

**PROPERTIES OF A COMPOSITE MADE FROM AN ULTRAFINE-GRAINED
TITANIUM MATRIX AND CONTINUOUS BORON FIBER***Il'nar Alfridovich Ramazanov*

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ABSTRACT

This paper presents the ultrafine-grained structure of the Ti-Al-V alloy foil. This structure was formed through combined processing, including repeated forging with varying deformation axes of the workpiece under gradual cooling from 800 to 650 °C and subsequent isothermal rolling at 550 °C. On the basis of analysis of the temperature and strain-rate conditions for superplasticity of the Ti-Al-V alloy in the ultrafine-grained state, a multilayer metal-matrix composite was obtained by vacuum pressing at 650 °C. The composite consists of alternating layers of 0.2 mm thick titanium foil and continuous high-strength boron fiber with a diameter of 140 μm. The superplasticity regime during pressing ensured uniform filling of the matrix material between the fibers and high-quality bonding of the foil layers in the resulting composite. The mechanical and dynamic properties of the resulting metal-matrix composite were studied at room temperature under three-point bending. Fractographic analysis of post-test specimen fractures revealed that the achieved increase in strength and impact toughness is due to the formation of a homogeneous ultrafine-grained state in the matrix material and the high strength of the fibers, which redistribute stress and alter the crack trajectory.

KEYWORDS

Metal matrix composite; continuous fiber; ultrafine-grained structure; titanium alloy; three-point bending strength; impact bending strength; crack; composite delamination.

**СВОЙСТВА КОМПОЗИТА ИЗ УЛЬТРАМЕЛКОЗЕРНИСТОЙ ТИТАНОВОЙ
МАТРИЦЫ И НЕПРЕРЫВНОГО БОРНОГО ВОЛОКНА***Ильнар Альфридович Рамазанов*

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АННОТАЦИЯ

В данной работе представлена ультрамелкозернистая структура фольги из сплава Ti-Al-V. Эта структура была сформирована в результате комбинированной обработки, включающей много-

кратную ковку с изменяющимися осями деформации заготовки при постепенном охлаждении от 800 до 650 °С и последующую изотермическую прокатку при 550 °С. На основе анализа температурных и скоростных условий сверхпластичности сплава Ti-Al-V в ультрамелкозернистом состоянии методом вакуумного прессования при 650 °С был получен многослойный металломатричный композит. Композит состоит из чередующихся слоев титановой фольги толщиной 0,2 мм и непрерывного высокопрочного борного волокна диаметром 140 мкм. Режим сверхпластичности при прессовании обеспечил равномерное заполнение матричного материала между волокнами и высококачественное сцепление слоев фольги в полученном композите. Механические и динамические свойства полученного металломатричного композита были изучены при комнатной температуре при трехточечном изгибе. Фрактографический анализ разрушений образцов после испытаний показал, что достигнутое увеличение прочности и ударной вязкости обусловлено формированием однородного ультрамелкозернистого состояния в матричном материале и высокой прочностью волокон, которые перераспределяют напряжения и изменяют траекторию трещины.

КЛЮЧЕВЫЕ СЛОВА

Металломатричный композит; непрерывное волокно; ультрамелкозернистая структура; титановый сплав; прочность на трехточечный изгиб; ударная прочность на изгиб; трещина; расслоение композита.

Introduction

Specific strength is a key design criterion in the aerospace and related industries [1–5]. Fiber composites exhibit the highest values for this indicator [5–8]. In addition to high specific strength, such materials are characterized by increased resistance to cyclic loading, which is due to the inhibitory effect of reinforcing fibers on the initiation and propagation of microcracks in the metal matrix. This effect ensures an increase in the interval between repairs and the overall service life of components. Therefore, increasing the durability of metal matrix composites (MMCs) is a pressing scientific and technical challenge.

