MELT GROWTH

Growth of Oxide Fibers by the Internal Crystallization Method

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Abstract—A new approach is developed for growing relatively inexpensive single-crystal and eutectic oxide fibers suitable for the fabrication of high-temperature composite materials. Sapphire (Al₂O₃), YAG (Y₃Al₅O₁₂), mullite (3Al₂O₃ · 2SiO₂), YAP (YAIO₃), and eutectic (Al₂O₃-Y₃Al₅O₁₂, Al₂O₃-ZrO₂(Y₂O₃), and Al₂O₃-Gd₂O₃) fibers are produced by the internal crystallization method, and their mechanical strength and microstructures are studied. © 2002 MAIK "Nauka/Interperiodica".

INTRODUCTION

The development of high-temperature materials retaining good mechanical properties above 1100°C is vital for improving the performance of heat engines, in particular, gas turbines (raising their efficiency, reducing exhaust emission, etc.). It is becoming increasingly evident that this problem can only be solved by devising advanced fiber-reinforced metal-, intermetallic-, and ceramic-matrix composites. For this purpose, structurally reliable fibers with a high creep resistance at working temperatures are required. The available reinforcing fibers for high-temperature applications, based mainly on oxides and silicon carbide, are not structurally stable above 1200°C [1]. For this reason, efforts are focused on melt-grown single-crystal and eutectic oxide fibers.

Figure 1 schematically illustrates the main methods for the melt growth of single-crystal fibers. Edgedefined film-fed growth (EFG) [2] is a modification of the Stepanov technique [3], as is micro pulling down (μ -PD) [4]. Wide use is also made of laser heating for crucibleless crystal growth: the laser-heated floating zone (LHFZ) technique [5] and laser-heated pedestal growth (LHPG) [6].

The oxide fibers grown from melt by the above techniques find application for the most part in optics but are too costly to be used in composites. It is the lack of inexpensive oxide fibers that impedes the development of oxide-fiber-reinforced metal- and ceramic-matrix composites. Therefore, one of the challenges in this area is to devise new fabrication techniques that would notably reduce the cost of fibers.

The internal crystallization method (ICM) is a new approach to producing single-crystal and eutectic oxide fibers. A key feature of this method is directional melt solidification in capillary channels made in a metallic block. The method was first employed to produce molybdenum-matrix composites [7, 8]. Later, efforts were focused on the growth and subsequent extraction of fibers from the matrix.

This paper outlines the use of the ICM for producing relatively cheap oxide fibers. Data are presented on the microstructure and mechanical strength of various ICM fibers.

GROWTH AND MICROSTRUCTURE OF ICM FIBERS

Continuous cylindrical channels in an accessory (molybdenum) matrix can be formed in a number of ways. At present, use is commonly made of diffusion bonding under special conditions to give an assembly of alternating foils and wires. The growth process, schematized in Fig. 1d, yields fibers of cross-sectional size *t*. Diffusion bonding conditions depend on the matrix material and geometry. To grow oxide fibers, molybdenum assemblies with a wire diameter of d = 0.05-0.2 mm are commonly used.

Infiltration and fiber growth are carried out in induction-heated units (Kristall-603 and RUMO-1S) in an argon atmosphere at a pressure of 1.1–1.3 atm or in a resistance-heated vacuum unit (ChIR). The inductionheated units are equipped with graphite susceptors and molybdenum crucibles; the largest possible assembly length is 100 mm. In the ChIR unit, fitted with a tungsten resistance heater, the assembly length can be up to 250 mm. The high-temperature zones of these units enable growth of up to 150 g of fibers per run.

Sapphire (Al_2O_3) fibers possess a unique combination of physical properties for use in composites: a melting point as high as 2053°C, good resistance to aggressive attack, and high strength at temperatures above 1000°C.



Fig. 1. Main methods for melt growth of fibers: (a) edge-defined film-fed growth (Stepanov technique), (b) micro-pulling-down, (c) laser-heated pedestal growth, (d) internal crystallization method; (1) crystal, (2) seed, (3) melt, (4) die, (5) polycrystalline rod, (6) oxide–molybdenum composite.

