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Sapphire/TiAl Composites - Structure and Properties

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Summary:

Ti-Al-intermetallic-based alloys with lamellar microstructure, γ(TiAl) +α_2(Ti_3Al) are characterized by a high melting point of 1460°C, a low density of ~3.9 g/cm^3, a high gas corrosion resistance up to a temperature of about 900°C, a high creep resistance up to a temperature of about 800°C, and a sufficiently high fracture toughness at low temperatures, up to 30 MPa·m$^{1/2}$. Hence, they are considered as excellent matrices for fibres of high melting point. Unlike well-developed SiC/TiAl composites, which have an obvious upper limit for the usage temperature due to SiC/TiAl interaction, Sapphire/TiAl composites remain nearly unknown because fibres to be used in such composites have not been really available. At the present time, such fibres are developed in Solid State Physics Inst. of RAS. The results of preliminary creep tests of Al_2O_3/TiAl composites obtained by using pressure casting have shown that usage of such composite systems shifts the temperature limit for light structural materials in terms of creep resistance to, at least, 1050°C: creep strength on 100 h time base reaches 120 MPa at that temperature. It occurs also that Sapphire-fibres/TiAl-matrix composite specimens have an increased gas corrosion resistance by more than one order of the magnitudes as compared with that of the matrix alloy.

Keywords:

Sapphire fibre, titanium aluminide, composite, microstructure, creep, oxidation resistance

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1. Introduction:

An obvious necessity to have light materials for temperature interval 800-1000°C yields the development of titanium-aluminide-matrix composites. The most advanced family of such composites is that based on SiC-fibres.
The microstructure and properties of SiC-fibre/titanium-aluminide-matrix composites have been studied and documented sufficiently well (1-4). They have high creep resistance at elevated temperatures; however, an upper limit for their service temperature is set up by chemical reactions on the fibre/matrix interface yielding formation of titanium silicides. The situation is improved by using SiC fibres with an outer layer enriched with carbon.
Single crystalline oxide fibres have been considered as a candidate for reinforcement to titanium aluminides due to high thermodynamic stability of refractory oxides and their "inertness" to many metals and intermetallics (5,6). A limited availability of such fibres up to the very recent times is certainly a reason for rather scarce and sometimes controversial information.
It was stated (7) that sapphire fibres could be introduced into a titanium aluminide matrix by using a liquid fabrication route without a risk of fibre degradation. In Ref (8) an effect of Nb/Y interface in a composite with sapphire fibre was studied in an attempt to prevent a detrimental effect of Ti to Al_2O_3 fibre and to provide a sufficiently weak fibre/matrix interface to enhance the material crack resistance. It is also known (9) that creep resistance of sapphire/γ-TiAl composites at temperatures up to about 1000°C and fracture toughness at ambient temperature can be sufficiently high and an appropriate balance between these two properties can be achieved by weakening the fibre/matrix interface via coating of the fibre with carbon.
The present paper, was stimulated by the availability of single crystalline oxide fibres produced by the internal crystallization method (ICM) developed in SSPI RAS (10,11) in such a way as to ensure the fibre cost sufficiently low for structural applications.
The purpose of present work was to determine the temperature limit of service of light titanium aluminide based material, strengthened by sapphire fibres. The main tasks were to investigate the structure characteristic features of creep and oxidation resistance at temperatures up to 1100°C and to study microalloying effect on these properties.

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2. Results and Discussion:

2.1. Fibre and matrix materials:

Sapphire fibres were produced by ICM, which consists in crystallizing alumina melt in continuous channels pre-made in an auxiliary molybdenum matrix and then extracting the fibres by dissolving molybdenum in an acid solution (10,11). The fibre axis coincides with *c*-axis of sapphire; the Weibull exponent is about 3, the average tensile strength at a length of 1 mm is about 700 MPa at RT. The fibres are similar to those described in (11) and characterized by a rather unusual cross-section shape that can be seen in micrographs of composite specimens presented in Fig.1a. The characteristic size of the fibre in a cross-section is between 80 and 100 μm.
A basic cast alloy for the matrix was either pure Ti-48at%Al intermetallic or that alloyed with either 1at.%Zr or Nb. All the materials had lamellar $\gamma(TiAl)+\alpha_2(Ti_3Al)$ microstructure (Fig.1b). Their 100h-strength is 250-280 and 180-210 MPa at 800 and 825°C, respectively (12).

