

# Single crystalline oxide fibres for heat-resistant composites

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## Abstract

A brief review of the methods of fabrication of single crystalline oxide fibres is presented. It is shown that the internal crystallisation method (ICM), that is crystallisation of the fibres in continuous channels in an auxiliary matrix (molybdenum carcass), yields high-productivity rate, which provides a base for the development of fabrication technology of oxide fibres as reinforcement for composites for high and very high use temperatures. The method has been used to produce a family of fibres including sapphire, aluminium–yttrium garnet, mullite as well as some oxide eutectics. Microstructure, strength and high-temperature creep of the fibres are discussed with the emphasis on creep properties. Results of creep tests of composites with matrices based on TiAl, nickel superalloys and oxides are also presented. It is shown that special microstructure of composites with brittle matrices, intermetallic and oxides, yields quasi-ductile behaviour of the composites.

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## 1. Introduction

Most efforts in using advance materials in machines for transportation and power generation have always been directed towards enhancing energy efficiency of the machines, which means decreasing fuel consumption per unit of useful work. Researches have done a lot of research including using structural materials with higher specific characteristics to enhance the ratio of a payload to weight of the structure, improving aerodynamics, applying appropriate coatings on hot details, cooling them, etc. However, enhancing the use temperature of structural materials for hot parts of machines has been and will always be a most efficient way to reach the goal.

Most heat-resistant materials for heavily loaded structural elements, those being Ni-based superalloys, are approaching their physical limit set by their microstructural stability and melting points. Perhaps, this lim-

it will be around 1100 °C and a dependence of the maximum use temperature on the year of alloy development is asymptotically reaching this limit. Since no other metal can be a real base for heat-resistant alloys due to well-known reasons, the only alternative is to exploit ceramics. Homogeneous ceramics cannot be used in heavily loaded structures because of their inherent brittleness. Hence, non-homogeneous materials (composites) being fully or partly ceramic should be considered as future heat-resistant materials.

Crystal oxides have always attracted attention as a potential base for heat-resistant materials because of a number of reasons:

1. They are inherently resistant to oxidation.
2. Single crystals of oxides are inherently strong.
3. They have high-melting points, high elastic moduli, and low density.

Certainly, single crystals of oxides are most promising substances since non-crystalline oxides, first, are

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not to be sufficiently stable at high temperatures (say, at temperatures higher than 1200 °C for alumina) and, second, polycrystals are known to reveal low creep resistance at high temperatures because of the same reasons as those for non-stability. Hence, single crystalline oxide fibres are wanted as a base for heat-resistant composites.

In the present paper, we start with a brief review of the fabrication technology schemes to produce oxide fibres focusing on the internal crystallisation method (ICM) invented by the present author and his colleague, V.I. Kazmin; then proceed with a brief outline of high-temperature creep characteristics of oxide single crystals, then continue with special features of some oxide fibres obtained by ICM. After describing a procedure for evaluating creep properties of the fibres, we will finally present their creep characteristics. Applications of ICM-fibres as reinforcements for composites with Ni-, intermetallic-, and oxide-matrix will also be discussed. Examples of non-brittle behaviour of composites with brittle matrices, TiAl and alumina will be presented.

## 2. Crystallising separate fibres

Certainly, the only way to produce either single crystalline oxides or those with typical eutectic microstructure is to crystallise oxide melts.

### 2.1. Edge feeding growth (EFG)

Edge feeding growth, which was used to produce sapphire fibres for the first time by LaBelle and Mlavsky [1], can actually be positioned within a concept of crystallising a melt by using a shaper, which was formulated by Stepanov before the WWII [2]. Stepanov introduces a shaper to pre-determine a shape and size of the capillary column at the top of which the liquid/solid interface arises (Fig. 1). The technical adjustment of Stepanov's method to crystallising sapphire fibres was a lifting of the growth zone above the melt surface with a capillary tube in the crucible. The lower end of the tube is located near the bottom of the crucible and the growth zone is now fixed relative to the heater independent of the level of the melt surface which goes down with time.

Both a review of the corresponding techniques and discussion of the fibre growth parameters, structure and mechanical properties of sapphire fibres are presented in a paper dated 1985 [3]. It appears that a stable growth takes place at rates no more than 0.5–1.0 mm/s, which makes productivity rate of the process low, so that the cost of the fibres is too high to use them in structural applications. This is despite a certainly realised possibility to grow tens of fibres in a bundle. The macrostructure and strength of sapphire fibres depend

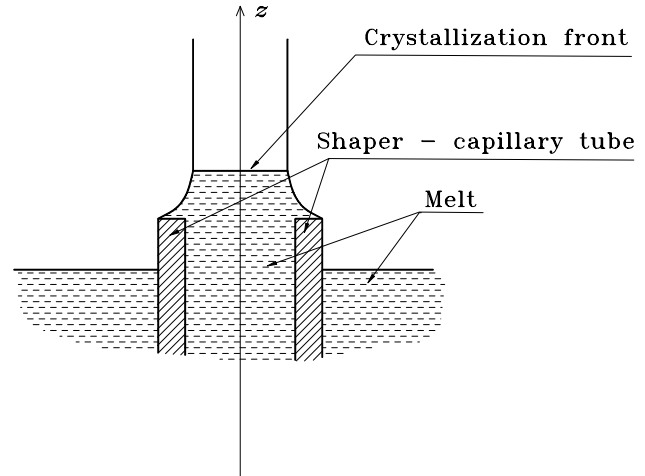


Fig. 1. Capillary shaping-crystallization zone, a schematic view.

strongly on the crystallization rate. Crystallographic orientation of a fibre is determined by that of a seed.

### 2.2. $\mu$ -pulling down ( $\mu$ -PD)

Developers of this method, Japanese researchers [4,5], did actually turn down a scheme presented in Fig. 1, which simplified growth procedures slightly. Fukuda and collaborators have applied the method to produce a variety of oxide fibres [6–8]. They claim that the usage of the  $\mu$ -PD method yields crystals with low thermal strains compared to the other growth methods and using this method makes it possible to grow crystals even from incongruent melts [8]. Obviously, the method does not allow an essential decrease in the fibre cost as compared to that by LaBelle and Mlavsky.

### 2.3. Laser heated pedestal growth (LHPG)

For the first time, single-crystal Cr-doped  $\text{Al}_2\text{O}_3$  fibres were grown by a floating-zone technique from small source rods locally melted at the end by a  $\text{CO}_2$  laser by Burrus and Coldren [9]. Then, it has been used rather intensively to grow mainly optical fibres [10], although attempts to grow structural fibres are also known [11]. There are some obvious advantages of the method: the absence of a crucible allows growing sufficiently pure crystals, a small volume of the melts increases thermal efficiency despite a low efficiency of the laser heating and reduces mass exchange around the process zone that would be useful in growing such materials as mullite, which is characterised by a complicated phase diagram.

### 2.4. General remark

The methods just described have been used to produce continuous oxide fibres of high quality, which are effectively used in various non-structural applications

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