HIGH TEMPERATURE CREEP OF SINGLE CRYSTALLINE AND EUTECTIC OXIDE FIBRES PRODUCED BY INTERNAL CRYSTALLISATION METHOD

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ABSTRACT

The internal crystallization method, which can be used to produce oxide fibres sufficiently cheap for their usage in structural composites, is briefly described. It is shown that a variety of the fibres with high creep resistance at ultra-high temperatures can be obtained by using the method.

1 INTRODUCTION

Well-known methods, such as EFG [1], LHPG [2] and micro-pulling down [3], which are normally used to produce single crystalline oxide fibres, yield fibres of too high cost to be used in structural materials. High temperature mechanical properties of such fibres have not been studied in details, sapphire fibres being rather an exception. Microstructure and high temperature properties of sapphire, aluminium-yttrium garnet (YAG) and alumina-YAG eutectic bulk crystals are rather well characterised (see e.g. [4,5]) and definitely show that composite materials for use at temperatures higher than 1400^{o}C are certainly to be based on either single crystalline or eutectic oxide fibres provided methods of crystallising then are invented that would decrease the fibre cost.

The present authors have invented such a method based on crystallising fibres in an auxiliary molybdenum matrix containing continuous cylindrical channels [6], called the internal crystallisation method (ICM). The method is actually similar to those to produce bulk oxide crystals; hence, the fibres produced by using ICM are expected to be sufficiently cheap to be used in high-temperature structural materials. A special field of interest for application single crystalline oxide fibres is that of composites for very high temperatures. Hence, their creep properties at very high temperatures are of a primary importance.

There is no reason to expect creep properties of the fibres to be different from those of the bulk single crystals. Normally creep characteristics of fibres are being obtained by testing separate fibres. On the same time, it is clear [7,8] that the behaviour of a fibre in matrix can be considerably different from that of a separate fibre. A pragmatic goal of the composites science is to provide such conditions for the fibre, which would lead to an effective use of the fibre in a composite. This certainly calls for a study of the behaviour of fibres in various environments.

Hence, a primary goal of the present study to get creep characteristics of some oxide fibres in an ideal matrix, molybdenum being an example of such matrices, and to start to compare behaviour of oxide fibres in oxide matrices with the ideal case mentioned. The preliminary results of the study are reported in the present paper.

2 INTERNAL CRYSTALLISATION METHOD

Despite the internal crystallisation method to produce single crystalline oxide fibres have been disclosed in a number of publication [6,9,10,11,12] it is still not widely known. Therefore, a brief description of the method is given here.

The schematic of the method is illustrated in Fig. 1. Since a number of the channels can be very large, the method is similar to that of crystallising bulk crystals; hence, the fibre cost is expected to be comparable with that of the bulk crystals.

The method was applied to obtain sapphire, YAG, alumina-YAG eutectic fibres [9,10,11] as well as single crystalline mullite fibres [12,13]. Fig. 2 gives a view of a cross-section of an oxide-fibre/Mo-matrix block before removal of molybdenum by dissolution in a mixture of acids to free the fibres for a following usage. A configuration of the fibre system can be also seen in Fig. 3 that presents another block with the external layer of the matrix being removed.

Fibres can be obtained either as a loose or constrained bundle. Some examples are shown in Fig. 4. It should be noted that single crystalline fibres of lanthanum aluminate and lithium niobate shown in Fig. 3 and Fig. 4b, respectively, can be used in composites with special physical properties. Obviously, a number of such kinds of complex oxides produced in the fibrous form can be very large. Hence, the internal crystallisation method launches a whole family of new oxide fibres.

