

## Effects of laser processing parameters on solidification microstructures of ternary $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$ eutectic in situ composite and its thermal property

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Received 10 August 2009; accepted 15 September 2009

**Abstract:** Rapid surface resolidification with a high powered  $\text{CO}_2$ -laser was performed in preparing directionally solidified  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  ternary eutectic ceramic in situ composite. The effects of laser processing parameters on the solidification microstructure characteristics and thermal properties were studied by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), X-ray diffractometry (XRD) and synthetically thermal analysis (STA). Detailed investigations of the influence of laser power and scanning rate on the preparation and microstructural parameters of the ternary eutectic were presented. Moreover, the eutectic phase separation rule at high temperature was discussed. The results indicate that solidification microstructure of the ternary eutectic composite is greatly influenced by the laser processing parameters. The synthetically thermal analysis shows that the eutectic temperature of ternary  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  composite is 1 738 °C, well matching the phase diagram of  $\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3\text{-ZrO}_2$ .

**Key words:**  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$ ; eutectic in situ composite; ceramic; microstructure; laser resolidification

### 1 Introduction

In the past few decades, considerable efforts have been devoted to develop high-performance materials with excellent high-temperature strength, which can serve for a long time at above 1 650 °C in an oxidizing atmosphere, aiming to improve thermal efficiency of aircraft engines and gas turbines[1–3]. In comparison with conventional superalloys and current SiC/SiC and C/C composites, directionally solidified oxide eutectic ceramic in situ composite possesses higher melting point (above 1 700 °C), low density ( $<5 \text{ g/cm}^3$ ), superior high-temperature strength and microstructural stability (almost up to melting point), as well as excellent creep resistance and oxidation resistance. Therefore, it attracts great interest in the fabrication of new ceramic eutectic in situ composite by directional solidification[4–6]. Moreover, the mechanical properties of the oxide eutectics are superior to those of each constituent phase, compared with sintered ceramics, because of the clean

and strong interfaces provided by directional solidification[7].

Among a series of oxide eutectic ceramic systems, directionally solidified  $\text{Al}_2\text{O}_3/\text{YAG}$  is considered to be the most promising candidate as ultra-high-temperature structural material[4–9]. For instance, the flexural strength of the composite maintains at 360–500 MPa from room temperature to 1 800 °C in air[10]. Also, it possesses outstanding corrosion resistance at high temperatures[11]. However, the binary eutectic ceramic is inherently brittle at room temperature, which prevents its further application[12]. There has been an increasing effort to overcome its shortcoming, such as through microstructure refinement and incorporating third phase reinforcements[13–15]. The incorporation of the third ductile phase, such as  $\text{ZrO}_2$ , is a promising method to overcome the inherent deficiencies of  $\text{Al}_2\text{O}_3/\text{YAG}$ [6]. The three oxides of  $\text{Al}_2\text{O}_3$ , YAG and  $\text{ZrO}_2$  are not intermediate compounds, which have good compatibility at high temperatures. It has been reported that flexural strength of  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  ternary eutectic composite

**Foundation item:** Project(50772090) supported by the National Natural Science Foundation of China; Project(04G53048) supported by the Aeronautical Science Foundation of China; Project(20040699035) supported by the Specialized Research Fund for the Doctoral Program of Higher Education of China; Project(W018101) supported by the Foundation Research Fund of Northwestern Polytechnical University, China; Project(2007AMM004) supported by the Opening Project of State Key Laboratory for Advanced Metals and Materials, China; Project supported by the Fund of the State Key Laboratory of Solidification Processing in Northwestern Polytechnical University, China; Project supported by the Scientific Research Start-up Foundation for Outstanding Persons in Northwestern Polytechnical University, China

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DOI: 10.1016/S1003-6326(09)60065-0

reaches 800 MPa at 1 600 °C and the toughness is up to  $8.0 \text{ MPa}\cdot\text{m}^{1/2}$ , which are more than twice as high as that of the  $\text{Al}_2\text{O}_3/\text{YAG}$  binary eutectics[16]. As a result, much more attention has been recently paid to  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  ternary eutectic composite and various preparation techniques have been developed[13, 17–19].