2.2. Fabrication of composites:

Composite rods with a diameter of about 5 mm and a length of about 65 mm are produced by pressure casting of a matrix alloy melt into a quartz mould containing fibres. The casting temperature – time - pressure regime is 1600°C - 2.5 min - 0.6 MPa. The cooling time to a temperature of 1450° is 10 min. Details of the equipment developed in SSPI RAS and used in the present work is described in (13). The average fibre volume fraction is between 0.20 and 0.25. Fibres are rather gomogeniously distributed in the matrix (Fig.1a).

2.3. Microstructure and fracture surface:

Limited dissolution of the fibres occurs upon their impregnation under pressure with the liquid TiAl-matrix. Initially sharp corners of the ICM-fibres become rounded as seen in Fig.1c. The contact between liquid matrix and quartz mould cause the formation of silicon containing phases that seen as white inclusions in the matrix (Fig.1c). There are two types of inclusions: $Ti_{62}Al_{12}Si_{26}$ and $Ti_{62}Al_{21}Si_{17}$. These compositions are close to the $Ti_5(Al,Si)_3$ compound on the Ti_5Si_3 base. Total content of these phases is too small to be

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determined by X-ray analysis. In the TiAl-matrix alloyed with 1at.%Zr, it was found that about 2,5at.%Zr replaced Ti in these ternary compounds.

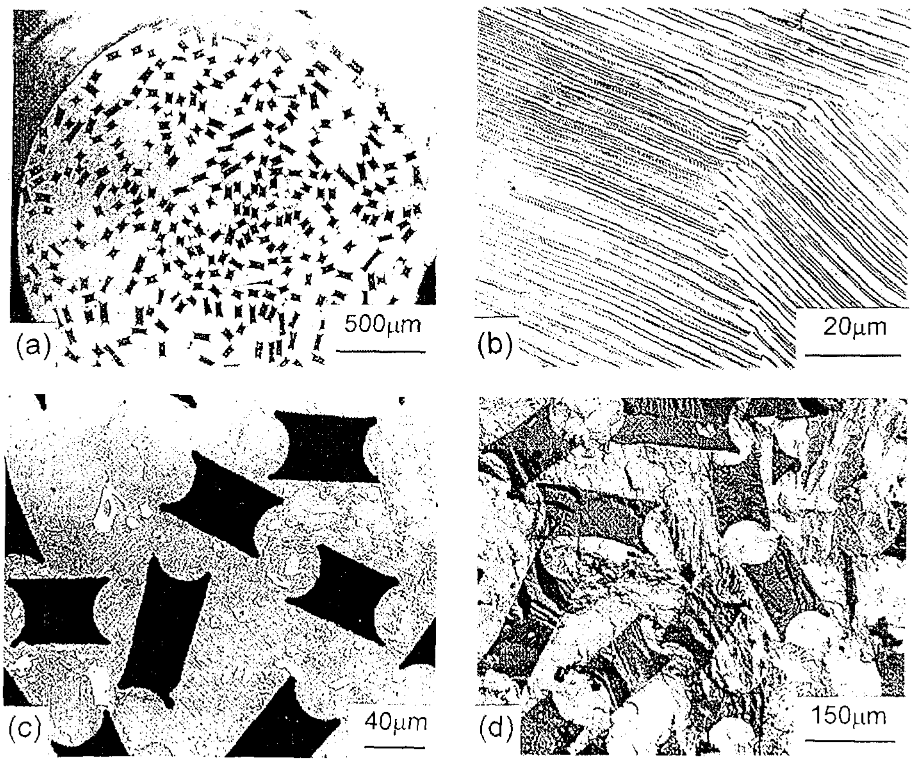

Figure 1. SEM micrographs of the cross-sections (a), (b), (c) and fracture surface (d) of the Sapphire/TiAl composites. (b) TiAl-matix as-cast alloy.

The characteristic microstructure (Fig.1c) and fracture surface (Fig.1d) reveal, on the qualitative level, a high interface strength in Sapphire/TiAl composites. This allows to load the fibres in a very weak matrix to large stresses.