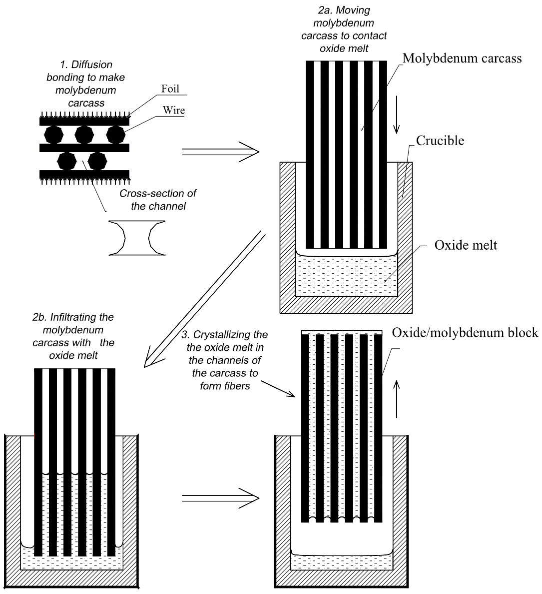


Fig. 1 Schematic view of the internal crystallisation method.

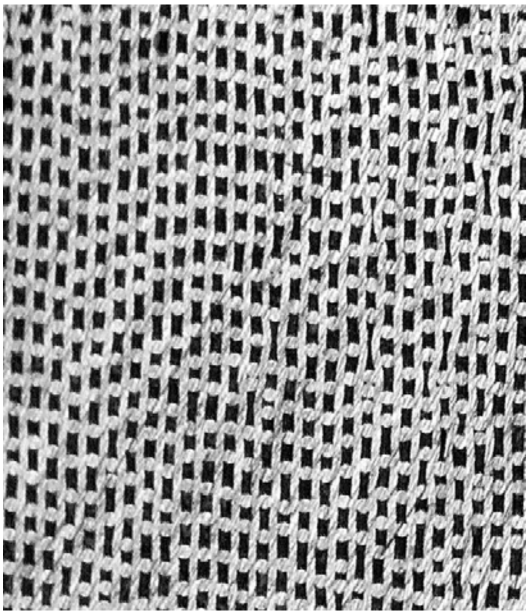


Fig. 2.Cros-section of an oxide/molybdenum block (the fibres are black).

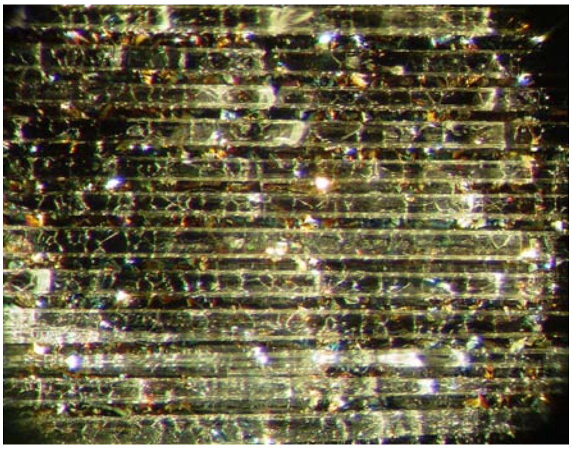


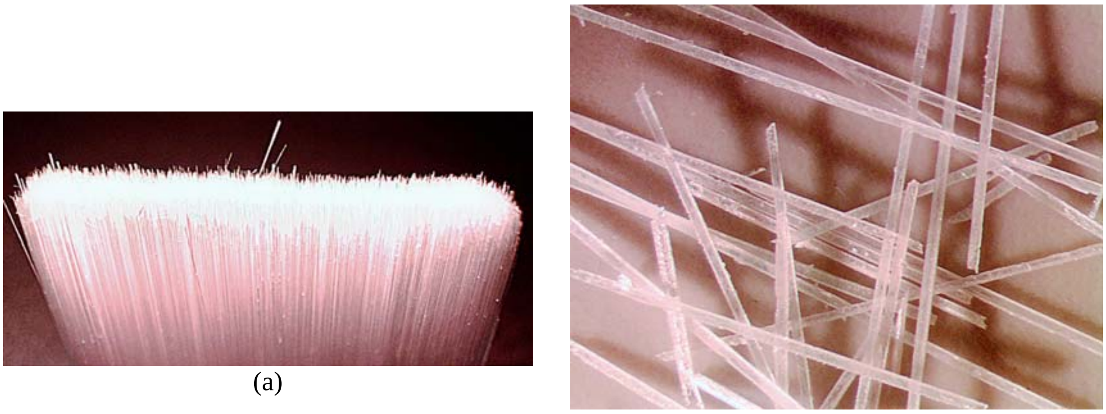
Fig. 3. Fibres of $LaAlO_3$ in an auxiliary matrix with the external layer of the matrix removed.

