Scripta Materialia 145 (2018) 23–27

Contents lists available at ScienceDirect

Scripta Materialia

journal homepage: www.elsevier.com/locate/scriptamat

Regular article

Crystallography and interfacial structure in a directionally solidified $Al_2O_3/Y_3Al_5O_{12}/ZrO_2$ eutectic crystal

Xu Wang [a,*], Yujie Zhong [b,*], Qian Sun [c], Yiran Li [d], Wen Zhang [a], Dongqing Qi [d], Dong Wang [e], Bailing Jiang [a]

[a] School of Materials Science and Engineering, Xi'an University of Technology, 5 South Jinhua Road, Xi'an 710048, China
[b] School of Materials Science and Engineering, Xi'an Shiyou University, Xi'an 710065, China
[c] School of Materials Science and Engineering, University of Science and Technology of China, 72 Wenhua Road, Shenyang 110016, China
[d] Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, 72 Wenhua Road, Shenyang 110016, China
[e] Superalloys Division, Institute of Metal Research, Chinese Academy of Sciences, 72 Wenhua Road, Shenyang 110016, China

ARTICLE INFO

Article history:
Received 5 August 2017
Accepted 7 October 2017
Available online xxxx

Keywords:
$Al_2O_3/Y_3Al_5O_{12}/ZrO_2$ ternary eutectic crystal
Directional solidification
Interface structure
EBSD
HRTEM

ABSTRACT

The single crystal of $Al_2O_3/Y_3Al_5O_{12}/ZrO_2$ ternary eutectic was prepared by an optical floating zone furnace. The crystallography and interfacial structure of the directionally solidified ternary eutectic were investigated by means of electron backscattered diffraction and transmission electron microscopy. We found that the preferred crystallographic orientation of the $Al_2O_3/Y_3Al_5O_{12}/ZrO_2$ eutectic crystal was $\{11\bar{2}0\}\langle 1\bar{1}00\rangle$ $Al_2O_3||\{001\}\langle 001\rangle$ $Y_3Al_5O_{12}||\{001\}\langle 001\rangle$ ZrO_2. Most of the tiny ZrO_2 locates dispersively between Al_2O_3 and $Y_3Al_5O_{12}$. It can be concluded that interfacial energy between Al_2O_3-$Y_3Al_5O_{12}$-ZrO_2 played an important role in deciding the preferred crystallographic orientations of the $Al_2O_3/Y_3Al_5O_{12}/ZrO_2$ eutectic and the precipitation behavior of the ZrO_2 phase.

The directionally solidified eutectics, such as $Al_2O_3/GdAlO_3$, $Al_2O_3/Y_3Al_5O_{12}$ (YAG), and $Al_2O_3/YAG/ZrO_2$ have received considerable attentions because of its good oxidation resistance, microstructure stability, creep resistance and outstanding high-temperature (close to the melting point) mechanical properties. The eutectic ceramics, thus, are supposed as one of the most promising structural materials in a new generation gas turbines operating at 1923 K [1–5].

The directionally solidified $Al_2O_3/YAG/ZrO_2$ ternary eutectic exhibits a flexural strength of ~860 MPa, which is about 57 times higher than that of the sintered ternary composite with the same composition (~15 MPa) and is higher than the value of the *a*-axis sapphire (~450 MPa) [6]. Furthermore, previous studies have proved that the directionally solidified $Al_2O_3/YAG/ZrO_2$ ternary eutectic presents superior performance to the directionally solidified Al_2O_3/YAG binary eutectic grown at the same rate because of its smaller domain size [7–9]. For instance, the average flexural strength of the directionally solidified $Al_2O_3/YAG/ZrO_2$ ternary eutectic at 1873 K is approximately 800 MPa, more than twice the value of the directionally solidified Al_2O_3/YAG binary eutectic (350 MPa) [6,10]. The room-temperature fracture toughness of directionally solidified $Al_2O_3/YAG/ZrO_2$ ternary eutectic is 4–5 $MPa\cdot m^{1/2}$ [8,11], twice that of the Al_2O_3/YAG binary eutectic [12–15]. Up to now, several directional solidification techniques, such as laser floating zone technique [16,17], micro-pulling-down technique [18], edge-defined film-growth technique [19,20] and Bridgman technique [1,21] have been applied to prepare the $Al_2O_3/YAG/ZrO_2$ eutectic. Nevertheless, the Achilles heel for structural applications of the directionally solidified $Al_2O_3/YAG/ZrO_2$ ternary eutectic is their low toughness, which makes them prone to catastrophic failure. As is known, the mechanical performances of the directionally solidified $Al_2O_3/YAG/ZrO_2$ eutectic depend on its microstructure, especially the preferred orientations and interfacial structure. In addition, ZrO_2 phase acts usually as the toughening element in the directionally solidified ternary eutectic. The morphology, size, distribution of ZrO_2, and especially the orientation relationships between ZrO_2 and the matrix will undoubtedly affect the fracture toughness and strength of the directionally solidified $Al_2O_3/YAG/ZrO_2$ eutectic ceramic. A better study of the precipitation behavior and characteristics mentioned above of ZrO_2 is scientifically and technologically meaningful.