Carbon fiber-reinforced polymer composites (CFCs), which are widely used today, have a significant drawback: low thermal and electrical conductivity, which leads to structural failure during sudden temperature changes or exposure to atmospheric electrical discharge on an aircraft [9]. Metal matrix composites (MMCs), in particular those based on titanium alloys reinforced with carbon or boron fibers,

do not suffer from this drawback. However, the implementation of such MMCs is hampered by technological difficulties, primarily the high chemical reactivity of titanium with respect to fibers at elevated temperatures, which occur during the composite production stage. This causes fiber degradation and a reduction in strength characteristics below the calculated values [10, 11]. Thus, reducing the temperature of the technological process is a critical factor.

Recent studies have demonstrated the fundamental possibility of producing MMCs using solid-phase isothermic compaction, including a nanostructured titanium alloy matrix (with a grain size of 100–200 nm) and continuous boron fiber with a diameter of 140 μm, under conditions of low-temperature superplasticity of the matrix material [12]. The advantage of this method is the reduction in temperature, pressure, and processing time, which minimizes the risk of chemical degradation and mechanical damage to the fibers due to excessive and non-uniformly distributed stresses [12, 13].

The mechanical and dynamic properties of the matrix alloy also significantly influence the integral characteristics of the composite. Grain refinement to the submicron level leads to a significant change in the combination of strength and plasticity properties. For titanium alloys of the Ti–Al–V system (e.g., VT6) in the ultrafine-grained state, an increase in the yield strength from 800 to 1000 MPa and tensile strength from 900 to 1150 MPa has been recorded. At the same time, the relative elongation decreases slightly (from 12 to 8%), but the impact toughness increases by 1.3–1.5 times due to the activation of additional deformation mechanisms [14, 15]. Furthermore, the nanostructured state provides increased sensitivity to strain rate: under dynamic loading (strain rates $>10^3 \text{ s}^{-1}$), strength properties increase by 15–20% compared to static conditions, due to changes in the dislocation slip mechanism and the suppression of diffusion processes. A reduction in grain size reduces stress concentration at the boundaries and promotes a more uniform strain distribution, which positively impacts resistance to brittle fracture under impact loads.

Based on the above, the aim of this work is to comprehensively evaluate the mechanical and dynamic characteristics of a metal-matrix composite obtained in the low-temperature superplasticity mode based on an ultrafine-grained titanium alloy (Ti–Al–V system) with boron fiber.

1. Material and methods

To obtain an ultrafine-grained microstructure with a grain size of 400–500 nm in the VT6 alloy, multiple forging was used with changes in the deformation axes of the blanks under conditions of gradual cooling from 800 to 650 °C [16]. To obtain a foil semi-finished product for the composite material and to maintain an ultrafine-grained homogeneous structure with predominantly high-angle grain boundaries, the obtained samples were rolled

to a thickness of 0.2 mm under isothermal conditions at a temperature of 550 °C with a deformation rate of 0.2 m/min. The total degree of reduction during isothermal rolling exceeded 95%. This rolling method activates the grain boundary sliding mechanism and, as a result of the relative displacement and rotation of the grains, an ultrafine-grained structure is formed, which is most suitable for further deformation under superplasticity conditions at temperatures below $0.65 T_m$.

To study the material's microstructural features in detail, a complex of transmission electron microscopy studies was conducted using a JEOL JEM-2100 instrument. Visualization was performed in both darkfield and brightfield modes at an accelerating voltage of 200 kV. The objects of study were thin foil samples prepared by jet polishing at a temperature of –25 °C and a voltage of 25 V.

A prototype metal-matrix composite reinforced with continuous boron fiber with a diameter of 140 μm and a strength of $\sigma_b = 3100 \text{ MPa}$ was fabricated using a multilayer compaction method using a foil-fiber-foil arrangement, where the fibers were laid in the form of a woven grid, sparsely intertwined with thin wire. The sheet surfaces were pre-ground to remove the oxidized layer after isothermal rolling. The process was carried out at a temperature of 650 °C, maintaining a strain rate of 10^{-3} s^{-1} . After final compaction of the package, the temperature was increased to 700 °C and maintained under pressure in order to improve diffusion bonds on the surfaces being joined.

