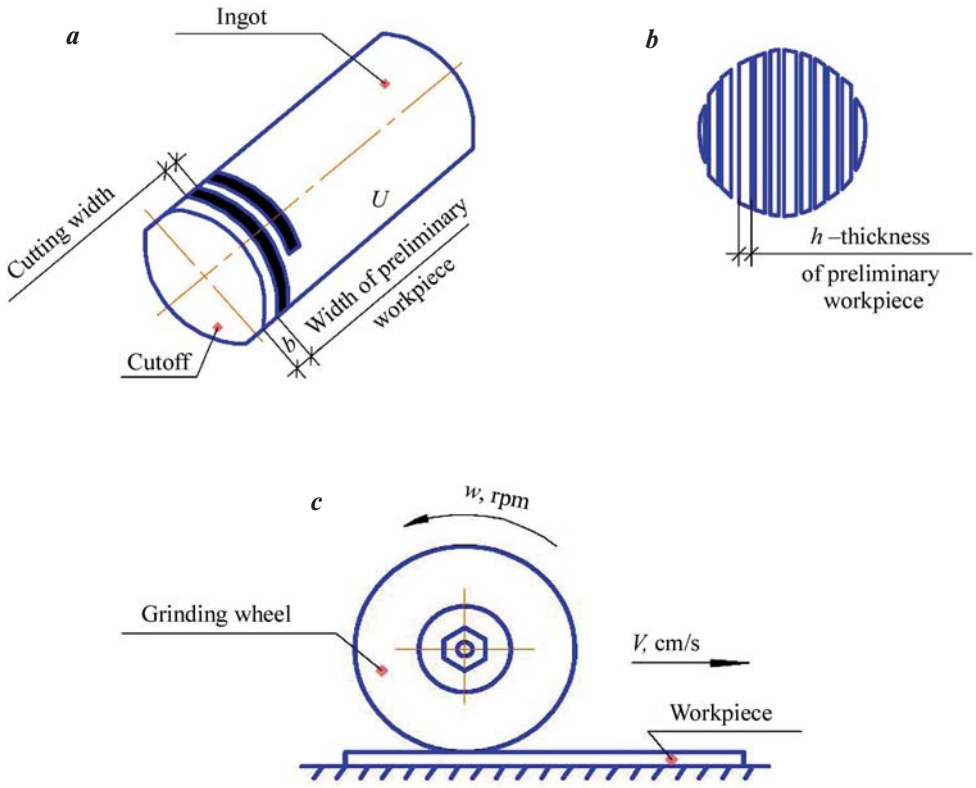

Fig. 2. Schematic view of fabrication of samples for tensile tests
a – transversal cutting of ingot, ***b*** – longitudinal cutting of workpiece, ***c*** – grinding of workpiece

Рис. 2. Схема изготовления образцов для испытаний на растяжение
a – поперечная резка слитка, ***b*** – продольная резка заготовки, ***c*** – шлифование заготовки

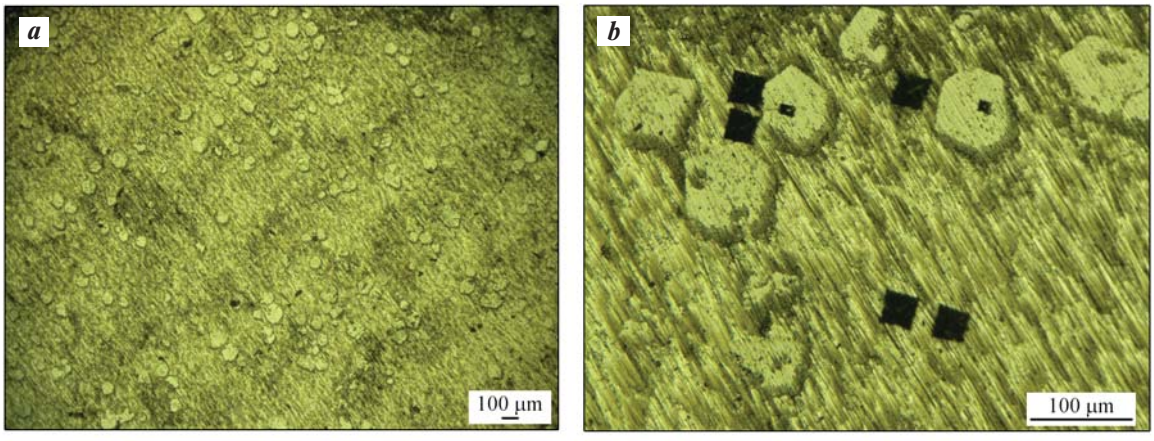

Fig. 3. Microstructure of the obtained material in initial state (***a***) and with indentation of pyramid with the weight of 100 g of PMT-3 hardness meter (***b***)

