A STUDY OF THE FIBRE/MATRIX INTERACE IN AN OXIDE-FIBRE/NICKEL-ALUMINIDE-MATRIX COMPOSITES

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SUMMARY: The paper describes microstructure of the fibre/matrix interface in oxide-fibre/Ni_3Al-based-matrix composites obtained by pressure casting. Fibres of single-crystal alumina, alumina-YAG, and alumina-zirconia melt grown eutectics were used. Fibre/matrix interaction takes place during casting process in all the composites that does not normally produced the interface microstructure of a type that would yield sufficiently strong fibre/matrix bonding. Only YAG-containing fibres seem to be surrounded by a new formed phase that is bonded to both the fibre and matrix. Also the interface strength can be certainly improved due to a fibre surface roughness arisen because of different dissolution of different phases in eutectic fibres.

KEYWORDS: sapphire fibres, eutectic fibres, oxide/nickel interface, Ni_3Al alloy

INTRODUCTION

For oxide-fibre/nickel-based matrix composites the most important structural feature is a microstructure of the fibre/matrix interface [1]. Since the interface strength determines an effective fibre strength that, in turn, determines the composite strength studying its microstructure to find ways to control it and, hence, to control composite strength is an important task. This problem has been studying from the very beginning of metal matrix composites [2]. During the last decade, nickel-aluminide-based alloys have emerged as potential matrix materials. A number of publications has addressed the interface problem mainly in composites produced by using powder metallurgy routes at relatively low temperatures (see e.g. Refs. [3,' 4]). It was found, rather unexpectedly, intensive fibre/matrix interactions. At the same time, the interaction of single crystalline alumina fibres with NiAl melt was not observed at all [5]. Hence, a problem of the interface between the melt grown oxide fibres and a variety of nickel-based alloys remain to be of great importance. In the

present paper, oxide fibres produced by crystallizing the melts were introduced in a Ni_3Al-based alloy by using a liquid-phase route and the interface obtained was studied.

EXPERIMENTAL

Fibres of 50-80 μm in a cross-sectioned size were produced by internal crystallization method (ICM) [6]. Three compositions of the fibre melt were used: (1) pure (99.9%) alumina (sapphire fibres), (2) *Al_2O_3 -34wt. % Y_2O_3* (alumina -YAG eutectic fibres) and (3) *Al_2O_3 -41.2wt. % ZrO_2 - 1.8wt. % Y_2O_3* (alumina – zirconia eutectic fibres). The matrix material called VKVA-4u that is *Ni_3Al* alloying with of *Co*, *Ni*, *W*, *Mo*, *Ti* and *Cr* [*7*] was melted in vacuum and then pressure infiltration technique was used to fabricate composites. To look at wetting behavior of the ICM fibres in the matrix melt, experiments with immersion of fibre bundles into the melt at T=1480^{o}C were also conducted.
Composite microstructure was investigated on polished sections of the specimens by SEM (Jeol-25s) and TEM (Jeol-100, thin foil technique). Chemical composition of the fibre/matrix interface was studied by EDS (Jeol-2000FX equipped with EDS Link AN10000). The surface of the fibres chemically etched from composites was investigated by SEM.

RESULTS

An interaction at the fibre/matrix interface depends clearly on the fibre material.

Sapphire fibres

The only feature of the surface of an fibre of extracted from the matrix is a relief typical for ICM fibres (Fig.1) which reflects the surface topography of the molybdenum carcass used for fibre crystallization.

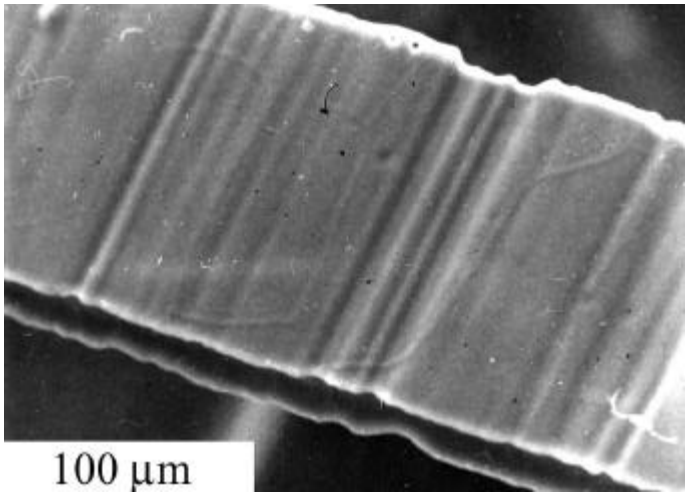

Fig.1. ICM sapphire fibre extracted from composite, SEM

No traces of an interaction at the interface except possibly some dissolution of alumina in the matrix melt. Even at exposure for 6 hours under a pressure of 25 atm, neither new phase on the interface nor significant dissolution of the fibre sharp edges was observed. However, in this case there occurred more uniform distribution of fibres in the matrix than after shorter

exposures and lower pressure. Perhaps this relates to a decrease in the wetting angle between sapphire and matrix melt.