The temperature and strain rate conditions for pressing the composite correspond to the low-temperature superplasticity regime of the VT6 alloy in a nanostructured state, determined from the results of mechanical tensile tests with temperature 600 and 650 °C with short-term switching of deformation strain rates, increasing the rate and returning it to the initial

position after stabilization of the flow to analyze the change in the sensitivity of the flow stress to a change in the strain rate ($\dot{\epsilon}$) from 10^{-2} to 10^{-4} s^{-1} .

To assess the ultimate strength, three-point bending tests were performed on strip specimens with longitudinal reinforcement 7 mm wide and 1 mm thick at room temperature using INSTRON testing equipment.

Dynamic impact bending tests were conducted at room temperature on a TSMK-50 pendulum impact tester on strip specimens without a concentrator with a cross section of $8 \times 1.2 \text{ mm}^2$. The maximum stored energy of the impact hammer is 50 J.

To comprehensively study the nature of material failure, the fracture morphology of fractured test specimens was examined using a JEOL JSM-6490LV scanning electron microscope.

2. Results

The realization of superplasticity depends on the ratio of the deformable body size D to the grain size d : at $D/d \leq 10$, superplasticity disappears, despite maintaining the required fine-grained structure and other conditions. To achieve a fiber volume fraction of approximately 40% in the composite, the minimum interfiber distance ($\geq 140\text{--}150 \text{ }\mu\text{m}$) is $10\text{--}20 \text{ }\mu\text{m}$, with a grain size of $5\text{--}10 \text{ }\mu\text{m}$ in the matrix material, making the realization of the superplasticity effect impossible during composite compaction.

To develop the production of MMC composites, it is necessary to reduce the grain size to less than 500 nm in order to satisfy the condition of the scale factor $d \leq 0.1t$ for the implementation of the SP effect in the foil of the matrix material [17].

To produce a semi-finished foil product, the VT6 titanium alloy of the Ti-Al-V system was subjected to combined processing. During the combined processing, which

consisted of repeated forging with changes in the deformation axes of the blanks under conditions of gradual cooling from 800 to 650 °C and subsequent isothermal rolling at a temperature of 550 °C, the initial coarse-grained structure of the VT6 alloy was transformed into a homogeneous ultrafine-grained structure with a grain size of approximately 160 nm (Fig. 1, *a, b*).

Mechanical testing of the VT6 alloy with an ultrafine-grained structure established the temperature and strain rate conditions for the realization of high strain rate superplasticity in a titanium alloy foil semi-finished product (Fig. 1, *c, d*). Based on the analysis of the flow stress values, the composite will be manufactured at a temperature of 650 °C.

The resulting VT6 alloy foil (hereinafter referred to as the matrix or matrix material) was used to fabricate a multilayer metal-matrix composite by vacuum pressing using a foil-fiber-foil arrangement. The boron fiber reinforcement is shown in Fig. 1, *e*. High-strain rate superplasticity at 650 °C ensured uniform filling of the matrix material between the fibers and the bonding of the foil layers (Fig. 2, *a*). The fiber volume fraction was 25%.

To comprehensively study the mechanical and dynamic properties of the resulting composite, all tests were conducted in three-point bending at room temperature. For comparison, tests were also conducted on a matrix material with an ultrafine-grained structure.

Impact bending tests were carried out on a pendulum impact machine with an impact velocity of 3.8 m/s, the work of destruction was 18.8 J. The impact toughness value of the matrix material was 380 kJ/m², the impact toughness value of the composite was 510 kJ/m². Similar impact toughness values of 120 kJ/m² were obtained in the Ti-Al₃Ti composite with a volume fraction of intermetallic particles of up to 80% [18].

