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Materials Science and Engineering A 479 (2008) 380–388

MATERIALS SCIENCE & ENGINEERING A

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Rapid solidification behaviour of $Al_2O_3/Y_3Al_5O_{12}$ (YAG) binary eutectic ceramic *in situ* composites

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Received 10 January 2007; received in revised form 21 June 2007; accepted 22 June 2007

Abstract

Rapid solidification of $Al_2O_3/Y_3Al_5O_{12}$ (YAG) binary eutectic ceramic *in situ* composites during the remelting process using high-energy laser, which results in refinement of the solidification microstructure and improvement of the mechanical properties, is studied in this paper. The rapidly solidified Al_2O_3/YAG eutectic consists of Al_2O_3 and YAG phases without any other phases, effectively avoiding grain boundaries. The eutectic interspacing is extremely refined due to the high-temperature gradient and solidification rate of laser rapid solidification, and the minimal interspacing is as fine as 0.5 μm. The eutectic exhibits an obvious faceted–faceted eutectic growth characteristic which mainly derives from the high-fusion entropies of eutectic phases and large kinetic undercooling. The mean hardness and fracture toughness measured by an indentation technique are 17.5 GPa and 3.6 MPa $m^{1/2}$, respectively. The increase of fracture toughness can be attributed to the refinement of eutectic phases which restrain and arrest crack propagations.

Keywords: $Al_2O_3/Y_3Al_5O_{12}$ (YAG); Eutectic ceramic *in situ* composite; Laser rapid solidification; High-temperature gradient; Fracture toughness

1. Introduction

With the rapid development of the advanced aerospace technology, performances of materials at ultra-high temperatures have increasingly become the key factors strongly affecting the development of the space shuttle, spacecraft, satellite and future aero-space plane. The turbine inlet temperature for thrust-weight ratio above 10 is beyond 2000 K, such as the fourth generation fanjet used in F119 [1]. Meanwhile, future aerospace propulsion system will require materials that are lighter, stiffer, and stronger at higher temperature than currently available materials. Moreover, at present, the earth environment and energy-saving are the most severe problems that the mankind has to face, and have become international problems all over the world. Research results show that a 1% improvement of thermal efficiency would lead to an annual energy cost-saving of around $1000 billion for the entire world [2]. In order to achieve the above-mentioned points, there is a strong demand to develop a new ultra-high temperature structure material that can further improve the thermal efficiency and curb the emission of pollutants such as CO_2 and NO_x in aircraft engines and other high-efficiency gas turbines.

In the past century, oxide ceramics and oxide ceramic–matrix composites for promising structure materials have been taken into account, because of their excellent heat resistance, corrosion resistance, abrasion resistance and low density. However, oxide ceramics generally cannot be applied as high-temperature structure materials above 1300 K for their sensitivity to plastic deformation at elevated temperatures [3]. Whereas they have far superior oxidation resistance properties to ordinary ceramics, e.g., SiC and Si_3N_4, and if their mechanical properties, especially the room-temperature ductility could be evidently improved, the oxide ceramics are very hopeful to become the thermo-structural materials with outstanding mechanical properties retention at high temperatures even close to their melting points at the high-temperature oxidizing atmosphere over a long period of time [4].

In general, most conventional oxide ceramics are dominatedly fabricated by the powder sintering method. However, in many cases, impurities and amorphous phases often exist at the grain boundaries, which extremely deteriorate the high-temperature strength and creep resistance. Directional solidification technique is proven to be an advanced method for successfully preparing high-temperature superalloys, single

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doi:10.1016/j.msea.2007.06.080

crystals with outstanding performances by eliminating grain boundaries, and accordingly improving high-temperature performances retainment of materials. In this context, one potential approach to improve the mechanical properties of oxide ceramic composites is to fabricate the ceramic composites by directional solidification from eutectic melts [5–7]. In 1997, Waku et al. [8] reported the excellent mechanical properties of directionally solidified Al_2O_3/YAG and $Al_2O_3/GdAlO_3$ eutectic ceramics at high temperatures. For example, the flexural strength can be maintained at 360–500 MPa from room temperature almost up to the melting point (about 2100 K) and the compression creep strength at 1873 K is about 13 times higher than that of sintered composites with the same chemical composition [8–10]. Therefore, in the subsequent decade, directionally solidified Al_2O_3-based eutectic ceramics and their preparation techniques have always been paid much more attention to because of their micro-structural stability, low porosity, clean interface and high thermodynamical-compatibility between two strongly bonded phases with typical interspacing in the micron range for ultra-high temperature structural use [11–15].