Alumina-YAG eutectic fibres

Moving a bundle of alumina-YAG eutectic fibres into the melt does not yield penetrating the melt into the bundle. Hence, the contact angle between the fibre and the melt is high initially. After exposure in the melt, the bundle was withdrawn at a rate of 1 mm/min, and the height of a "hillock" that arose on the melt surface surrounding the withdrawing bundle was estimated. The exposure less than 120 min yielded the "hillock" height of 0.5-0.8 mm, the exposure of 180 min yielded the height up to 4 mm. Therefore at long time exposures there was interactions between the fibre surface and the melt that yield a decrease in the contact angle.

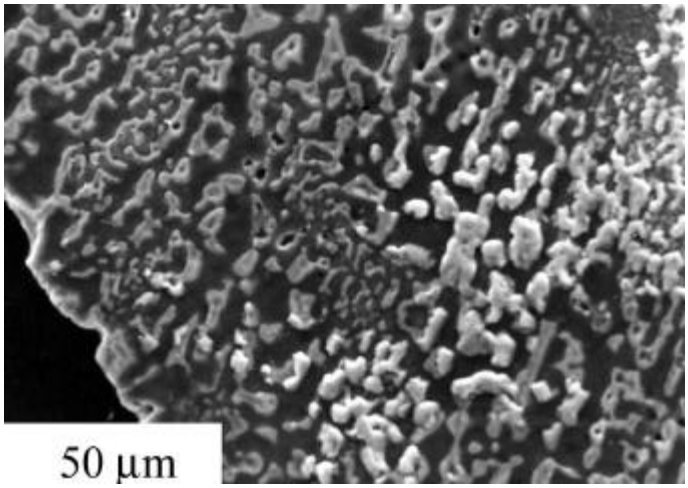

Fig. 2. Surface of ICM alumina-YAG fibre extracted from melt, SEM

A larger dissolution of alumina in the melt than that of YAG makes the fibres surface to be rough as shown in Fig. 2. Observation of a cross-section of a composite tested in tension by SEM, Fig. 3, reveals delamination at the fibre/matrix interface. It should be noted that delamination cracks are arrested by interface regions where a white phase appears.

Alumina-zirconia eutectic fibres

Because of different dissolution of the eutectic fibre phases in the melt the surface of the Al_2O_3 - ZrO_2 fibres extracted from melt is similar to that of alumina-YAG fibres.

A zone of a new phase is observed around each fibre in the composite (Fig. 4). TEM investigations reveals a chain of the microcracks between the fibre and the zone mentioned (Fig. 5).

Sometimes similar microcrack chains occur between the zone and the matrix.
Electron diffraction shows a single crystal structure of the zone similar to that of the matrix which is mainly mixture of γ and γ' phases. These phases are of similar crystallographic structure, which makes difficult their indentification by electron diffraction technique. EDS shows an increase in *Zr* and *Al* content in the zone as compared to the matrix composition, that

can be explained by fibre dissolution. The microstructure of the interface will be described in more details elsewhere [8].