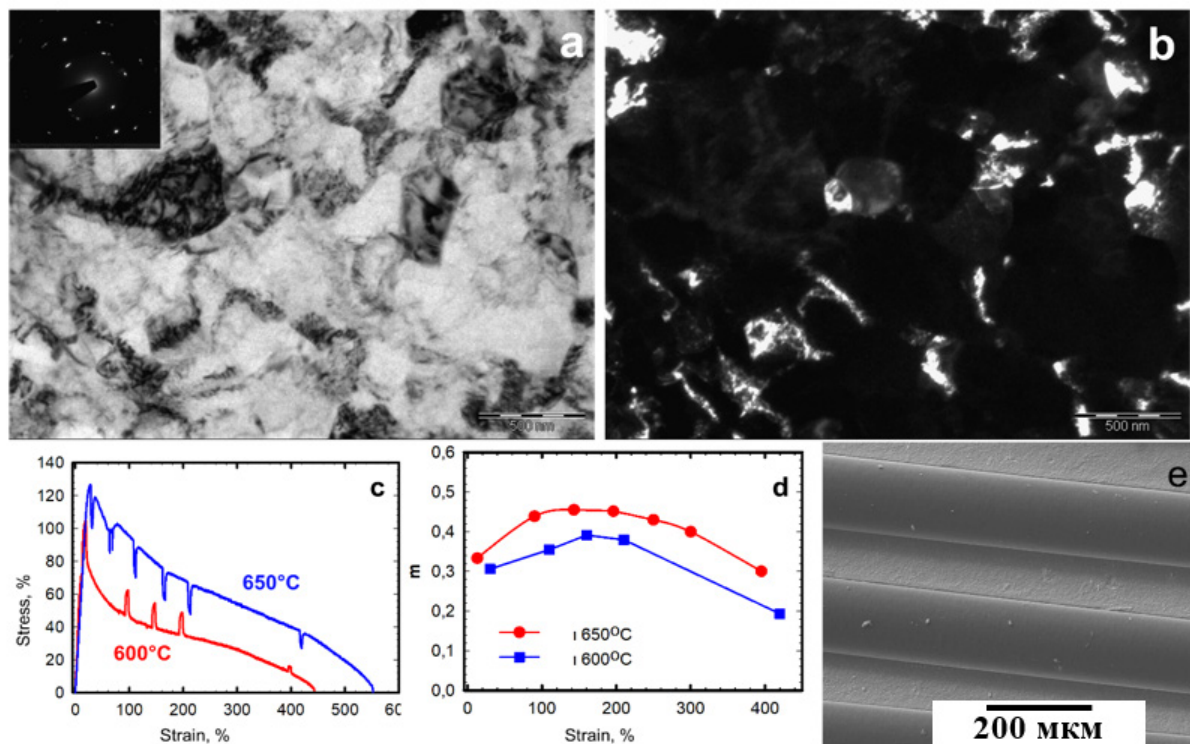


Fig. 1. Microstructure of VT6 alloy in foil semi-finished product after combined processing:
a – bright field with electron diffraction pattern; *b* – dark field; *c* – tensile curve recorded at 600 and 650 °C;
d – change strain-rate sensitivity parameter during a tensile test; *e* – boron fiber with a diameter of 140 μm

Рис. 1. Микроструктура сплава ВТ6 в полуфабрикате из фольги после комбинированной обработки:
a – светлое поле с дифракционной картиной электронов; *b* – темное поле; *c* – кривая растяжения
 при 600 и 650 °C (*c*); *d* – изменение параметра чувствительности к скорости деформации в ходе испытания
 на растяжение; *e* – борное волокно диаметром 140 мкм

Static tests of three-point bending tests of composite samples in the form of strips with longitudinal fiber reinforcement at room temperature showed a strength level of 2430 ± 5 MPa, while the matrix material showed a strength level of 1920 ± 5 MPa (Fig. 2, *b*). The bending angle before crack formation was 6° . For comparison, in [19], mechanical tests of three-point bending tests of metal-matrix carbon-aluminum composite samples showed a maximum tensile strength of 520 ± 50 MPa.