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However, a high strength of the interface in can cause a low crack resistance (fracture toughness) due to easy crack propagation between sapphire fibre and TiAl-matrix without changing propagation direction along the interface.

2.4. Creep tests:

Since a temperature limits for the service of titanium-aluminide-based materials with sapphire fibres is to be higher than that of TiAl alloys, the creep tests were started at the temperatures higher than an upper temperature usage limit for titanium-aluminide alloys (that is about 850°C). All the tests were conducted in vacuum in order to avoid the effect of atmosphere. 3-points bending tests were performed due to its simplicity; a length between supports is ~42 mm. Each specimen was loaded by a step-wise increasing force to determine a stress sensitivity of the deflection rate and, characteristic stress in the creep law that can be intrinsic to a particular specimen. Details of the experimental technique and interpretation of the results are described in Refs (14,15).

The steady state portions of the curves were used to calculate creep properties of each specimen. Two examples of the flexure creep curve are presented in Fig.2.

2.5. Discussion of creep tests results:

An interpretation of the bending creep data obtained at variable loading by using corresponding creep models and introducing them into a calculation of a creep response of a rod under bending yields tensile creep characteristics of the composite and needs a special technique described in detail elsewhere (16,17). The technique is based on the assumption of a unique power law of the composites for creep under tension and compression:

$$\dot{\varepsilon} = \eta_n \left(\frac{\sigma}{\sigma_n} \right)^n \qquad (1)$$

where η_n, σ_n, and n are constant, one of which can be chosen arbitrarily, so in what follows we take $\eta_n = 10^{-4}\,h^{-1}$. A solution of a simple creep problem for 3-point bending of a beam with a circular cross-section yields:

$$\dot{f} = \eta_n \frac{L}{2^{2(n+1)}(n+2)\mu(n)} \left(\frac{Q}{\sigma_n R^2} \right)^n \left(\frac{L}{R} \right)^{n+1} \qquad (2)$$

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where $R = d/2$.

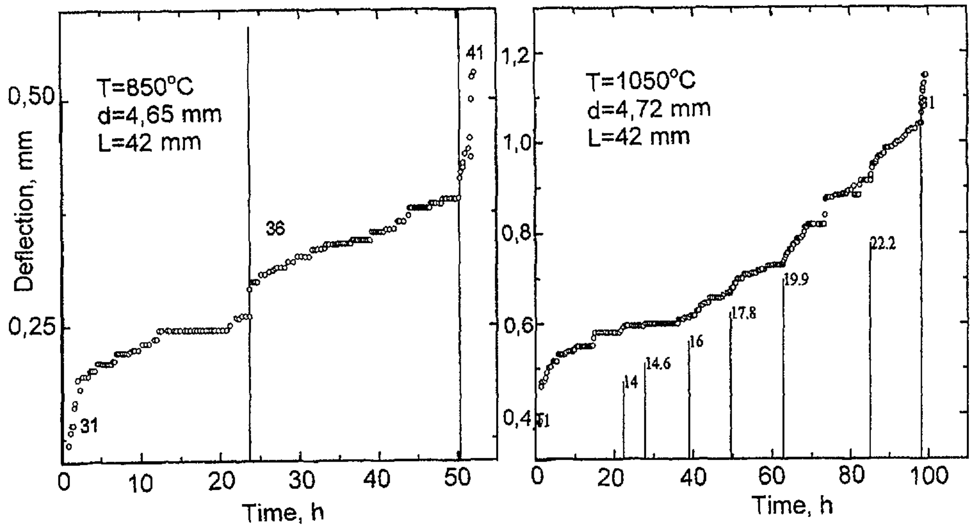

Figure 2. Examples of the creep curves of composite specimen tested in 3-point bending. Deflection at the specimen center versus time under step-wise loading of the CM specimen. Load values (in kgf) are shown near the lines.