In the present paper, the preferred growth directions and interfacial structure of the $Al_2O_3/YAG/ZrO_2$ eutectic was studied by electron backscattered diffraction (EBSD) and high-resolution transmission electron microscopy (HRTEM). The results can help us to understand the mechanism of ternary eutectic growth in directional solidification of the $Al_2O_3/YAG/ZrO_2$ eutectic and pave way for the design of high performance eutectic ceramics.

Commercially available Al_2O_3, Y_2O_3 and ZrO_2 powders were mixed and ball milled for 12 h at a mole ratio of Al_2O_3:Y_2O_3:ZrO_2 = 65:16:19 according to the ternary eutectic point in the ternary phase diagram [22,23]. Precursors were prepared with a pressure of 40 MPa for 5 min and cold

* Corresponding authors.
E-mail addresses: xwang@alum.imr.ac.cn (X. Wang), yjzhong11s@alum.imr.ac.cn (Y. Zhong).

https://doi.org/10.1016/j.scriptamat.2017.10.008
1359-6462/

isostatic pressure at 280 MPa for 30 min. Next, the precursors, which were used as the feed bars, were sintered at 1823 K for 2 h. The directional solidification was performed using an optical floating zone furnace with four 3-kW xenon arc lamps as radiation sources in an argon atmosphere with slight overpressure (~1.2 bars). Neither the seed rod nor the source rod was rotated. The withdrawal rate was 10 mm/h. Crystals were prepared with up to 10 mm in diameter and ~130 mm in length.

The as-grown Al_2O_3/YAG/ZrO_2 eutectic crystal was sectioned by a fixed diamond endless wire saw. All of the samples were carefully ground with SiC paper till 2000# and further polished down to 2.5 μm diamond paste. Thin-foil specimens for transmission electron microscopy (TEM) investigation were prepared by slicing, mechanical grinding to ~30 μm, and ion beam milling at 5.0 kV.

The microstructure was characterized with scanning electron microscopy (SEM, LEO, SUPRA 35, Ammerbuch, Germany). The crystal orientation was investigated by means of electron-backscattered diffraction (EBSD, NordlysNano, Oxfordshire, UK). A 300 kV TEM (FEI Tecnai G^2 F30, Oregon, USA) was used for structural investigation of the interfaces and the HRTEM observation. Fast Fourier transformation (FFT) was carried out using the Digital Micrograph package (Gatan, California, USA).

Fig. 1 shows the microstructure of the as-grown directionally solidified Al_2O_3/YAG/ZrO_2 eutectic at the distance of ~100 mm from the seed. Shown are Al_2O_3 in black, the YAG in grey and ZrO_2 in white. Colonies can be observed in the Al_2O_3/YAG/ZrO_2 eutectic. Longitudinal image (Fig. 1a) shows that the colonies exhibit along the growth directions while the transverse image (Fig. 1c) shows that the colonies present irregular non-circularity shapes, which confirms the previous study of Benamara [18]. As can be observed from Fig. 1b and d, ZrO_2 appears more dispersed and smaller than the other two phases, and most of them locate between Al_2O_3 and the YAG.

Fig. 2 shows the typical transverse EBSD orientation maps of the Al_2O_3/YAG/ZrO_2 eutectic at the distance of ~100 mm from the seed. Fig. 2a shows the EBSD band-index maps of the Al_2O_3/YAG/ZrO_2 eutectic crystal. The continuous black dotted line is the colony boundary. It can be observed that the crystallographic orientation relationship in and out of the colony boundary is invariable. Patterns in Fig. 2b, the YAG, show the growth direction of ⟨001⟩, patterns in Fig. 2c, the Al_2O_3, show the growth direction of ⟨10$\bar{1}$0⟩ while patterns in Fig. 2d, the ZrO_2, show the growth direction of ⟨001⟩.

Fig. 3 shows the corresponding pole figures of Fig. 2. The orientation relationship of the Al_2O_3/YAG/ZrO_2 eutectic is {11$\bar{2}$0}Al_2O_3||{001}YAG||{001}ZrO_2, as marked by the red circles in Fig. 3a, b and c. It can be naturally obtained that planes {0001}Al_2O_3, {001}ZrO_2 and {001}YAG are in parallel (the black circles in Fig. 3a, b and c). Notably, the deviation of the Al_2O_3/YAG/ZrO_2 eutectic is about 5° from the normal direction (the white circles). The crystallographic orientation relationships of the three phases are as follows:

⟨1$\bar{1}$00⟩ Al_2O_3|| ⟨001⟩ YAG|| ⟨001⟩ ZrO_2,

{11$\bar{2}$0}Al_2O_3||{001}YAG||{001}ZrO_2.