Laser zone remelting is a recently developed solidification technique for preparing oxide eutectic ceramic in situ composite, which has advantages of nonexistence of crucible, high melting temperature, large thermal temperature gradient ( $\sim 10^6 \text{ K/m}$ ), as well as high growth rate and low cost[14, 20]. This technique has been used to grow  $\text{Al}_2\text{O}_3/\text{YAG}$  binary eutectic composite [14]. However, due to the complexity of oxide physical properties and the non-equilibrium of laser rapid solidification processing, the study on the effects of laser processing parameters on solidification behavior of ternary  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  eutectic composite is limited up to now. Understanding the solidification behavior and the formation of the composite can help us to recognize the mechanical properties and to gain the expected properties through particular processes. Additionally, the preparation and forming of ternary eutectic ceramic composite are more difficult than those of binary eutectic. In the present study, rapid surface resolidification using a high powered  $\text{CO}_2$ -laser was performed in producing directionally solidified  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  ternary eutectic ceramic in situ composite, aiming to investigate the effects of laser processing parameters on solidification microstructures and eutectic growth. Also, the high-temperature thermal properties and phase separation rule of the ternary eutectic were investigated.

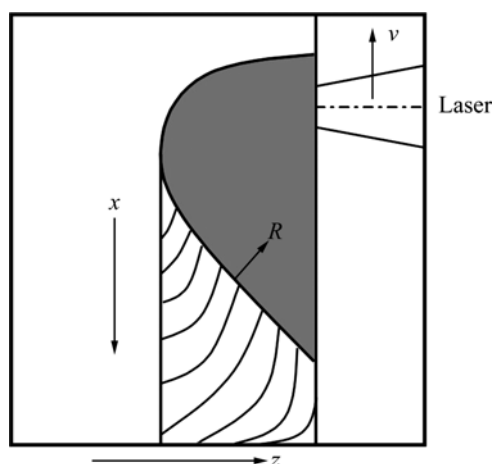
## 2 Experimental

### 2.1 Preparation of eutectic samples

The starting materials were prepared from a mixture of commercial high-purity ( $>99.99\%$ ) nano-powders of  $\text{Al}_2\text{O}_3$ ,  $\text{Y}_2\text{O}_3$  and  $\text{ZrO}_2$  according to the ternary eutectic composition. The molar ratio of eutectic in the  $\text{Al}_2\text{O}_3\text{--Y}_2\text{O}_3\text{--ZrO}_2$  ternary system was 65.8/15.6/18.6 [21]. The oxide powders were mechanically mixed by wet ball milling with an aqueous solution of polyvinyl alcohol to obtain homogeneous slurry and then dried at 200 °C in air for 1 h. Afterwards, the precursor rods of 7 mm in diameter and up to 60 mm in length were prepared by uniaxially die pressing at 25 MPa for 10 min, followed by sintering at 1 773 K for 2 h to increase the density and provide handling strength.

The directional solidification experiment was performed in a vacuum chamber using a 5 kW  $\text{CO}_2$  laser (ROFIN-SINAR 850) equipped with a numerically-controlled worktable with 5-axis and 4-direction coupled motion. The precursor samples were located on the laser

worktable and exactly moved along the sample axis to scan the sample along the laser direction, as shown in Fig.1. Under the high-energy laser beam, the samples were rapidly remelted and solidified to form the as-solidified eutectic samples. More detailed experimental procedure has been described elsewhere [14]. Finally, the crackless rod eutectic ceramic samples with smooth surface and high density can be obtained.



**Fig.1** Schematic diagram of laser zone remelting for growing ternary eutectic composite: *R*—Solidification direction; *v*—Scanning direction; *x*, *z*—Worktable moving direction

