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Effect of solidification path on the microstructure of Al_2O_3–Y_2O_3–ZrO_2 ternary oxide eutectic ceramic system

Haijun Su*, Jun Zhang, Haifeng Wang, Kan Song, Lin Liu, Hengzhi Fu

State Key Laboratory of Solidification Processing, Northwestern Polytechnical University, Xi'an 710072, PR China

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Abstract

The solidification path of the Al_2O_3–Y_2O_3–ZrO_2 ternary oxide eutectic composite ceramic is determined by a high temperature DTA and laser floating zone (LFZ) directional solidification method to investigate the effect of solidification path on the microstructure of the ternary oxide. The DTA and microstructure analyses show that the YAG or Al_2O_3 tends to form as primary phase under the unconstrained solidification conditions, and then the system enters ternary eutectic solidification during cooling from 1950 °C at rate of 20 °C/min. The as-solidified composite ceramic shows a divorced irregular eutectic structure consisting of Al_2O_3, YAG and ZrO_2 phases with a random distribution. The primary phases are however completely restrained at the directional solidification conditions with high temperature gradient, and the ternary composite by LFZ presents well coupled eutectic growth with ultra-fine microstructure and directional array. Furthermore, the eutectic transformation and growth mechanism of the composite ceramic under different solidification conditions are discussed.

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Keywords: Eutectic ceramic; Solidification; Microstructure; Al_2O_3–Y_2O_3–ZrO_2; DTA; Oxide composite

1. Introduction

In modern industry and high-technique field, there is a great and increasing need of advanced materials with excellent strength combined high toughness and oxidation resistance at elevated temperature above 1500 °C.[1–3] At this point, the conventional monolithic materials generally can not meet the rigorous requirements, and thus has gradually been displaced by the multicomponent and multiphase materials having complex microstructure characteristic.[4–6] Moreover, the properties of multiphase composites are primarily determined by the microstructure produced during solidification and subsequent processing stages.[7] In order to further design materials with optimized properties, hence it is highly necessary to obtain a detailed understanding of microstructural formation and reliable database during solidification process. Among them, the solidification path plays an important and vital role in determining microstructure characteristic.[8–10] In the past few years, the fundamental knowledge on solidification are mainly focused on pure metallic materials and binary metallic alloys exhibiting single-phase or two-phase growth, the solidification behavior and processing are less well understood for cases where multiphase reactions occur along the solidification path, especially for oxide ceramic composites.[11–13]

Recently, the solidification processing of oxide ceramics has attracted great interests in the development of high-performance materials with excellent high-temperature strength at above 1650 °C in an oxidized environment in order to improve the heat efficiency of gas turbine or aerospace engine.[14–18] The melt growth of ceramic materials by directional solidification allows producing *in situ* binary or multiphase composites with fine microstructure and strongly bonded phases, leading to keep a higher strength almost constant up to temperatures close to melting point, good creep resistance, and excellent stability of high-temperature microstructure in comparison with the sintered ceramic. Consequently, directionally solidified (DS) oxide eutectic ceramic *in situ* composites have recently been paid much more attention as potential candidates for high temperature hot-section components used in advanced engines and gas turbines.[19,20] One of the most interesting and promising ceramic systems is the ternary eutectic ceramic from Al_2O_3–Y_2O_3–ZrO_2 system owing to its excellent properties. Fox example, the

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* Corresponding author. Tel.: +86 29 88492228; fax: +86 29 88494080.
E-mail address: shjnpu@yahoo.com.cn (H. Su).

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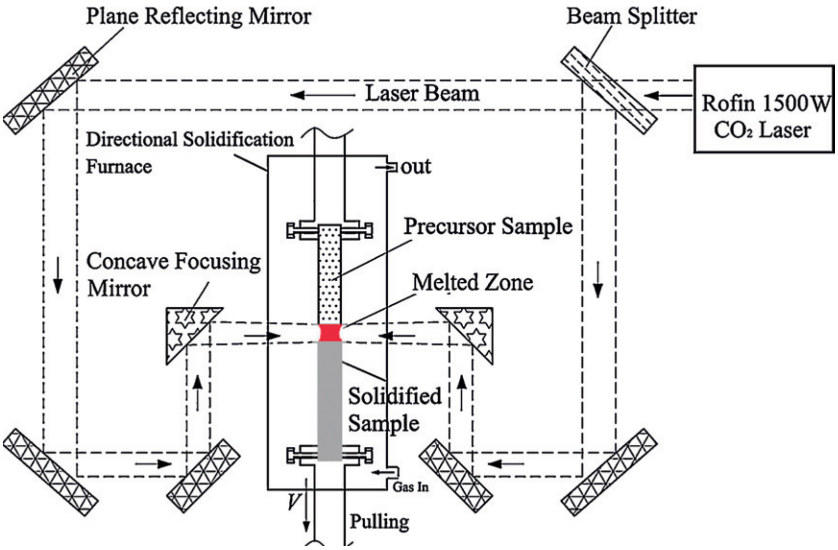