It was shown in the very first studies dealing with the ICM [7, 8] that Al_2O_3 crystallization in channels of a molybdenum matrix yields, under certain conditions, single-crystal sapphire fibers. However, their crystallographic orientation may vary even within the same sample. Composites reinforced with such fibers have a low creep resistance above 1000°C since a fraction of the fibers ("ill-oriented" ones) may experience creep. This gives rise to stress redistribution, raising the stress in "well-oriented" fibers, which begin to fracture at relatively low loads. Consequently, sapphire fibers in high-temperature composites must be uniform in crystallographic orientation, preferably with their *c* axis parallel to the fiber axis.

To achieve uniform orientation of ICM fibers, use can be made of oriented seeds [9]. The main steps of the top-seeded growth process are schematized in Fig. 2a. Initially, a molybdenum assembly with empty channels is placed above the melt contained in a crucible and a seed cut from a pregrown sapphire ribbon is mounted on top of the assembly. The crucible is heated to the temperature, determined experimentally, at which the underside of the sapphire seed begins to melt, and the seed subsides. The crucible is then raised to bring the melt into contact with the assembly, and the melt ascends the channels under the action of capillary forces. The next step is directional solidification of the melt in the channels, which takes place as the assembly and seed are elevated to the low-temperature zone at a pulling rate of 1 to 30 mm/min. Fiber growth begins at the seed, and the growth front moves down the channels. The assembly is pulled until uniformly oriented sapphire fibers are grown throughout the channels (the assembly is above the molybdenum ring). Next, the temperature is lowered at 30°C/min. Figure 3 shows a set of assemblies (with platelike seeds on top) after fiber growth, and Fig. 4 shows sapphire fibers after dissolving away the molybdenum matrix in a mixture of HCl, HNO₃, and H₂O.

Examination by optical microscopy reveals two salient features of the fiber microstructure. First, the flat fiber surface is a negative replica of the surface of the molybdenum foil. Since Mo recrystallizes during fiber growth, its surface contains \sim 1-µm-high steps along grain boundaries (Fig. 5). Second, the fibers contain linear arrays of pores just below the surface.

Polarized-light images demonstrate that the ICMgrown sapphire fibers are single-crystal throughout their length, independent of the solidification rate. As the rotation angle of a flat bundle of fibers relative to crossed nicols changes from 0° to 45° , complete extinction is observed [9]. The orientation of the grown fibers coincides with that of the seed.



Fig. 2. Sequence of steps (1-5) in seeded fiber growth: (a) melt supply from a crucible, (b) crucibleless process. The schematics show the seed (\boxtimes), empty capillary channels (\square), melt-filled channels (\blacksquare), channels containing grown fibers (\blacksquare), and melt (\boxtimes).

Yttrium aluminum garnet (YAG), $Y_3Al_5O_{12}$, *fibers* were grown by the infiltration–solidification process described above with application to sapphire fibers (Fig. 2a) and also by a crucibleless process (Fig. 2b). In the latter case, during partial melting of a YAG crystal mounted on top of the molybdenum assembly, the melt fills the channels from the top down. The remainder of the crystal serves as a seed during subsequent melt solidification to yield uniformly oriented single-crystal YAG fibers (Fig. 6), as in the case of melt supply from a crucible.

Typical of the ICM, the surface of YAG fibers is a negative replica of the Mo foil surface. Phase analysis of powder samples indicates that the predominant phase in the fibers is YAG. Rarely do the fibers contain trace levels of Al_2O_3 and $YAIO_3$, which may result from melt overheating.

Eutectics crystallize from the melt without seeding. The creep resistance of eutectic fibers is independent of the crystallographic orientation of the constituent phases and seems to be determined by the deceleration of dislocations at interfaces. Eutectic fibers are fabricated by a process similar to the one schematized in Fig. 2a, but without seeding. In this way, Al_2O_3 -

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 $Y_3Al_5O_{12}$, Al_2O_3 –ZrO₂(Y_2O_3), Al_2O_3 –AlGdO₃, and other eutectic fibers were produced. The surface morphology of eutectic fibers is characteristic of the ICM. Figure 7 shows the cross-sectional image of an Al_2O_3 – $Y_3Al_5O_{12}$ fiber, which has a Chinese script eutectic microstructure with a characteristic cross-sectional colony size of 2–4 µm. At the curved fiber surfaces, the eutectic microstructure may be rougher (7–10 µm), pointing to a reduced solidification rate in those regions owing to the influence of the molybdenum wire.