The matrix material in the ultrafine-grained state demonstrates high strength values after static and dynamic testing compared to the industrial equivalent of a sheet semi-finished product made of VT6 alloy. The high-strength state of the VT6 alloy is achieved by grain refinement to 160 nm, an increase in dislocation density to $\rho = 25 \times 10^{-14} \text{ m}^{-2}$ [12], and an increase in the number of high-angle grain boundaries.

Fracture analysis of matrix material and composite samples after static three-point bending and impact bending is presented in Fig. 3 and 4. The fracture of the matrix samples is ductile, dimple-type, and velvety in appearance, characteristic of an ultrafine-grained structure [15, 16]. The fracture initiation surface is a quasi-cleavage containing micron-sized dimples and microcrack junctions. Further crack propagation is accompanied by plastic deformation and acquires a developed relief. In the composite, the matrix material fails similarly, but traces of crack arrest are observed on the fiber rows. Overall, the composite failure surface has a developed relief. Fiber fractures are at the same level as the matrix and do not form deep “tears”, indicating the absence of fiber degradation and sufficient bonding between the fibers and the matrix.

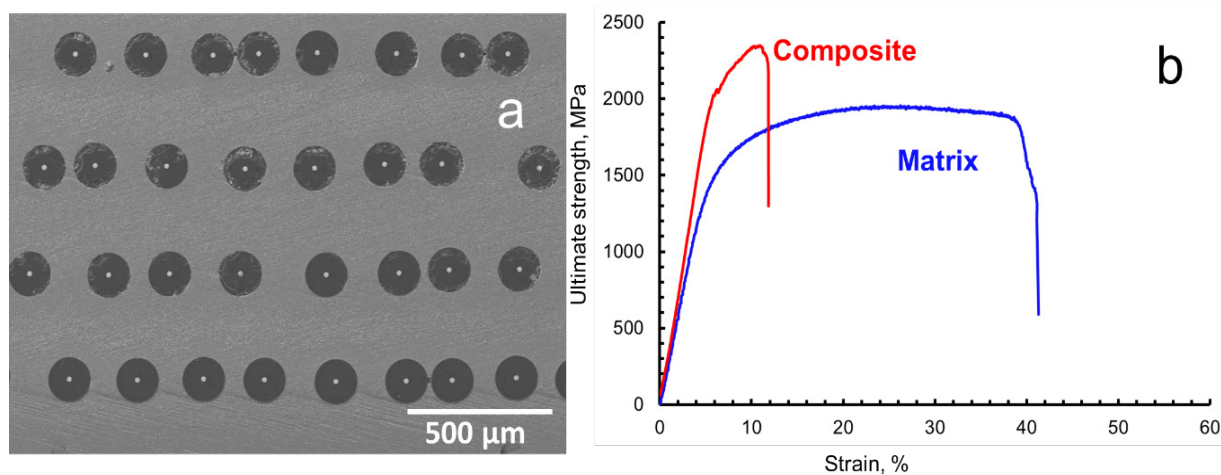


Fig. 2. Metal matrix composite in cross section (a); curves after three-point bending tests (b)

Рис. 2. Металломатричный композит в поперечном сечении (a); кривые после испытаний на трехточечный изгиб (b)