Рис. 3. Микроструктура полученного материала в исходном состоянии (***a***) и с отпечатками пирамидки массой 100 г твердомера ПМТ-3 (***b***)

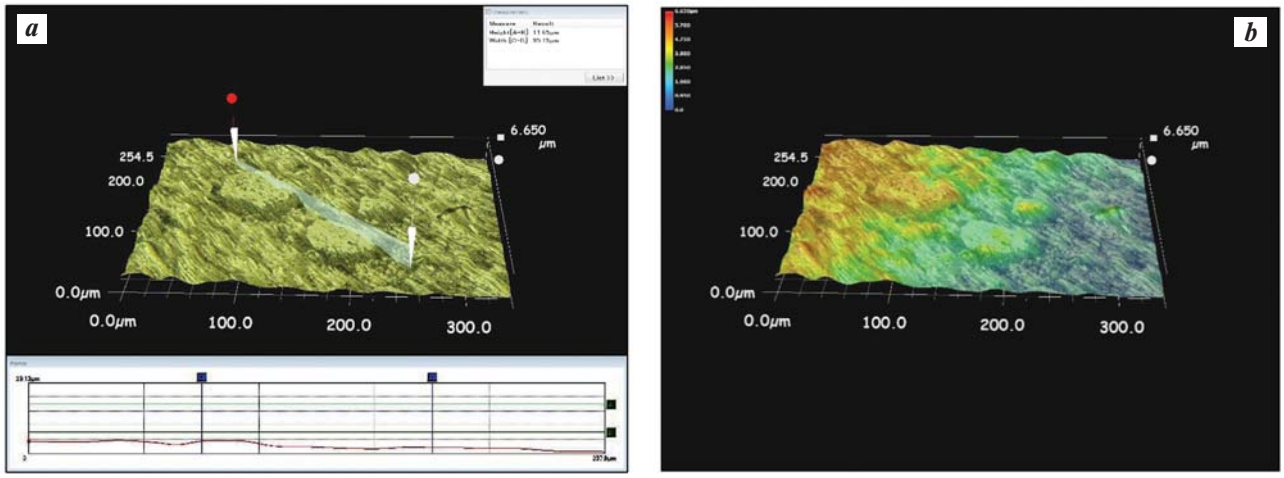

Fig. 4. Topographic image of surface and profile of samples

a – relief across the highlighted cross section with determination of height across this cross section; ***b*** – analysis of relief heights using colored image

Рис. 4. Топографическое отображение поверхности и профиль образцов

a – рельеф по выделенному сечению с определением высот по этому сечению; ***b*** –исследование высот рельефа с использованием цветного отображения

mum shear depending on orientation of each single grain.

The general macrogeometry of fracture surface is heterogeneous, however, individual sites were identified on it. They were characterized by morphologically unified fracture surfaces with nearly similar reliefs and existence of single type fracture elements. This is illustrated in Fig. 5, *e* in the form of degree of surface roughness in the direction perpendicular to the plane of load application, related to the stability of high energy propagation of crack upon fracture at these sites.

A relatively moderate amount of dispersed inclusions (in comparison with the second batch of samples) in the matrix of analyzed material located at significant distances leads to formation of moderate seams. This can be attributed to the fact of their bypassing by crack frontal zone, which is simpler than over the body of high solid phases. The profilogram distinctly illustrates a rather acute single change in the profile oriented at the angle ≈45° to the tension axis (see Fig. 5, *e*).

This is related to a slowing of crack growth and the blunting of its tip as a consequence of significant plastic deformation with formation of a cup-like structure and can be interpreted as a shear at this site characteristic for the shear area.