Mullite $(3Al_2O_3 \cdot 2SiO_2)$ in the form of single-crystal fibers can also be prepared by the ICM [10]. Given that mullite melts incongruently, such fibers can hardly be produced by other melt growth processes. Single-crystal mullite fibers exhibit the best creep behavior above 1400°C. The preparation of such fibers opens up a possibility of raising the working temperature of composites by replacing nickel alloys with higher temperature matrices, probably intermetallics or oxides.

Polarized-light images demonstrate that ICMgrown mullite fibers are single-crystal throughout their length (Fig. 8), although some of them are cut across by Al_2O_3 grains. The influence of these flaws on the strength of the fibers seems, however, to be significant



Fig. 3. Molybdenum assemblies and seeds after fiber growth in the channels.



Fig. 4. A bundle of uniformly oriented ICM-grown sapphire fibers after dissolving away the molybdenum matrix.

only on a fairly large length scale, which is of little importance to the mechanical properties of the composite, provided it has been adequately designed.



Fig. 5. ICM-grown sapphire fiber reproducing the surface contour of the molybdenum foil and containing pores in the surface region.

STRENGTH OF FIBERS

Determination of Strength Characteristics

The cross-sectional shape of ICM fibers hampers the use of conventional testing techniques. In view of this, a special procedure was devised to assess the room-temperature strength of fibers [11]. A fiber with a cross section similar to the one shown in Fig. 1d, of thickness d, is bent around a rigid cylinder with a relatively large radius R, and the number of breaks is counted. If the number of breaks is sufficiently small, i.e., the average separation between neighboring breaks exceeds d (Fig. 1d) by more than a factor of 10 (end effects can be neglected), the fiber is then bent around a smaller radius cylinder. The procedure is repeated until the average separation between breaks is approximately 10d. Typically, five to seven fibers chosen at random are tested.

The highest stress in a fiber bent around a rigid cylinder is given by

$$\sigma = E \frac{d}{2R},\tag{1}$$

where *E* is Young's modulus of the fiber material. It can be taken as a first approximation that σ is the bending strength of the fiber measured over a gauge length equal to the average separation between fiber breaks. Clearly, this will lead, in subsequent calculations, to a fiber strength somewhat higher than the actual value. However, by reducing the cylinder radius in small steps, the error can be made sufficiently small.

To determine the Weibull parameters describing the tensile strength σ of fibers, use is made of experimental data obtained as described above. The distribution function is used in the form

$$P(\sigma, g) = 1 - \exp\left(-\frac{g}{g_0}\left(\frac{\sigma}{\sigma_0}\right)^{\beta}\right), \qquad (2)$$

where g is a geometric parameter equal to the fiber



Fig. 6. Single-crystal YAG fibers.



Fig. 7. Cross-sectional microstructure of an Al_2O_3 - $Y_3Al_5O_{12}$ eutectic fiber.

length *l*, surface area *S*, or volume *V*, depending on the particular distribution of characteristic defects in the fiber; g_0 is the corresponding constant (l_0 , S_0 , and V_0 are constants having the dimensions of length, area, and volume, respectively); and σ_0 and β are the other two Weibull parameters. Clearly, one of the three parameters can be chosen arbitrarily. The average strength of a fiber with a geometric parameter *g* can be obtained from Eq. (2) in the form

$$\langle \sigma(g) \rangle = \int_{0}^{\infty} \sigma dP(\sigma) = \sigma_0 \left(\frac{g}{g_0}\right)^{\beta} \Gamma\left(1 + \frac{1}{\beta}\right),$$
 (3)

where Γ is the gamma function.

As shown recently [11], the assumption that defects form linear arrays on the fiber surface is inconsistent with experimental data. At the same time, mechanical tests, taken alone, provide no sufficient evidence to conclude whether a surface or volume defect distribution is actually the case. Moreover, it cannot be ruled out that there are two or more distinct defect systems responsible for fiber failure.