### 2.2 Characterization

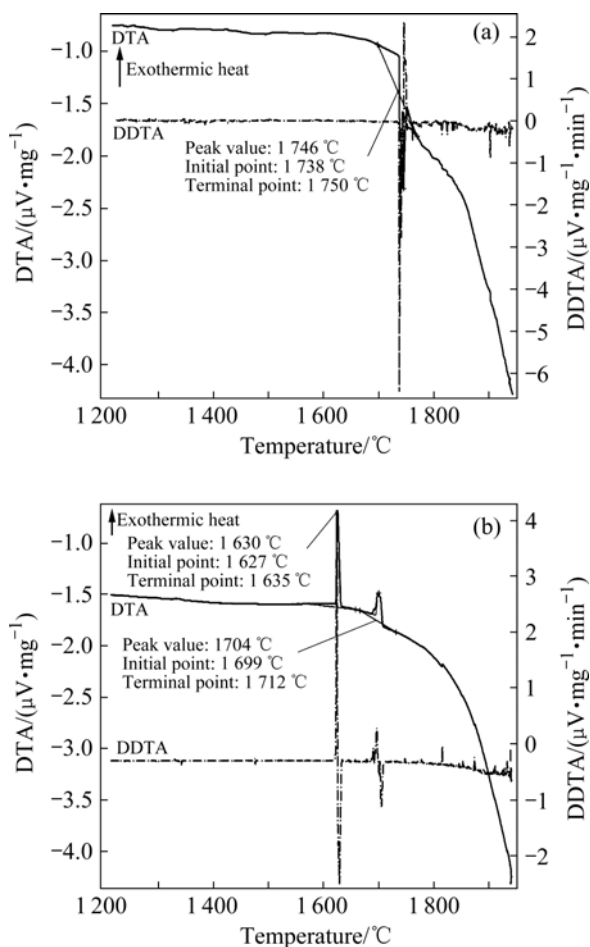
The microstructure and phase composition of the ternary eutectic were analyzed by the scanning electron microscopy (SEM, Supra-55), the energy disperse spectroscopy (EDS, Link-Isis) and the X-ray diffraction (XRD, Rigakumsg-158) techniques. Quantitative calculation of the eutectic phase volume fraction was performed by the digital image analysis software of SISC IAS V8.0. The thermal properties and phase separation behavior of the composite were investigated by the simultaneous thermal analyzer (STA, Netzsch-409CD). Approximately 25 mg of the as-solidified sample was heated in a Mo crucible at a heating rate 10 °C/s up to 1 950 °C in a flowing stream of Ar, and the corresponding cooling rate was 10 °C/s.

## 3 Results and discussion

### 3.1 Thermal behavior

The DTA-DDTA curves in the heating and cooling processes are shown in Figs.2(a) and (b), respectively.

For the heating state (Fig.2(a)), it can be clearly seen that there is only a sharp endothermic peak occurring in the temperature range of 1 738–1 750 °C, indicating the melting of ternary eutectic structure, which well agrees with the eutectic reaction point (1 715 °C) of the phase diagram of the  $\text{Al}_2\text{O}_3\text{--Y}_2\text{O}_3\text{--ZrO}_2$  system[21]



**Fig.2** DTA-DDTA curves of laser remelted  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  ternary eutectic composite: (a) Heating process; (b) Cooling process

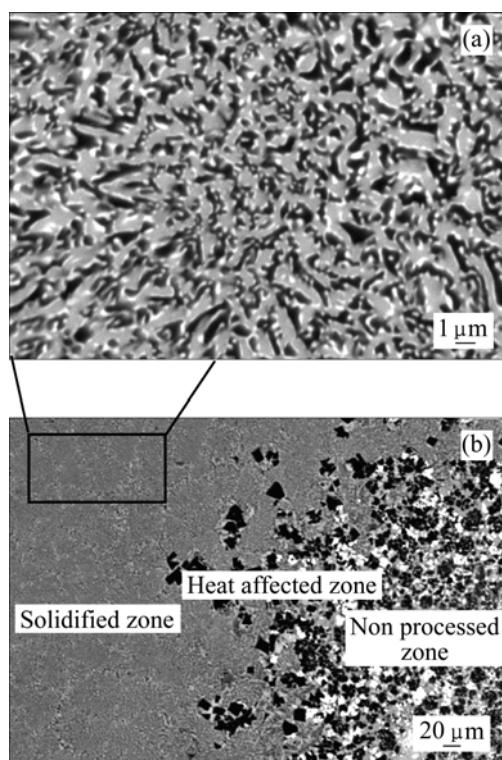
and verifies that the composition of the composite lies in the eutectic point or very near the eutectic point. However, during the cooling, there are two exothermic peaks appearing at the temperatures of 1704 °C and 1630 °C, respectively, and the latter is much more sharp than the former, as shown in Fig.2(b). The formation may be attributed to the following reasons. On one hand, in the  $\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3\text{-ZrO}_2$  ternary system, there is a very narrow coupled growth zone for eutectic solidification. In common, the  $\text{ZrO}_2$  is relatively easy to volatilize than  $\text{Al}_2\text{O}_3$  or  $\text{Y}_2\text{O}_3$  at elevated temperatures. A little volatilization of  $\text{ZrO}_2$  may drive the composite to deviate the eutectic composition, consequently, resulting in a hypoeutectic composition. Therefore, during the solidification, the first exothermic peak may be due to the nucleation of the primary  $\text{Al}_2\text{O}_3/\text{YAG}$  phase, and then the composite enters into the  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  equilibrium eutectic solidification process, accordingly, occurring the second obvious exothermic peak. Moreover, in the experimental investigation of  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  ternary hypoeutectic, the  $\text{Al}_2\text{O}_3/\text{YAG}$