Microstructural studies (see Fig. 5, *d*) illustrate an alteration of crystalline segments and cleavage edges around oxides according to the transcrystalline mechanism with thin fibrous stripes. In some places a dendritic structure with intercrystallite fracture and quasi-cleavage can be observed.

The pattern of fracture surface of DSCM sample *2*, with 25 % of solid phase inclusions, is illustrated in Fig. 6. The fracture center is far from the tension axis but does not appear at the free surface of the sample. It is possible to observe a mixed fracture pattern, homogenous in terms of macrogeometry, with a fan-like fibrous structure (Fig. 6, *a*). In addition, crystalline pimples are detected which differ from previously considered samples by distribution over the fracture surface, as well as breakaways of other geometrical sizes. The higher amount of dispersive inclusions in the matrix of the analyzed material in comparison with DSCM *1*, located at shorter distances between themselves, smooths out the roughness at certain sites. This can be attributed to a decrease in the distance of bypassing of the solid phase by crack frontal zone. The profilogram (see Fig. 6, *e*), and the 3D structure, indirectly confirms the provisions discussed above. It does not contain morphologically heterogeneous in terms of macrogeometry area with significant differences of fracture surface relief.

Contrary to DSCM *1*, comprised of fibrous area and shear area, at some segments of fracture surface of sample *2* a radial area is detected (see Fig. 6, *c*, *d*). Its occurrence is related to an increase in the content of solid phase in alloy and characterizes transfer of crack from slow growth to its unsteady propagation with formation