Let l^0 be a characteristic fiber length. Then, for a group of fibers, g is equal either to $V_0 = l^0 A_{av}$ or to $S_0 = l^0 P_{av}$, where A_{av} and P_{av} are, respectively, the average cross-sectional area and average perimeter of the fibers. V_0 and S_0 appear in the scaling relations

$$\sigma^*(V) = \sigma_{0V}^{\mathsf{b}} \left(\frac{V}{V_0} \right)^{-1/\beta}, \quad \sigma^*(S) = \sigma_{0S}^{\mathsf{b}} \left(\frac{S}{S_0} \right)^{-1/\beta},$$

where the pairs of Weibull parameters $[\beta, \sigma_{0V}^{b}]$ and $[\beta, \sigma_{0S}^{b}]$ referring to bending can be determined from experimental data using Eq. (2).

The Weibull parameters for tensile strength can be determined in a similar way, by integrating Eq. (2) under the assumption that defects are present only in the surface layer or throughout the fibers [11]. This procedure yields the following expressions for the tensile



Fig. 8. Polarized-light image of mullite fibers.

strength σ^t in the cases of volume (0V) and surface (0S) distributions, respectively:

$$\sigma_{0V}^{t} = M_{V}(\beta)\sigma_{0V}^{b}, \quad \sigma_{0S}^{t} = m_{S}(\beta)\sigma_{0S}^{b}.$$

Here, $M_{V} = \left(\gamma - \frac{\pi}{4}\right)^{-1/\beta} \left[\int_{0}^{1} \xi^{\beta}(\gamma - \sqrt{1 - \xi^{2}})d\xi\right]^{1/\beta}$ and
 $M_{S} = \left(\frac{(\beta + 1)(\pi + 2\gamma)}{2}\right)^{-1/\beta}$, where $\gamma = t/d$ (Fig. 1d).

The effective characteristics of high-temperature tensile strength were determined by testing oxide-fiber-reinforced molybdenum-matrix composites. The experimental procedure and results were described in detail in [12].

Measurement Results

Sapphire fibers. Figure 9 shows a log–log plot of bending strength σ^* versus average distance between fiber breaks. Such data are used to evaluate the characteristics of tensile strength as described above. The results for oxide fibers prepared at pulling rates of 1.3, 3.3, and 30 mm/min are summarized in Table 1. The pulling rate was found to have an insignificant effect on the strength of the fibers.

The experimental data in Fig. 9 and Table 1 demonstrate that an ~1- μ m-thick metal, ceramic, or pyrolytic carbon coating notably raises the strength of the fibers. This finding suggests two important conclusions. First, it is clear that a substantial fraction of the characteristic defects, which determine the strength of the fibers, reside in the surface layer. Second, since the effect of the matrix would be expected to be similar to that of a coating, the composite strength cannot be assessed by measuring only the strength characteristics of individual fibers. The same refers to the strength characteristics determined by testing fibers removed from the matrix—the procedure sometimes used to analyze the effect of fiber–matrix interaction on fiber strength [13].



Fig. 9. Log–log plot of bending strength vs. fiber length for two groups of sapphire fibers (Table 1): no. 1 (uncoated, open circles) and no. 1SCO (coated with ~1- μ m SiC_{1-x}O_x, filled circles).



Fig. 11. Log–log plot of bending strength vs. fiber length for uncoated (open circles) and ~ 1 -µm SiC_{1-x}O_x coated (filled circles) sapphire–YAG fibers.

YAG fibers. The experimental data are displayed in Fig. 10. Table 1 lists the Weibull parameters for four groups of fibers. The strength characteristics are seen to be independent of the pulling rate and vary little.

YAG–sapphire eutectic fibers. A set of experimental data on the strength of such fibers is presented in Fig. 11. As in the case for sapphire fibers, coatings notably increase fiber strength. Typical Weibull parameters are listed in Table 1.

Single-crystal mullite fibers. The experimental data are shown in Fig. 12, and the Weibull parameters for the group of fibers tested are listed in Table 1.

Other fibers. We also determined the strength characteristics of some other fibers, in particular, Al_2O_3 -based eutectic fibers. The experimental data and Weibull parameters for Al_2O_3 -ZrO₂ and Al_2O_3 -GdAlO₃ eutectic fibers are presented in Table 1. Note the rather high strength of Al_2O_3 -ZrO₂ fibers.