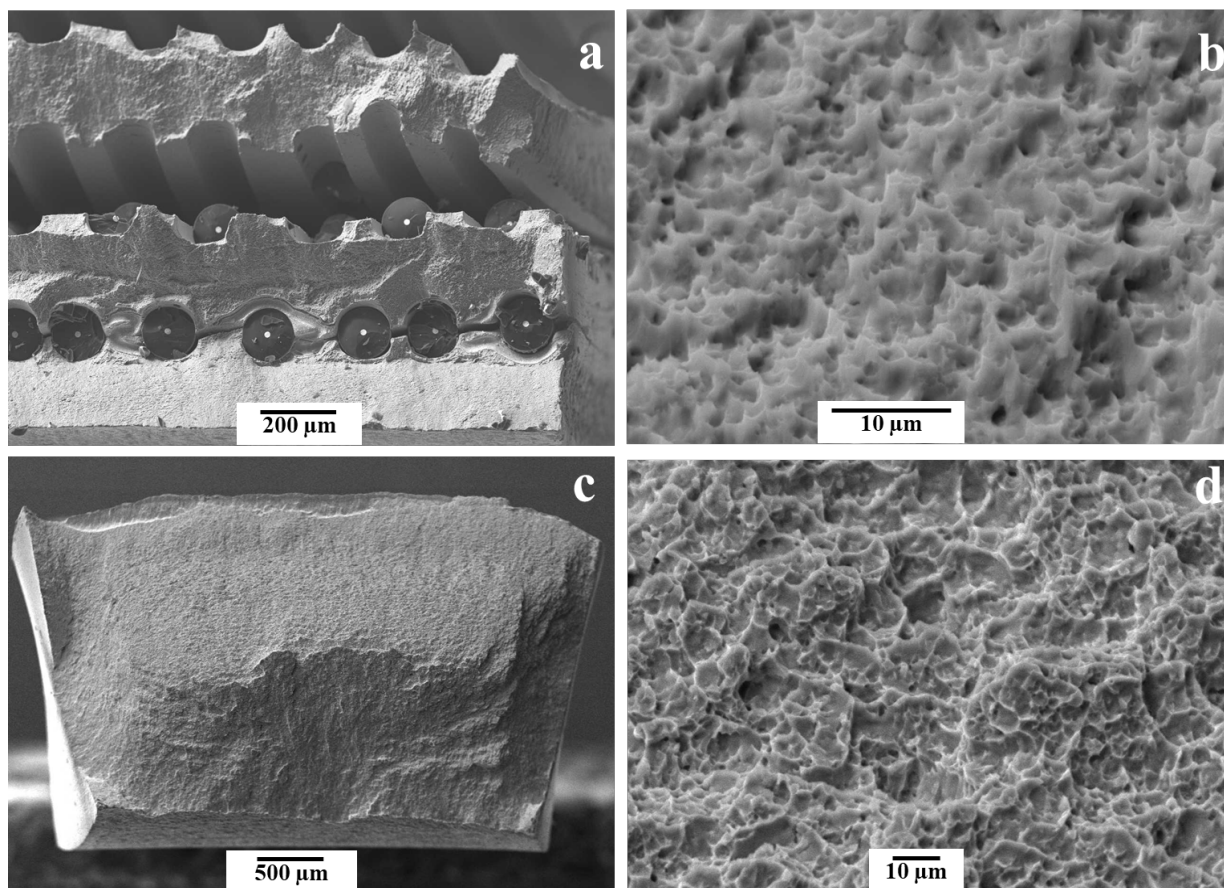


Fig. 3. Fracture surface of samples after impact bending tests:
a, b – composite; c, d – matrix material with UFG structure; b, d – enlarged section

Рис. 3. Поверхность излома образцов после испытаний на ударный изгиб:
a, b – композит; c, d – матричный материал с ультрамелкозернистой структурой;
b, d – увеличенный фрагмент