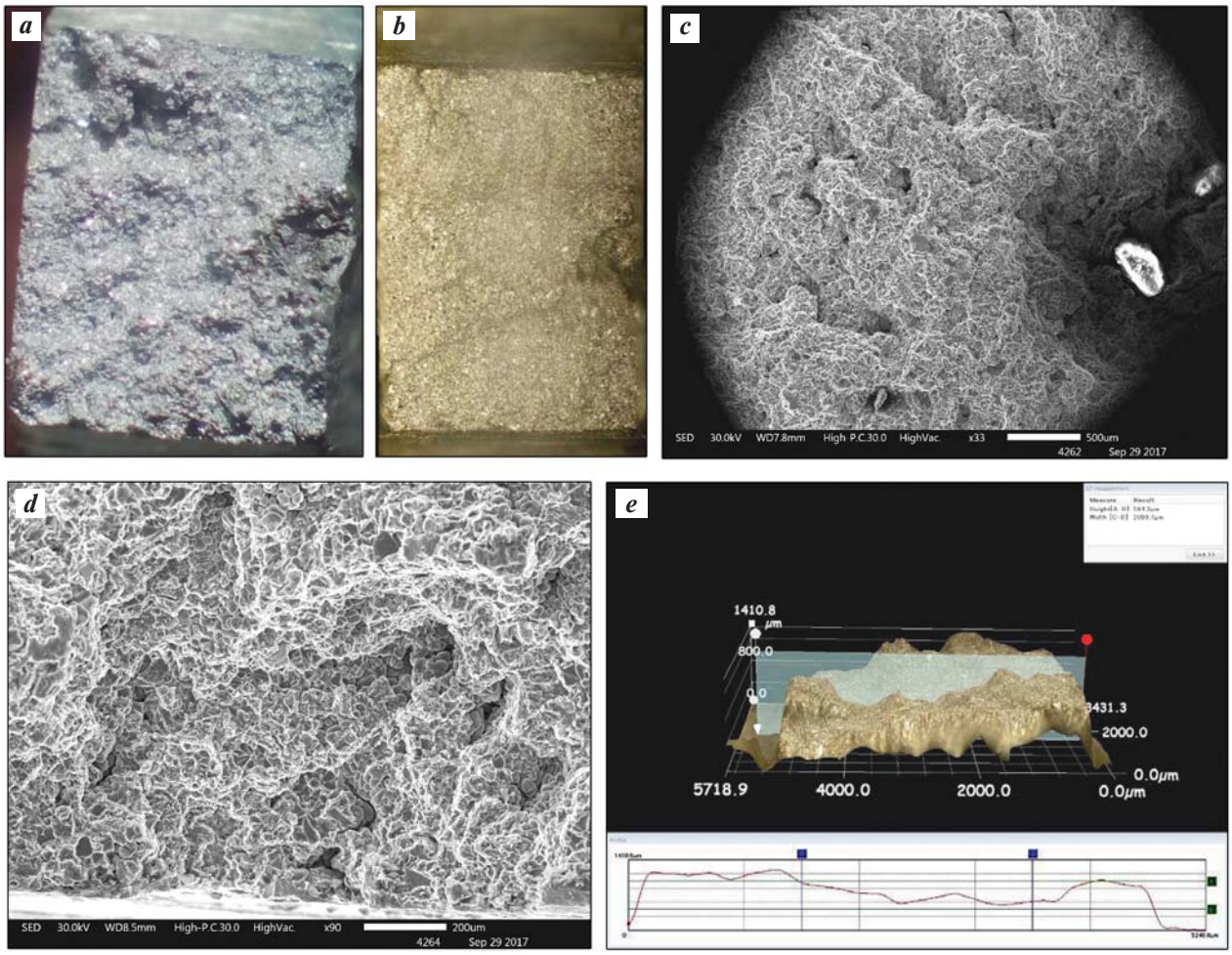

Fig. 5. Fracture surface of DSCM sample *1*

a – optical macrostructure (×40); ***b*** – macrostructure in Optimal Image regime; ***c*** and ***d*** – scanning electron microscopy; ***e*** – 3D structure with profilogram across the highlighted cross section

Рис. 5. Поверхность излома образца ДУКМ *1*

a – оптическая макроструктура (×40); ***b*** – макроструктура в режиме Optimal Image; ***c*** и ***d*** – результаты растровой электронной микроскопии; ***e*** – 3D-структура с профилограммой по выделенному сечению

of radial seams. It is possible to observe the chaotic alteration of viscous fracture according to the tearing mechanism and shear with a brittle cleavage fracture in the form of crystalline fracture. This fracture has stepwise relief and occurrence of dendritic sites with intercrystallite structure (see Fig. 6, *d*).

Such features are caused by the non-compliance of the general direction of crack propagation and the shortest direction from its front to a free surface. This is related to unsteady vortex crack propagation at a microlevel, characterized by stepwise (either fast or slow) propagation across the material body. However, in contrary to the DSCM sample *1* with lower content of solid phase inclusions, this is not related to the existence of single type fracture elements, but with an absolutely opposite fracture mechanism. This is comprised of the alteration of viscous fracture according to tearing and shearing mechanisms with consideration for brittle cleavage fracture at a separate fracture site surface (see Fig. 6, *d*).

Conclusions

The fracture surface of dispersion strengthened aluminum matrix material, with 15 % and 25 % content of dispersed phase exposed to deformation under conditions of static uniaxial tension was studied in this paper. The following differences in morphology of fracture surface were established.

In the material with lower Al_2O_3 content analyzed, the macroanalysis of fracture surface demonstrates