Fig. 10. Log–log plot of bending strength vs. fiber length for two groups of YAG fibers (Table 1): no. 4 (filled circles) and no. 5 (open circles).



Fig. 12. Log–log plot of bending strength vs. average distance between fiber breaks for mullite fibers (Table 1, no. 6).

SOME OBSERVATIONS ABOUT THE COST OF FIBERS

At this stage, the actual cost of fibers cannot be evaluated precisely. Some estimates are nevertheless possible. Under the assumption that the cost of crystals is dominated by the power consumption for crystal growth, the substantial difference in cost between large oxide crystals (hundreds of US dollars per kilogram) and sapphire fibers (tens of thousands of US dollars per kilogram) is determined by the productivity of the growth process and, hence, the associated power losses.¹ Since the ICM is comparable in productivity to the growth of bulk single crystals, the processes involved must be comparable in power consumption.

¹ In spite of its rather low efficiency in electric power–light–heat conversion, laser heating and local melting (LHPG method) is viewed as a viable approach to fiber fabrication, which lends support to the above assumption.

GROWTH OF OXIDE FIBERS

No.	Fiber material	β	σ_{0V}^{b} , MPa	σ_{0V}^{t} , MPa	$V_0, {\rm mm}^3$	β	σ_{0S}^{b} , MPa	σ_{0S}^{t} , MPa	S_0 , mm ³
		volume defects				surface defects			
1	Al ₂ O ₃	3.07	1783	1457	0.00158	3.17	1133	536	1.782
1 SCO	Al_2O_3	3.25	2709	2221	0.00148	3.27	1262	607	5.242
2 Ni	Al_2O_3	2.81	1345	861	0.01336	2.85	843	296	2.669
2 NiO	Al_2O_3	1.82	1986	1162	0.01370	1.98	933	235	2.853
3	Al_2O_3	3.00	2146	1402	0.01718	3.00	1360	517	3.171
3 C	Al_2O_3	2.95	3273	2172	0.01199	2.96	1523	612	6.782
4	$Y_3Al_5O_{12}$	4.74	1013	728	0.0091	4.74	681	352	3.93
5	$Y_3Al_5O_{12}$	2.71	1225	796	0.0089	2.74	693	260	2.81
6	$3Al_2O_3 \cdot 2SiO_2$	3.42	1113	759.1	0.007766	3.44	783.7	346.1	1.861
7	Al ₂ O ₃ –ZrO ₂	2.63	1674	1073	0.0101	2.63	788	280	4.434
8	Al ₂ O ₃ –GdAlO ₃	3.52	567	393	0.0063	3.37	495	221	0.864

Table 1. Strength characteristics of oxide fibers; $l^0 = 1 \text{ mm}$; E = 450 (fibers 1–3), 392 (4), 362 (5), 320 (6), and 300 GPa (7)

Note: SCO, C, Ni, and NiO designate ~1- μ m-thick SiC_{1-x}O_x, pyrolytic carbon, Ni, and NiO coatings, respectively.

 Table 2. Nickel-based matrix composites (X marks the fiber-matrix combinations studied)

Fiber⇒ Matrix↓	Al ₂ O ₃	Y ₃ Al ₂ O ₁₂	Al ₂ O ₃ /Y ₃ Al ₅ O ₁₂ eutectic	Al ₂ O ₃ /ZrO ₂ (Y ₂ O ₃) eutectic
VKNA-4U	X	X	X	X
ZhS-32	X	X	X	

The increase in the cost of fibers due to the use of molybdenum does not radically change the situation. Consequently, ICM fibers must be comparable in cost to bulk single crystals and can be used in various structural and, of course, high-temperature composites.

SOME APPLICATIONS OF OXIDE FIBERS

The range of potential applications of single-crystal oxide fibers, exhibiting various physical properties, is extremely broad. Here, we focus on the use of oxide fibers in structural composites. The corresponding fabrication processes were described in detail in a previous publication [14]. Microstructural analysis of the composites is beyond the scope of this article.