3 CREEP TESTS

Creep tests were performed on either oxide-fibre/Mo-matrix or oxide/oxide composites. The fibre melts wet molybdenum and the fibres have been crystallised in the channels of the molybdenum matrix the fibre/matrix interface is considered to be an ideal; hence, the fibre strength is not affected by possible surface flaws [8]. A contribution of the matrix, which is fully recrystallised molybdenum, to the creep resistance of the composites even at the lowest temperature, 1000^{o}C, is negligible, less than 10 MPa [6].

Creep tests were performed in either tension or bending. In the latter case, calculation of the tensile creep characteristics by using results of the bending tests was done following a procedure described elsewhere [14].

An example of the original tensile creep curve of a molybdenum-matrix composite is presented in Fig. 5. Such type of the curves was used to estimate creep and rupture stress characteristics of the fibres. An example of the fibre characteristics is given in Fig. 6.



(a)

(b)

Fig. 4. (a) Constrained bundle of sapphire fibres. (b) Separate fibres of lithium niobate.

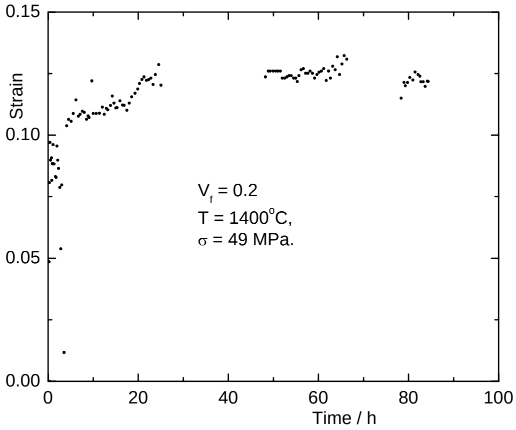


Fig. 5. Tensile creep curve of alumina-YAG-fibre/Mo-matrix composite specimen k10002.

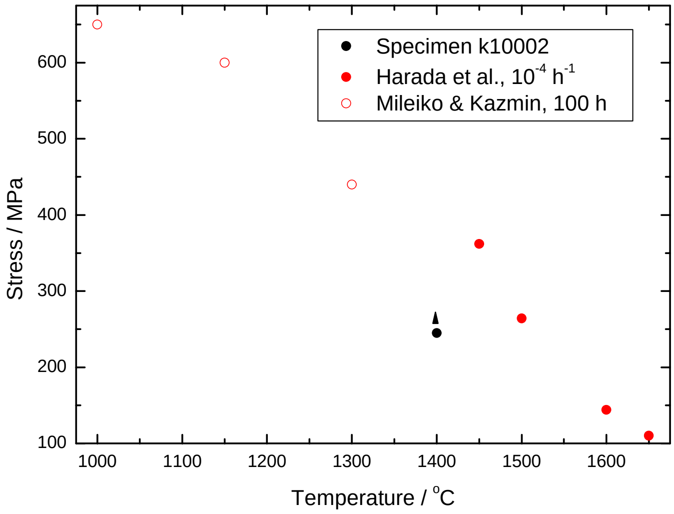


Fig. 6. Creep resistance of alumina-YAG eutectic fibres on the testing time base 100 h versus temperature. Creep curve of specimen k10002 is given in Fig. 5. Data by Mileiko & Kazmin [6] correspond to creep rupture on the 100-h base, those by Harada et al. [4] correspond to stress to cause creep strain 10^{-4} for 100 h (calculated by using the original experimental data).

Similar experimental data have been obtained by testing YAG/Mo specimens up to 1600^{o}C.