More 20 samples was tested at loads 11 kgf at 1100°C, 17,8-34,6 kgf at 1050°C, 24-33,1 kgf at 1000°C and 26-46 kgf at 850-900°C. The dependence of $\dot{f}$ on Q for each specimen gives a value of *n* for a particular specimen. If we have *n*, we express σ_n for each particular set of the values of $\dot{f}$ and Q by using Equation (2) and then average the values of σ_n over all points for one specimen to obtain the tensile creep characteristics, expressed by Equation (1), for a particular specimen. At $\eta_n = 10^{-4}h^{-1}$ the value of σ_n is the stress to cause 1% of creep strain for 100 h (Fig.3). Doping the basic matrix alloy with zirconium, which is similar to titanium, does not change the creep resistance

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of the composites. Doping the basic matrix alloy with 1at.%Nb decreases the strength and creep resistance of the composites at 1050°C.

The stress sensitivity of creep rate characterized by the value of n intense decreases with temperature increasing: n = 17, 15, 7, 6, and 5 at 850, 900, 1000, 1050, and 1100°C, respectively.

If to take in account the matrix properties at 1050°C and features of temperature dependence of n it can be thought that the matrix contribution in tensile stresses cannot be neglected even at such a high temperature. If $\sigma_{ll} \approx 120$ MPa for the CM at 1050°C (see Fig.3), then the effective stress on the fibres is about 500 MPa.

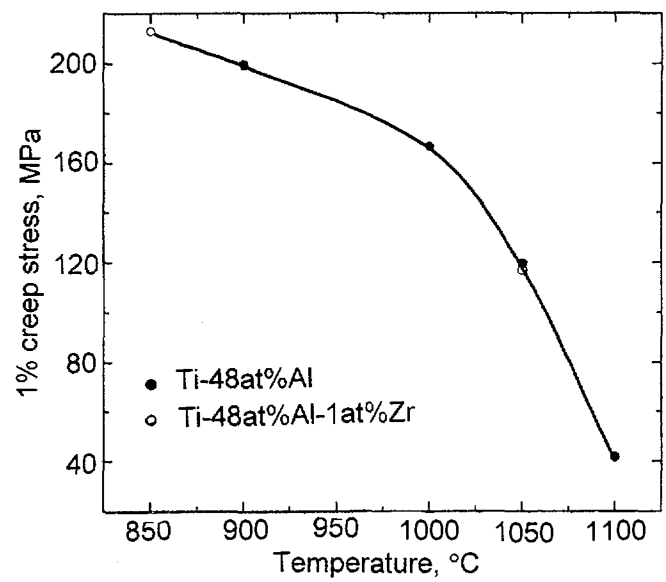

Figure 3. Stress to cause 1% creep strain for 100 h versus testing temperature.

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This nearly corresponds to the effective strength of sapphire fibres in a molybdenum matrix, of ≈620 MPa, at the same temperature (15). The molybdenum matrix strength (15) is by an order of the magnitudes larger that the stress in the TiAl-matrix under creep conditions, hence, the critical fibre length in the Mo parent matrix is essentially smaller than that in the TiAl-matrix.

3. Resistance of CM to atmosphere corrosion:

The weight gain was measured after testing at 1000°C in air for 100 h. The data on the oxidation resistance of the Ti-48at.%Al matrix and Sapphire/TiAl composites with matrix alloyed with Si, Zr, V, Nb in comparison with published data on the oxidation resistance of the matrix alloys are shown in Fig.4a,b (18-20). Most of the data on the oxidation resistance of TiAl alloys were published for temperatures below 900°C. This complicates their comparison with the present data. The mass gain at 1000°C of Ti-47at.%Al alloy (18) is nearly the same, as in the present work on the Ti-48at.%Al matrix alloy ($\Delta g = 1000 \pm 100$ $g \cdot m^{-2}$ to 100 h). For composites with 20-25vol.% Sapphire-fibre/TiAl-matrix (doped ≤2,5at.%Si during preparation of specimens in quartz mould), the weight gain at 1000°C is smaller than that of TiAl alloys, doped 0,5at%Si or 2-2,5at.%Mo at 900°C (21). Comparison of all the data allows to conclude that both Zr and Nb increase the corrosion resistance of TiAl+Si, whereas V and Ta decrease it. However, in all the cases, the corrosion resistance of Sapphire/TiAl-alloy composites is much higher than that of the TiAl-alloy matrix. In any case, alloying the matrix is a resource to decrease the interface strength in these composites to improve low-temperature fracture toughness.