Fig. 1. Schematic representation of the LFZ set-up.

flexural strength of DS Al_2O_3/YAG/ZrO_2 eutectic ceramic has reached 4.6 GPa.[14,17,20] It is known that understanding the solidification path is important to control microstructures and further improve properties for multi-phase composites. Recently, we have investigated the thermal behavior of the laser re-melted ternary eutectic composite,[21] but lacked more information obtained from the mixed ternary oxide eutectic powders. Because of the complexity of the multi-component and multiphase system and very high melting points, the solidification behavior on the ternary oxide eutectic ceramic system is still unclear up to date yet. Herein, a high temperature DTA with superheating treatment and the laser floating zone melting method with high temperature gradient are applied to investigate the effect of the solidification path on microstructure of the ternary oxide system at unconstrained and directional solidification conditions, respectively. The relationship between the ternary eutectic selection and different microstructure characteristics is discussed.

2. Experimental

Starting materials are prepared from high purity (>4 N) nano-powders of Al_2O_3, Y_2O_3 and ZrO_2 by wet mixing homogenously according to ternary eutectic mole composition ratio of $Al_2O_3/Y_2O_3/ZrO_2 = 65/16/19$ from the phase diagram.[22] The $\Phi 4\,\text{mm} \times 50\,\text{mm}$ rods are prepared from the mixed powder by die pressing and pressureless sintering at 1500 °C for 2 h to act as the precursors for the laser floating zone (LFZ) melting experiment. The set-up of LFZ equipment is newly established by State Key Laboratory of Solidification Processing, China, as shown in Fig. 1. It consists of a 1500 W CO_2 laser, a light conduction system and a directional solidification furnace with strong cooling system. The laser is uniformly divided into two beams by the beam splitter and then focused from opposite directions to remelt a small zone of the precursor. At the same time, the sample is pulled down to achieve continuous and directional growth of the composite.

The mixed powders with approximate 50 mg are heated and remelted by a DTA apparatus (DTA, Netzsch-409 CD) in a cone-shaped W crucible at a heating rate of 20 °C/min. The maximal heating temperature is 1950 °C and the melt is held for 15–30 min to reach a superheating treatment. Then the remelted samples are cooled to room temperature at a rate of 20 °C/min to examine the melting and solidification behavior at unrestrained condition. As a comparison, the sintered rod precursor is directionally solidified from melt by LFZ method at the solidification rate of 400 μm/s to investigate the solidification behavior at directional solidification condition with high cooling rate.

The phase component and crystal structure of the specimens obtained by DTA and LFZ are determined using X-ray diffraction (XRD, Rigakumsg-158, Tokyo, Japan) and energy disperse spectroscopy (EDS, Link-Isis, Oxford, England). The microstructures are observed by scanning electron microscopy (SEM, JSM-5800, Tokyo, Japan).

3. Results and discussion

Fig. 2(a) shows the macroscopic photograph of the as-solidified ternary composite remelted by DTA. The plate sample has the diameter of 3 mm and the thickness of 2 mm, and presents smooth surface. The result indicates that there is almost no chemical reaction between the sample and Mo crucible. The inset is the magnification of the sample surface. It can be seen that it is very dense in the sample center, but there are pores or cracks only found at the outer edge along diameter, which is similar to the result of laser re-melted oxide eutectic ceramic.[6] The formation of pore mainly results from the high melt viscosity and survival O_2 in the mixed powder, which leads to the gas having no time to escape to reach the melt surface, remaining the trapped porosity in the solidified phase.[23] Furthermore, the cooling rate is more rapid at the outer edge than inside, easily inducing the produce of cracks. By contrast, the directionally solidified rod by LFZ is about 3 mm in diameter and 30 mm in length, and shows uniform

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