also commonly acts as the primary phase to nucleate[19], thus leading to two exothermic heats. Similar DTA exothermic heats during the solidification process had also been observed by YASUDA et al[22] in the  $\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3\text{-ZrO}_2$  hypoeutectic composite.

On the other hand, for the  $\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3\text{-ZrO}_2$  ternary system the phase selection is determined by the maximum melting temperature before solidification and nucleation of YAG[23–24]. MIZUTANI et al[24] systemically investigated the phase selection of the  $\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3\text{-ZrO}_2$  system. The study indicates that there are two eutectic reactions in the system: one is the  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  equilibrium eutectic system and the other is  $\text{Al}_2\text{O}_3/\text{YAP}/\text{ZrO}_2$  metastable eutectic system. When the melt is cooled from temperature above 1977 °C, the nucleation of YAG is completely inhibited and consequently, the  $\text{Al}_2\text{O}_3/\text{YAP}/\text{ZrO}_2$  metastable eutectic system is selected. On the contrary, when the eutectic melt is cooled from temperature below 1977 °C, the solidification obeys the  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  equilibrium eutectic path, and is relatively easy to form the  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  eutectic. Moreover, the addition of  $\text{ZrO}_2$  can remarkably inhibit the YAG nucleation to form  $\text{Al}_2\text{O}_3/\text{YAP}/\text{ZrO}_2$  metastable eutectic. So, the first weak exothermic peak during solidification may also result from the formation of the metastable phase YAP, and then the YAP transforms into YAG, afterwards the melt enters into ternary eutectic solidification. Thus, a strong exothermic peak appears.

### 3.2 Ternary eutectic microstructure

Fig.3(a) shows the typical microstructure of the cross section of the laser remelted  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  eutectic ceramic composite. The XRD and EDS analyses show that the directionally solidified  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  ternary eutectic prepared by laser zone melting consists of  $\alpha\text{-Al}_2\text{O}_3$  (the black), YAG (the gray) and  $\text{ZrO}_2$  (the white) phases without any other phases. The process which effectively avoids existence of other compounds such as  $\text{YAlO}_3$  or  $\text{Y}_2\text{Al}_4\text{O}_9$  commonly appears in the primary sintered precursor with the sample composition. The  $\text{Al}_2\text{O}_3$  and YAG phases are homogeneously distributed and interconnected each other, and fine  $\text{ZrO}_2$  is embedded at the interface of  $\text{Al}_2\text{O}_3/\text{YAG}$  without grain boundaries. Fig.3(b) shows the microstructure of the interface between the solidified zone and non-processed zone in the  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  eutectic ceramic. It can be seen that the microstructure in the solidified zone exhibits a typical irregular lamellar structure with fine eutectic spacing and has no pore and crack, belonging to typical rapid solidification microstructure. However, in the non-processed zone, the microstructure is coarse and has obvious pores and grain