4. Conclusion:

The fibrous composites of a new type exceed other heat resistant materials by specific 100-h strength at 1050°C. Single-crystal sapphire ICM-fibres/Ti-48at.%Al-2,5at.%Si-matrix can reach service temperature level of ~1050°C in terms of creep and atmosphere corrosion resistance. This is by about 200-250°C higher than that of TiAl alloys.

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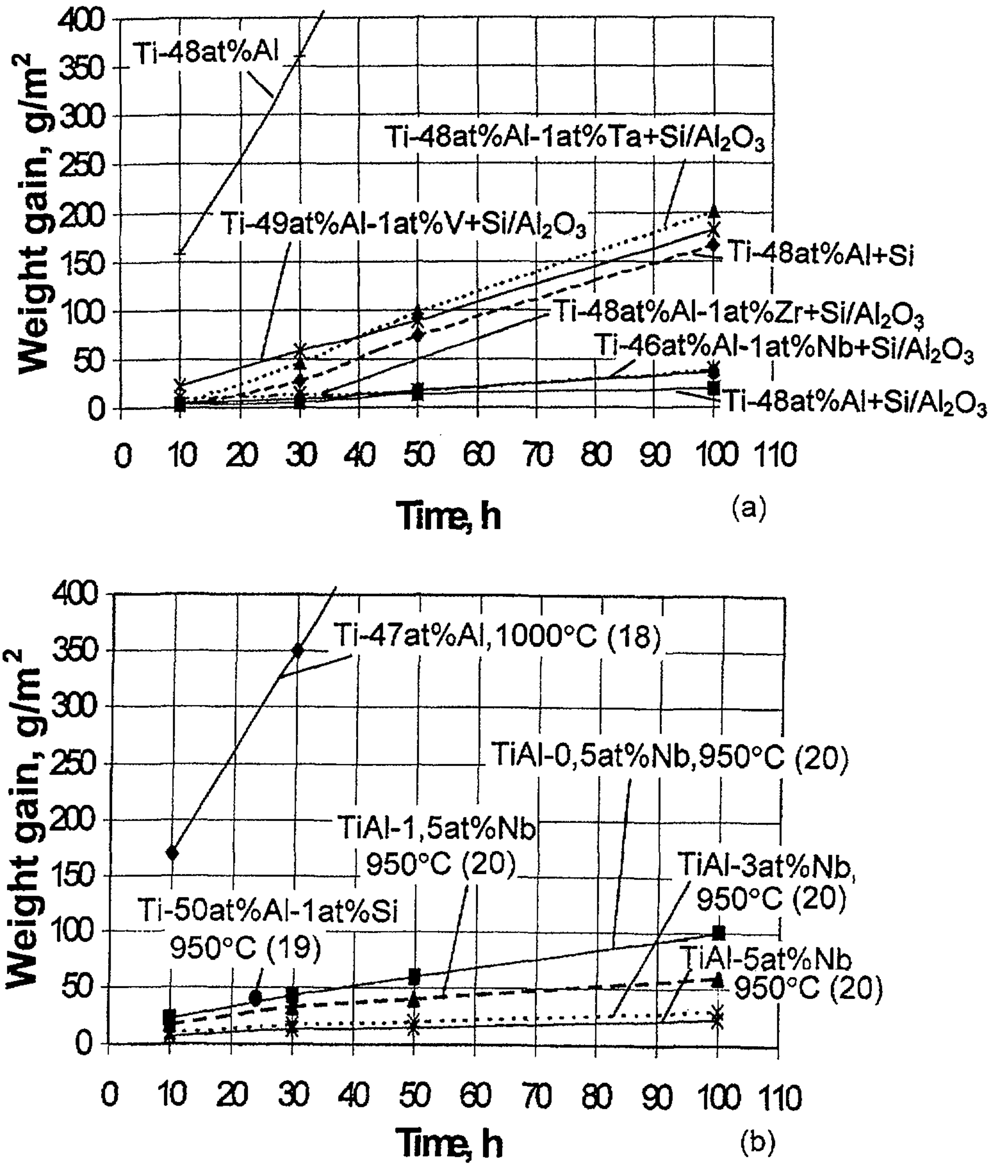

Figure 4. Weight gain in air versus time for the (a) TiAl-matrix and Al_2O_3/TiAl composites at 1000°C (this work) and (b) TiAl alloys at 950-1000°C (18-20).

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