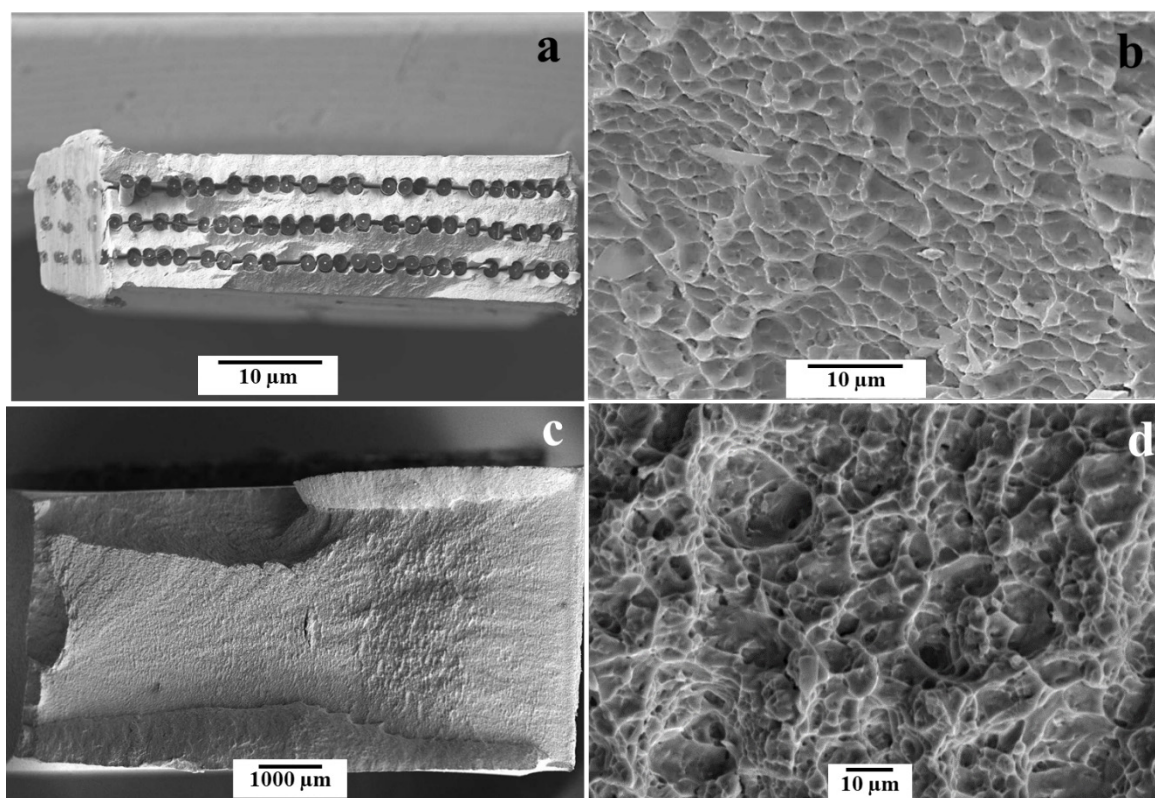


Fig. 4. Fracture surface of samples after three-point bending:
a, b – composite; c, d – matrix material with UFG structure; b, d – enlarged section

Рис. 4. Поверхность излома образцов после трехточечного изгиба:
a, b – композит; c, d – матричный материал с ультрамелкозернистой структурой; b, d – увеличенный фрагмент

Conclusions

The production of a multilayer metal-matrix composite by vacuum pressing using a foil-fiber-foil system under established temperature – strain rate conditions for superplasticity confirmed the feasibility of producing a titanium composite material reinforced with boron fibers with high-quality bonds at the matrix-matrix and matrix-fiber interfaces. The use of ultrafine-grained VT6 alloy allows the process to be carried out at reduced pressing forces and compaction temperatures [20].

This study investigated the impact toughness and three-point bending strength of a metal-matrix composite consisting of alternating layers of ultrafine-grained titanium foil and continuous boron fiber with a diameter of 140 µm. The achieved increase in composite strength is due

to the formation of a homogeneous ultrafine-grained state in the matrix material and the high strength of the fibers, which redistribute stresses and change the direction of crack propagation.

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