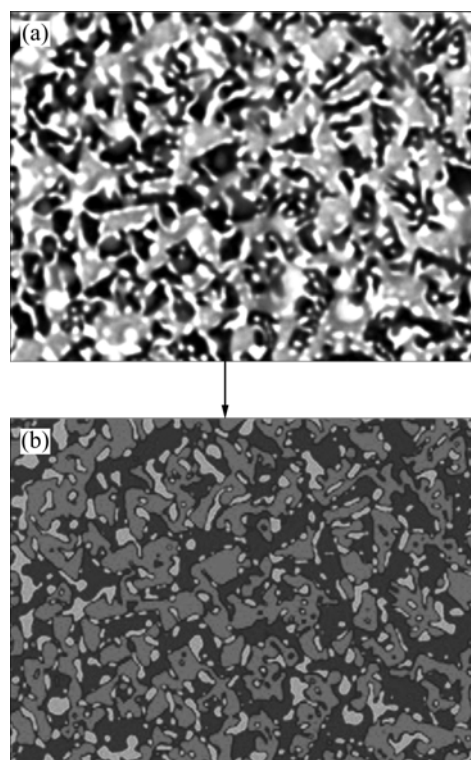


**Fig.3** Typical solidification microstructure of cross section of laser remelted  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  ternary eutectic ceramic (a) and microstructure of interface between solidified zone and non-processed zone (b)

boundaries. In the heat affected zone, it starts to appear coarse solidification microstructure, the grain boundaries gradually disappear and faceted  $\text{Al}_2\text{O}_3$  primary phase appears. Further nearing to the solidified zone, the microstructure gradually transits to ternary couple growth with fine eutectic spacing. The above analysis indicates that laser zone remelting can efficiently improve the microstructure and phase constituents of the oxide ceramic. Using the digital image analysis method, as shown in Fig.4, the volume ratio of the eutectic phases is measured to be  $\text{Al}_2\text{O}_3:\text{YAG}:\text{ZrO}_2=40:43:17$ , which well coincides with that expected for ternary eutectic composition[21].

### 3.3 Effects of laser processing parameters on solidification microstructures

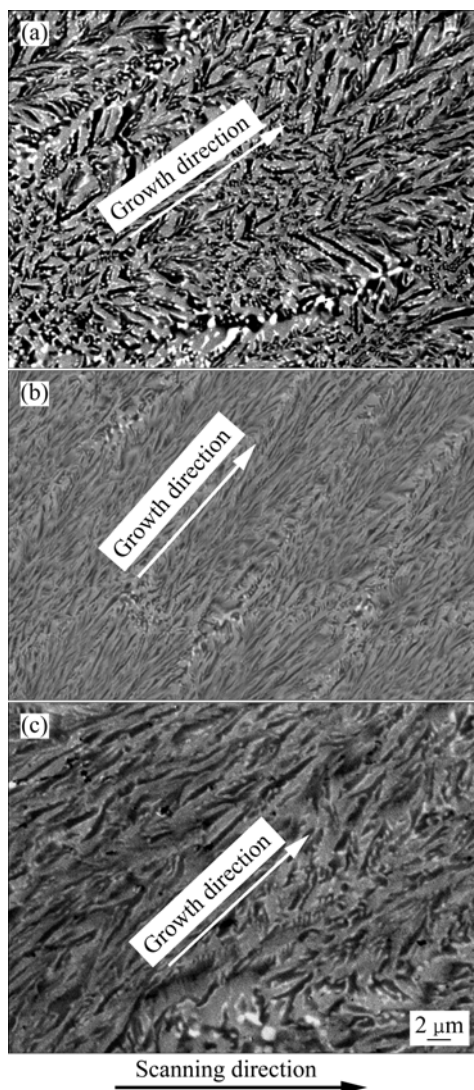
Fig.5 shows the typical longitudinal microstructures of the ternary  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  eutectic ceramic grown at different laser powers and scanning rates. During the laser zone remelting, the laser scanning rate and laser power are the primary solidification processing parameters for successfully preparing the oxide eutectic ceramic composite and determining the microstructure characteristic. Oxide ceramic has a high absorption of laser radiation ( $>60\%$ )[20], thus only low power (about



**Fig.4** Digital image analysis method for measuring eutectic phase volume ratio: (a) Original SEM image; (b) SEM image processed by two-value segmentation method