To further study the interfacial structure of the Al_2O_3/YAG/ZrO_2 eutectic, thin-foil specimens were prepared for HRTEM investigation. Fig. 4 shows the interfacial structure at the triple-junction between Al_2O_3-YAG-ZrO_2. The interfacial structure between Al_2O_3 and the YAG, Al_2O_3 and ZrO_2, the YAG and ZrO_2 can also be observed. As can be observed from the FFT patterns (Fig. 4b), the interfacial orientation relationships of the directionally solidified Al_2O_3/YAG/ZrO_2 eutectic are:

[1$\bar{1}$00]Al_2O_3||[100]YAG||[100]ZrO_2,

(11$\bar{2}$0)Al_2O_3||(004)YAG||(002)ZrO_2.

These results agree well with that obtained by the EBSD, and further perfect the previous results reported by Mazerolles [24]. According to Bonnet and Cousuneau [25], this kind of interfacial structures has minimum interfacial strains and neutral ionic charges. The net result (11$\bar{2}$0)[1$\bar{1}$00]Al_2O_3||(004)[100]YAG||(002)[100]ZrO_2 should be the most favorable crystallography orientation relationships.

Because the lattice parameters of Al_2O_3 (a = b = 0.475897 nm and c = 1.299649 nm), YAG (a = b = c = 1.20062 nm) and ZrO_2 (a = b = c = 0.5135 nm) are remarkably different from each other, the lattice disregistries of the Al_2O_3/YAG, Al_2O_3/ZrO_2, and

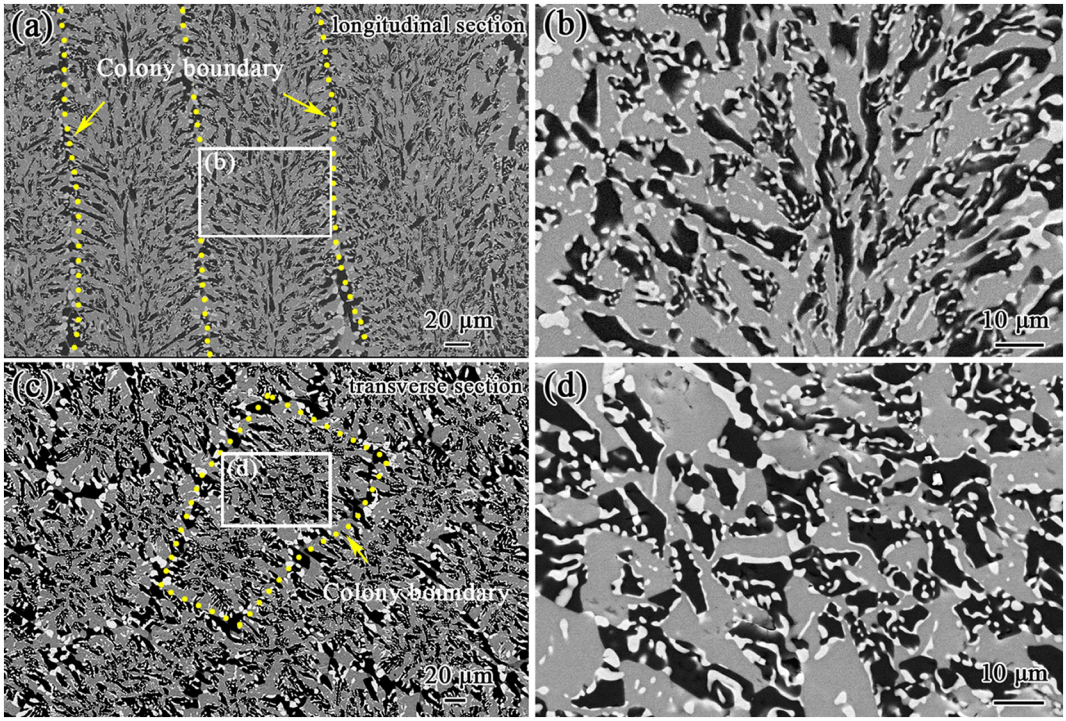

Fig. 1. SEM images showing longitudinal microstructure (a) and transverse microstructure (c) of the directionally solidified Al_2O_3/YAG/ZrO_2 eutectic crystal; (b) and (d) the magnified microstructure of the areas marked in (a) and (c). Yellow dotted curves in (a) and (c) are colony boundaries in longitudinal section and transverse section of the directionally solidified Al_2O_3/YAG/ZrO_2 eutectic. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)