However, most of Al_2O_3-based eutectic ceramics have extremely high-melting points (above 2000 K), which leads to the limitations in preparation, high-cost and low-efficiency using conventional directional solidification methods and equipments [8–10]. Recently, laser zone remelting technique has been successfully used to directionally grow oxide eutectic ceramics with advantages of very high-melting temperature, absence of any container or die avoiding possible sources of contamination, steep-temperature gradients, fast growing rates and low cost [11,12]. Larrea et al. [11] reported the preparation of $Al_2O_3–ZrO_2$ eutectic plates by laser zone surface remelting. Sayir and co-workers [12] utilized the laser heated float zone method to obtain fibers of $Al_2O_3/Er_3Al_5O_{12}$ eutectics. Whereas the fabrication of Al_2O_3/YAG eutectic ceramic rods with laser zone surface remelting is not available to date. In addition, due to difficulties in processing, limited experiment dates and complex physical properties for oxide eutectics, few studies of the solidification behaviour of the binary eutectics under rapid solidification conditions have been carried out. This paper aims to present the preparation of Al_2O_3/YAG eutectic ceramics *in situ* composites with the laser zone remelting technique, and mainly to study the rapid solidification behaviour and growth mechanism of Al_2O_3/YAG binary eutectic ceramic under laser rapid solicitation conditions. Additionally, the hardness and room-temperature fracture toughness of the as-solidified composites are preliminarily investigated by a micro-indentation method.

2. Experimental

The starting materials were prepared by mixing the high purity (>4N) nano-powders of Al_2O_3 and Y_2O_3 corresponding to the eutectic composition in the phase diagram of the $Al_2O_3–Y_2O_3$ system [16] using ball milling to obtain a homogeneous mixture. The mole fraction of the eutectic composition was $Al_2O_3/Y_2O_3 = 82/18$. Then the precursor rods of ø7 mm × 60 mm samples were prepared by die pressing uniaxially for 10 min at 25 MPa, followed by sintering at 1773 K for 2 h to increase density and handing strength. The laser zone remelting experiment was conducted in a vacuum chamber using a 5 kW ROFIN-SINAR850 CO_2 laser. The sample was moved by the numerically controlled worktable with five-axis and four-direction coupled motion to realize the laser beam scanning along the sample axis. The sample was solidified just behind the melting zone, as illustrated in Fig. 1. The experiments showed that with the laser power of 190–220 W, the scanning rate of 60–2000 μm/s and the beam diameter of 4 mm under the Ar atmosphere, the crackless samples with smooth surfaces and high densities could be obtained. The solidified samples were cut from the rods and polished sequentially with diamond paste down to 0.5 μm. The surfaces were coated with a thin layer of Au before observation. The microstructure and component of the composite were determined by scanning electron microscopy (SEM, JSM-5800), energy disperse spectroscopy (EDS, Link-Isis) and X-ray diffraction (XRD, Rigakumsg-158) techniques. Quantitative calculation of the phase volume fraction was performed by digital image analysis software of SISC IAS V8.0. The hardness and fracture toughness were determined by a Vickers indentation technique on the polished surface of the composite, using applied load of 9.8 N for 15 s. The indentation size and the crack length were measured by optical microscope.

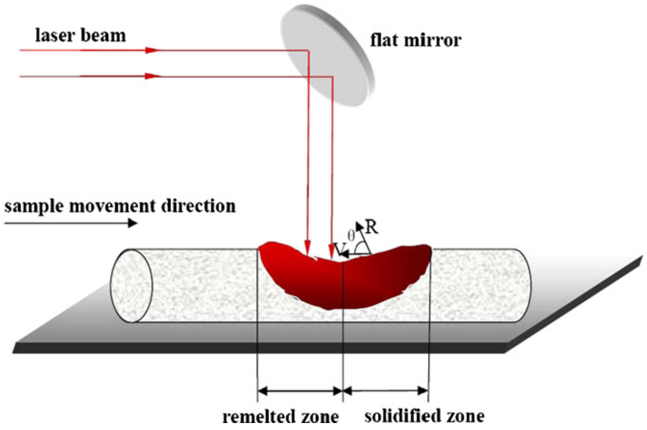

Fig. 1. Experiment setup for the laser zone remelting method applied to the growth of the rapidly solidified eutectic rods, V is the scanning rate, R the solidification rate, and θ is the solidification direction angle.

3. Results and discussion

3.1. Rapid solidification microstructure

The rapidly solidified rods of Al_2O_3/YAG eutectics are about 6 mm in diameter with a slick surface free of porosity and crack. When the scanning rate is high, the solidified rod is opalescent but is glazed and shiny when the scanning rate is low. The macroscopical pictures of the longitudinal and cross-section of the as-solidified original eutectic rod sample are shown in Fig. 2a and b, respectively. During the laser zone remelting, there is always a lamina precursor about 0.1–0.2 mm thick hardly to completely remelted at the bottom of the rapidly solidified eutectic rod. In order to obtain the fully remelted and solidified eutectic sample, the as-solidified near-circular rod sample is pro-