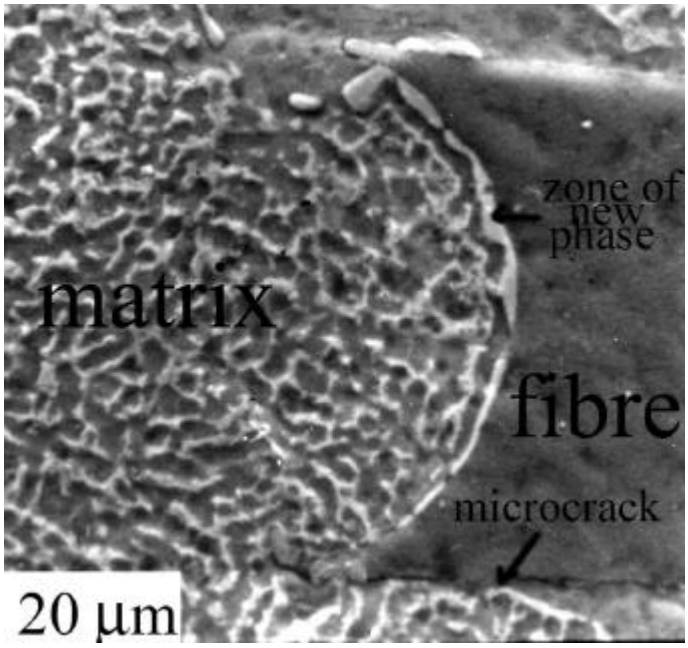

Fig. 3. Alumina-YAG fibre in Ni_3Al matrix after tension test, SEM

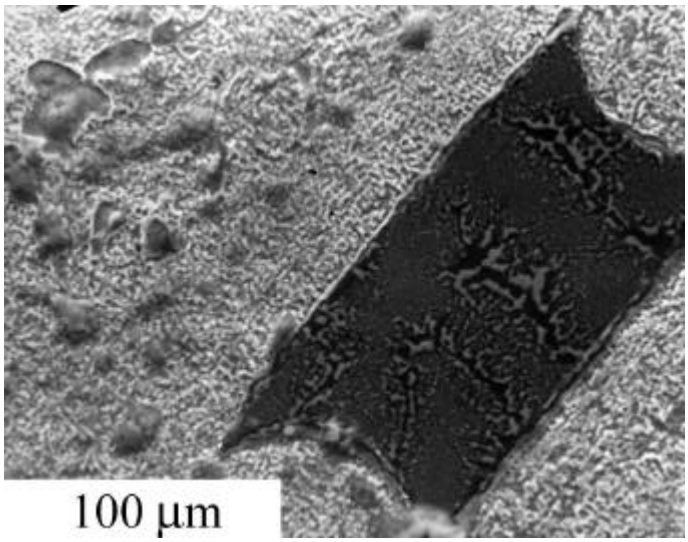

Fig. 4. Alumina-zirconia ICM fibre in *Ni_3Al* matrix, SEM

DISCUSSION

An important feature of the fibre/matrix interaction, which is dissolution of alumina, was earlier observed [9] in composites with sapphire fibres and *Ni_3Al* matrix with chromium additions (0.5-1 at. %). It is likely that the presence a large amount of *Cr* leads to fibre dissolution in the composites under consideration. However, unlike to earlier observations [9], in the present sapphire-fibre composites neither new phases nor strong bond at the fibre/matrix interface was revealed. Perhaps, the presence of 0.026 wt % of carbon prevent the formation of a new oxide phase at the interface. The second reason for the absence of an interface change in composite

the under consideration is a larger chromium content that can ease the phase formation in the matrix volume than at the interface. Similarly, Asthana et. al. saw [10] the formation of *NiAl-Cr* eutectic near the interface in sapphire/*NiAl* composite with 11% *Cr* content in the matrix. However, they did not see any oxide phases at the interface even at free oxygen content in the matrix about 300 ppm.

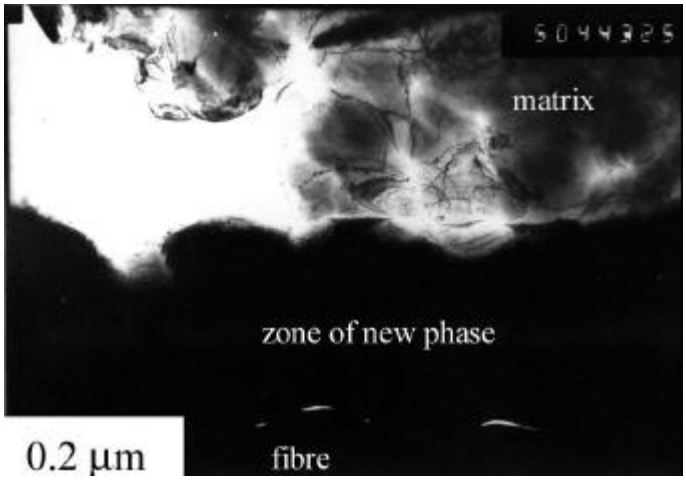

Fig. 5. TEM image of the interface in Al_2O_3 - ZrO_2/ Ni_3Al composite

It was shown [11] that only a little interaction takes place at the interface after pressure infiltration of polycrystalline Al_2O_3-ZrO_2 fibres with Ni_3Al matrix containing 7.9 at.% of *Cr*. Vacuum annealing at 1100°C resulted in the precipitation of Cr_7C_3. Annealing in air at 1100°C resulted in formation of $NiAl_2O_4$ spinel at the fibre/matrix interface. But $Ni(Al_xCr_{1-x})_2O_4$ spinel inclusions were observed only in matrix volume. The present results reveal formation of a new phase at fibre/matrix interface during pressure casting in argon atmosphere but it does not improve the interface strength.

CONCLUSIONS

1. A liquid phase route of composite fabrication results in an chemical interaction between ICM alumina-based oxide fibres and Ni_3Al-based matrix. The process is certainly triggered by dissolution of Al_2O_3 in a *Ni*-based solution.
2. The most important result of this interaction is formation of a zone around the fibre surface that has a chemical composition different from that of the matrix. The thickness and chemical composition of the zone are strongly depended on a fibre type.
3. As a rule, the fibres are weakly bonded to the matrix. Only in the case of alumina-YAG fibres the presence of a phase well bonded to both the fibre and matrix is observed at the interface.
4. In the case of eutectic fibres, such as Al_2O_3-$Y_3Al_5O_{12}$ and Al_2O_3-$ZrO_2(Y_2O_3)$, nongomogeneous dissolution of the fibre material make initially smooth fibre surface to get roughness.

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