Application of Fibers in High-Temperature Nickel-Based Matrix Composites

Preliminary Remarks. Table 2 lists some of the composites studied, which were prepared by injection molding in the form of either cylindrical rods for bend tests or dumbbell-shaped specimens for tension tests. Note that almost all the composites were also tested by a pushout technique to evaluate the fiber-matrix bond strength.

To achieve high strength of ICM fibers in a composite, one must ensure a sufficiently high fiber-matrix bond strength for short fibers to be fully loaded [15]. Control over interfacial bonding in oxide-fiber-reinforced nickel-based matrix composites produced via liquid-phase processing is the most important technical problem in composite fabrication.

Creep tests and interpretation of results. Most mechanical tests on nickel-matrix composites were performed in the range 1100–1200°C because the creep resistance of composites in this range determines their potential for high-temperature applications. At these high temperatures, even short-term strength tests are, in essence, creep tests, even though they involve fairly high strain rates (high crosshead speeds in the testing machine). In view of this, in interpreting the results, we will not differentiate between "short-term" and "normal" creep tests at creep rates of, e.g., 10^{-4} to 10^{-2} h⁻¹. All tests were performed in vacuum. The load was measured by a dynamometer mounted in a glass bell jar. The crosshead speed was 2 mm/min. Vacuum testing allows one to exclude the effect of the ambient atmosphere on the creep behavior of composites. Clearly, real materials must be protected by coating to prevent oxidation of the matrix and produce a thermal barrier.

The results of creep tests in bending can be used to assess tensile creep characteristics expressed through the *n* and σ_n parameters in the power-law relation

$$\dot{\varepsilon} = \eta_n \left(\frac{\sigma}{\sigma_n}\right)^n,$$
 (4)





Fig. 13. Creep data for composites consisting of Al₂O₃– Y₃Al₅O₁₂ fibers and a ZhS-32 matrix. (a) 1150°C creep resistance vs. volume fraction of fibers. The solid lines show calculation results for a matrix with $\eta_m = 10^{-4} h^{-1}$, m = 2.8, and $\sigma_m = 41.5$ MPa; (1) strong interfacial bonding and high fiber strength: $\alpha = 0.5$, $\beta = 3$, $\sigma_f^0 = 450$ MPa; (2) weak interfacial bonding and low fiber strength: $\alpha = 0.1$, $\beta = 3$, $\sigma_f^0 = 150$ MPa. Fiber geometry: $l_0 = 1$ mm, d = 0.1 mm. (b) Interfacial strength τ^* vs. volume fraction of fibers.

where $\dot{\epsilon}$ is the steady-state creep rate of the composite and $\eta_n = 10^{-4} h^{-1}$ is a constant found by approximately solving the problem of bending creep in a rod and determining exponent *n* by stepwise loading of each specimen. The solution of the problem in question was reported previously [16].

To analyze testing results, we use a model describing creep in a composite consisting of brittle elastic fibers (Young's modulus E_f) and a viscous matrix [16]. The strength of the fibers can be described by Eq. (2) (Weibull statistics). The constitutive relation is taken to have a power-law form, like Eq. (6), with parameters m, σ_m , and $\eta_m = 10^{-4} h^{-1}$. The fiber–matrix interface is an island-type boundary with a continuity coefficient α . At $\alpha = 0$, the interfacial shear strength is zero; at $\alpha = 1$, the interfacial shear strength is equal to that of the matrix (ideal interface). The steady-state creep rate $\dot{\epsilon}$ of the composite and the applied stress σ are related by

$$\sigma = \lambda \sigma_{\rm m} \left[\left(\frac{\sigma_0^{\rm (f)}}{\lambda \sigma_{\rm m}} \right)^{\beta} \left(\frac{l_0}{d} \right) \right]^{\frac{m+1}{n}} \left(\frac{\dot{\epsilon}}{\eta_{\rm m}} \right)^{\frac{1}{n}} V_{\rm f} + \sigma_{\rm m} \left(\frac{\dot{\epsilon}}{\eta_{\rm m}} \right)^{\frac{1}{m}} V_{\rm m}, (5)$$

where $V_{\rm f}$ and $V_{\rm m}$ are the volume fractions of the fibers and matrix, respectively; *d* is the characteristic crosssectional size of the fibers; and λ is a known function, $\lambda(m, \alpha)$.