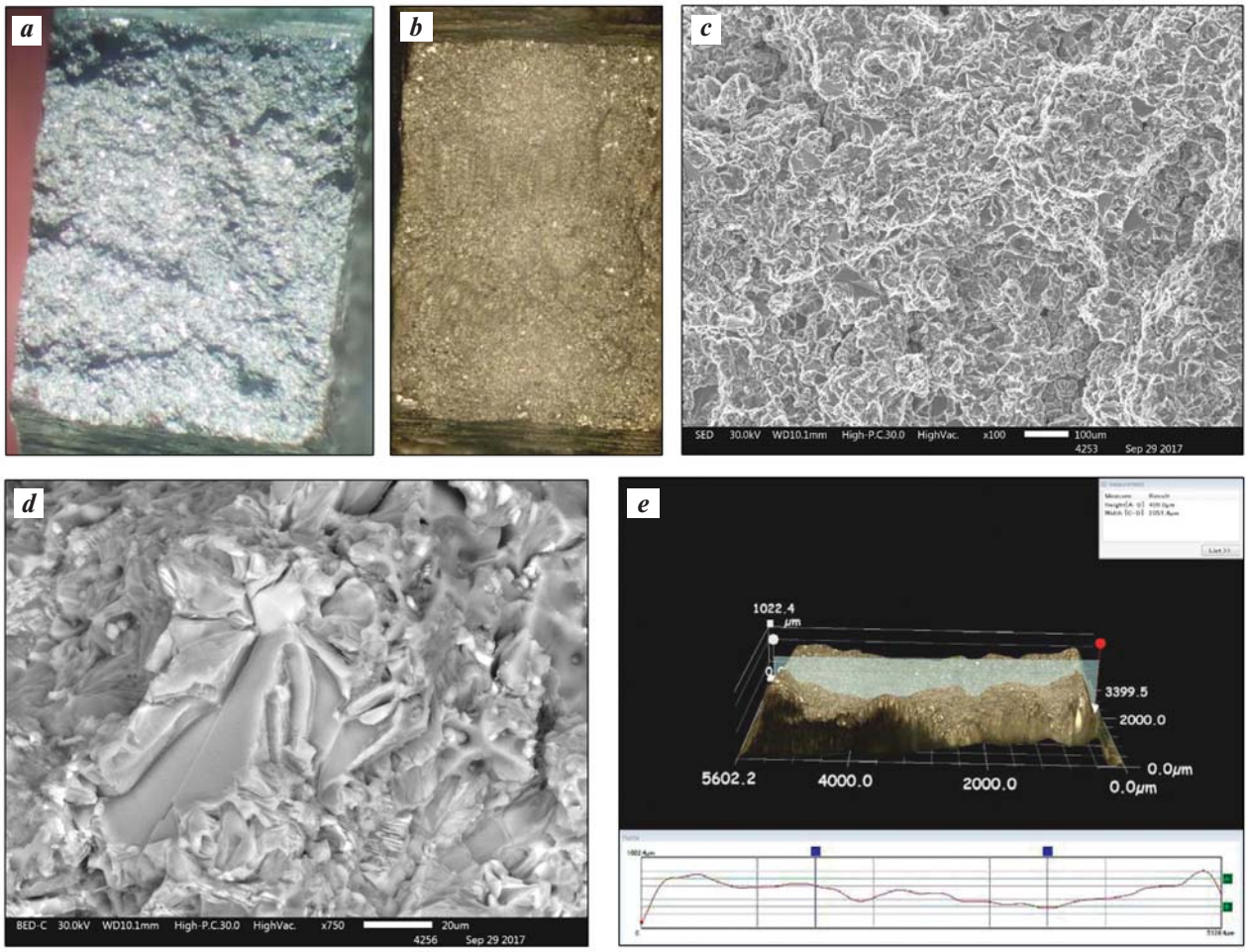

Fig. 6. Fracture surface of DSCM sample *2*

a – optical macrostructure (×40); ***b*** – macrostructure in Optimal Image regime; ***c*** and ***d*** – scanning electron microscopy; ***e*** – 3D structure with profilogram across the highlighted cross section

Рис. 6. Поверхность излома образца ДУКМ *2*

a – оптическая макроструктура (×40); ***b*** – макроструктура в режиме Optimal Image; ***c*** и ***d*** – результаты растровой электронной микроскопии; ***e*** – 3D-структура с профилограммой по выделенному сечению

mixed heterogeneous in terms of macrogeometry pattern. This can be characterized as a dry fibrous fracture, comprised of fibrous area and shear area and accompanied by secondary cracking.

With an increase in the content of solid phase, a radial area appears on the fracture surface, evidencing a change in the fracture mechanism and frontal zone of the main crack. The occurrence of features of the radial area is related to structural changes resulting from an increase in the content of solid phase in the alloy. This characterizes crack transfer from slow growth to its unstable propagation with formation of radial seams.

The structure of DSCM sample *2*, with 25 % content of strengthening phase, does not allow stable propagation of the crack. A higher amount of dispersed phases is a significant obstacle for its propagation, resulting in material strengthening. This consumes a significant portion of the fracture work. At higher (in comparison with the samples of the first batch) content of the strengthening phase in the material structure an obvious alteration of viscous fracture according to breakaway mechanism and shear with brittle fractures can be observed. However, the morphology of fracture surface becomes sufficiently homogeneous in terms of macrogeometry.

Analysis of fractures in the samples of the second batch did not reveal sharp differences in the relief. The profilograms were not characterized by any sharp relief drops or extreme values of the profile.

Therefore the macrostability of fracture processes can be stated, which cannot be attributed to fracture of DSCM sample *1*, where a rather strong single drop is observed.

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