200 W) can melt the ceramic precursor.

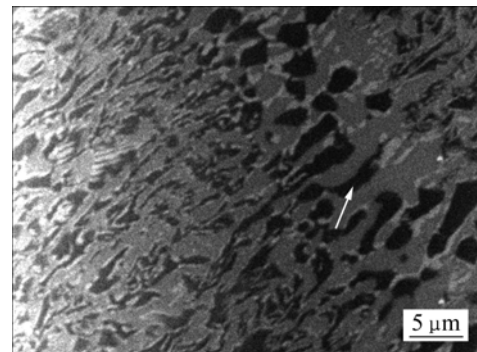
One of the important microstructural characteristics during rapid solidification of oxide eutectic composite is the eutectic spacing. Eutectic spacing depends on the thermal effects during solidification and influences the mechanical properties of the solidified ceramic. Figs.5(a) and (b) indicate that with increasing the laser scanning rate, the eutectic microstructure is rapidly refined when the laser power is kept at 200 W. The average eutectic spacing decreases from  $0.7\text{ }\mu\text{m}$  down to  $0.2\text{ }\mu\text{m}$  as the scanning rate increases from  $100\text{ }\mu\text{m/s}$  to  $600\text{ }\mu\text{m/s}$ . The ternary eutectic spacing is much smaller than that of the binary eutectic[10, 14]. The refinement of the ternary eutectic microstructure obtained is mainly attributed to rapid solidification rate and high temperature gradient during the laser zone remelting process. Moreover, the addition of  $\text{ZrO}_2$  also greatly contributes to the microstructure refinement. Furthermore, Figs.5(a) and (c) show that when the laser scanning rate is kept constant, with increasing the laser power, the eutectic spacing is increased. The average eutectic spacing increases from  $0.7\text{ }\mu\text{m}$  up to  $1.1\text{ }\mu\text{m}$  as the laser power increases from 200 W to 300 W. This is mainly because that when the laser power increases, the corresponding cooling rate is decreased, thus leading to the increase of the eutectic spacing. Similar phenomenon is also found in the laser



**Fig.5** Typical longitudinal microstructures of ternary  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  eutectic grown at different laser powers ( $p$ ) and scanning rates ( $v$ ): (a)  $P=200$  W,  $v=100$   $\mu\text{m/s}$ ; (b)  $P=200$  W,  $v=600$   $\mu\text{m/s}$ ; (c)  $P=300$  W,  $v=100$   $\mu\text{m/s}$

surface modified alumina ceramic. When further increasing the laser power, the pores may be produced. Additionally, because the solidification direction is gradually changed from bottom to top of the solidified eutectic sample (Fig.1), the growth direction has a deviation from the scanning direction, as the arrows show in Fig.5.

Moreover, laser zone remelting is a non-equilibrium solidification process. The asymmetry of the temperature field, the convection caused by the melt surface tension and gravity, and instability of laser worktable commonly lead to the non-homogenous microstructure in local zone, as shown in Fig.6. In Fig.6, the coarse eutectic microstructure and fine eutectic microstructure coexist in the ternary composite, which will influence the mechanical properties of the composite[6]. Furthermore,



**Fig.6** Non-homogenous microstructure and branching of faceted phases in ternary  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  eutectic composite

the non-steady eutectic growth is intrinsic to faceted crystals[25]. Because  $\text{Al}_2\text{O}_3$ , YAG and  $\text{ZrO}_2$  have high entropies of fusion[19], they commonly exhibit faceted growth interface. When the faceted crystal grows, frequent branching of three eutectic phases will occur, as the arrow shows in Fig.6. As a result, a complex entangled ternary eutectic microstructure is formed.

## 4 Conclusions

1) Directionally solidified  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  ternary eutectic ceramic in situ composite with fine irregular lamellar microstructure was obtained by laser rapid resolidification. The volume ratio of the eutectic phases is  $\text{Al}_2\text{O}_3:\text{YAG}:\text{ZrO}_2=40:43:17$ . Laser zone remelting efficiently improves the microstructure of the ceramic composite.

2) Synthetically thermal analysis indicates that the eutectic temperature of  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  obtained is 1 738  $^{\circ}\text{C}$ , well matching the phase diagram of  $\text{Al}_2\text{O}_3\text{-Y}_2\text{O}_3\text{-ZrO}_2$  ternary system.

3) The thermal effects associated with the laser zone remelting significantly influenced the rapid solidification microstructure in laser remelted  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  ternary eutectic composite. The eutectic spacing decreases with increasing the laser scanning rate, but increases with increasing the laser power.

4) The complex entangled ternary eutectic microstructure in the  $\text{Al}_2\text{O}_3/\text{YAG}/\text{ZrO}_2$  composite is formed mainly due to the faceted growth and the branching of the eutectic phases, and non-equilibrium of laser rapid solidification process.

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(Edited by YANG Hua)