The results were described in full detail elsewhere [10, 14, 16]. Here, we consider the most important result: the dependence of the creep resistance (the stress which produces a creep strain of 1% over a period of 100 h) on the volume fraction of fibers and interfacial strength (Fig. 13). When the volume fraction of fibers in the composite attains about 25%, the interfacial strength drops sharply, presumably because the matrix becomes deficient in doping elements responsible for interfacial bonding. As a result, the dependence of the creep resistance on the volume fraction of fibers, essentially linear at low $V_{\rm f}$, deviates from the one calculated in the above model.

To interpret such a dependence of the creep resistance on the volume fraction of fibers in the model in question, it is necessary to estimate α as a function of $V_{\rm f}$ from experimental data (Fig. 13b). For high volume fractions of fibers, we take $\alpha = 0.1$ and use the parameters of fiber strength similar to those obtained in an independent experiment. The corresponding calculated curve (Fig. 13a) passes through the data point for the highest $V_{\rm f}$. The behavior of the composite at low $V_{\rm f}$ cannot be described by merely increasing α by about a factor of 5. Adequate results can only be obtained under the assumption that the effective strength of the fibers in the composite with a high interfacial strength exceeds the strength of free-standing fibers by about a factor of 3 (Fig. 14). This implies that, at a high fibermatrix bond strength, the matrix material fills defects on the fiber surface, which have a strong effect on the strength of the fibers. The healing of defects is accompanied by an increase in the effective strength of the fibers in the composite. A similar effect is produced by coating the fibers, as illustrated in Fig. 9 and reported earlier for eutectic fibers [10].

This finding is of key importance not only in describing the properties of ICM fibers but also for the potential applications of these and other oxide fibers. Another important result is that high-temperature composites were obtained. The working temperature of the composites under consideration reaches about 1150°C [16], which is about 75°C higher than that of the best high-temperature alloys.

Application of Fibers in High-Temperature Titanium-Aluminide-Based Matrix Composites

The rather low cost of ICM fibers makes them candidate reinforcing agents for relatively low-temperature



Fig. 14. Creep resistance (100 h) as a function of testing temperature for sapphire-fiber-reinforced TiAl-matrix composites.

matrices, e.g., those based on titanium aluminides. Preliminary experimental results (Fig. 14) [17] demonstrate that such composites, having a density as low as ~4.5 g/cm³, retain an acceptable creep resistance up to ~1050°C, the working temperature of state-of-the-art high-temperature alloys.

Application of Fibers in Oxide–Oxide Composites

Fracture toughness, a key characteristic of ceramicceramic composites, is rather sensitive to the characteristics of the constituent materials and interfacial bonding (it should not be very high) [15]. To assess the serviceability of ICM-fiber-reinforced ceramic (polycrystalline oxide) matrix composites, we carried out model experiments on sapphire–corundum composites containing interfacial pyrolytic carbon layers [18]. The results, displayed in Fig. 15 as fracture toughness versus V_f data, appear encouraging, which is of practical importance because, as pointed out above, ICM fibers exhibit the best creep characteristics at temperatures above 1400°C.

CONCLUSIONS

A new approach was developed for producing single-crystal and eutectic oxide fibers cheap enough to be utilized in the fabrication of composite materials for use at temperatures as high as 1200–1300 (metallic and intermetallic matrices) and 1500–1700°C (ceramic matrices).

Sapphire (Al₂O₃), YAG (Y₃Al₅O₁₂), mullite (3Al₂O₃ · 2SiO₂), YAP (YAlO₃), and eutectic (Al₂O₃-Y₃Al₅O₁₂, Al₂O₃-ZrO₂(Y₂O₃), and Al₂O₃-Gd₂O₃) fibers were pro-

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Fig. 15. Critical stress intensity factor vs. volume fraction of fibers for sapphire-fiber-reinforced polycrystalline corundum matrix composites.

duced by the internal crystallization method, and their strength and microstructures were studied.

It is shown that, by using such fibers in metal-matrix composites, the working temperature of the composites can be raised by at least 75°C. The fibers can also be used in ceramic-matrix composites.

Clearly, ICM-grown oxide fibers, exhibiting a variety of physical properties, can be used to fabricate composites combining high strength with advantageous piezoelectric, optical, and other properties.

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