Applicability of ICM-fibres in oxide/oxide composites with sufficiently high fracture toughness has been shown earlier [15]. Here we present creep curve of a sapphire/alumina composite specimen without a week interphase at the fibre/matrix interface at a temperature of 1250^{o}C, Fig. 7. A comparison of creep behaviour of unidirectional sapphire/alumina composite with two-dimensional alumina/mullite composites is shown in Fig. 9.

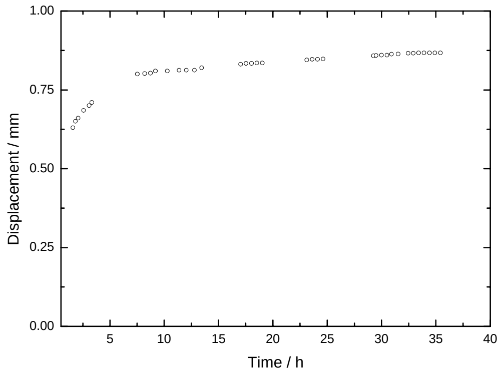


Fig. 7. Creep curve of a unidirectional sapphire/alumina composite specimen (#a0161) in 3-point bending. Fibre volume fraction is ~ 0.4 (see Fig. 8), Specimen diameter is 3.52 mm, distance between supports is 26 mm, load is 39.2 N, temperature is 1250^{o}C.

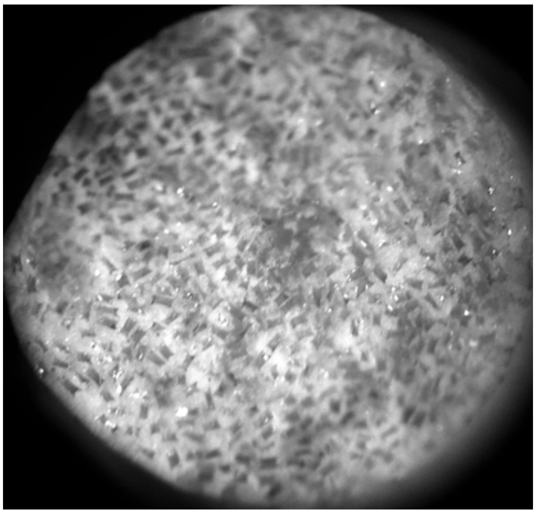


Fig. 8. Cross section of sapphire/alumina specimen #a0161.

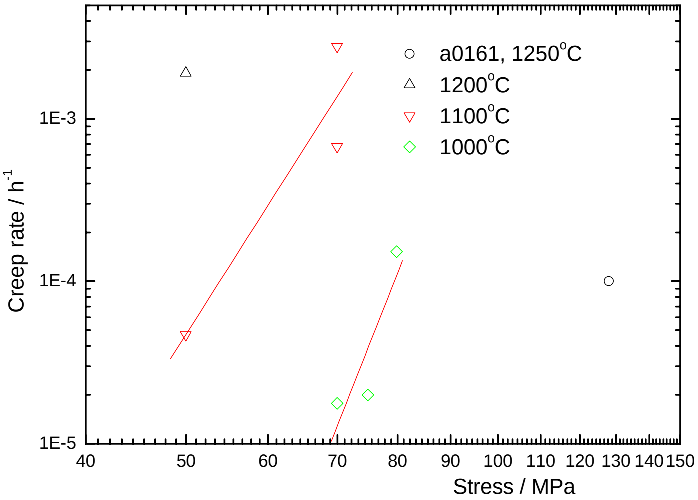


Fig. 9. Creep characteristics of 1D sapphire/alumina composite (Fig. 7 and Fig. 8) in comparison with those of 2D alumina/mullite composites [16].

4 CONCLUSIONS

1. Single crystalline and eutectic fibres produced by the internal crystallization method, which can be a base for fabrication technology of fibres suitable for structural composites, possess creep characteristics comparable with those of bulk crystals.

2. They can be used as reinforcement for ultra-high temperature composites.

3. Internal crystallization method can be used to fabricate a new family of oxide materials including those with special physical properties.

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