Journal of Crystal Growth 449 (2016) 67–74

Contents lists available at ScienceDirect

Journal of Crystal Growth

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

Shaped ceramic eutectic plates grown from the melt and their properties

Omar Benamara, Kheirreddine Lebbou *

Institut Lumière Matière, UMR5306 Université Lyon1-CNRS, Université de Lyon, Lyon 69622, Villeurbanne Cedex, France

ARTICLE INFO

Article history:
Received 6 March 2016
Received in revised form
27 May 2016
Accepted 29 May 2016
Communicated by: Satoshi Uda
Available online 30 May 2016

Keywords:
A1. Eutectics
A1. Morphological stability
A2. Growth from melt
B1. Oxides

ABSTRACT

Al_2O_3–YAG–ZrO_2 eutectic ceramic plates 10 mm in width, 2.4 mm in thickness and 100 mm in length were directionally grown by micro-pulling down technique under different growth rate (0.1–1 mm/min). The effects of the solidification rate on the crystallographic orientation and microstructure properties were analyzed in considerable detail. The microstructure spacing λ depends on the pulling rate v following the law: $\lambda=7.\ 2v^{-\frac{1}{2}}$ where λ is in μm and v in μm/s. For low growth rate ($v<0.25$), the ternary eutectic plates had homogeneous and regular microstructures and higher pulling rate heterogeneity in the morphology corresponding to colonies presence. The annealed plates did not undergo chemical reaction but slight grain growth was observed.

1. Introduction

In the last decade, the elaboration process, microstructure and properties of textured solidified eutectics ceramic oxides have been largely reviewed [1–4]. These materials have a performed properties which make them of great interest as potential engineering materials: High melting temperature, good resistance to corrosion by liquids and gases and high temperature potential strength. These performed properties allow to use them to solve the materials engineering problem in the highest temperature areas of gas turbine engines. The need for operation at higher gas turbine temperature continues to increase because it allows directly efficiency increasing and pollution decreasing very important for the environment. Unfortunately, the engineering design to develop the eutectic ceramic oxides with homogeneous microstructure is clearly difficult. A large portion of the efforts in eutectic ceramic oxides research have dealt with developing process technology [5–11]. To elaborate homogeneous morphology under flat solid-liquid interface during solidification at microscopic and macroscopic level it is necessary to control the thermal gradients along the solidification direction. It has been clearly shown in a number of eutectic system that the knowledge of the relationship between microstructures and properties is a key factor to control the system performance [12–16]. The functional properties of the eutectic oxides materials are strongly dependent on characteristics of the microstructure (morphology, phase geometry and size) which dependent on the solidification process. Based on the equilibrium diagram, binary Al_2O_3–ZrO_2 and ternary Al_2O_3–ZrO_2–Y_2O_3 eutectic system were studied in detail by Lakiza et al. [17]. The addition of Y_2O_3 oxide to Al_2O_3–ZrO_2 system permit the obtainment of the pseudo binary Al_2O_3–$ZrO_2(Y_2O_3)$ eutectic system in which different zirconia polymorphism (Monoclinic, tetragonal or cubic zirconia) could be formed just by changing yttrium concentration. Depending on zirconia polymorphism, a variety of microstructural morphology and residual stress distribution will control the properties [18]. The purity of the starting material is important to design optimum eutectic microstructures. The presence of impurities is one reason for localized constitutional supercooling and the formation of a cellular interface at the boundary between the liquid and solid during solidification process. Another important point to control the microstructures, it is thermal gradient across the liquid-solid interface be as high as possible. The magnitude of the thermal gradient limits the maximum rate at which solidification can occur without the undesirable breakdown of planar-liquid interface and the formation of a colony microstructures. In addition if the thermal gradient can be made sufficiently high, high solidification rate results in a finer microstructure and generally more desirable properties. In this paper, we have used micro-pulling down technique for unidirectional Al_2O_3–YAG–ZrO_2 eutectic plates crystallization from the melt under stationary stable regime. The microstructure, morphology, crystallography and residual stress evolution as function of the growth rate are discussed.

* Corresponding author.
E-mail address: kheirreddine.lebbou@univ-lyon1.fr (K. Lebbou).

http://dx.doi.org/10.1016/j.jcrysgro.2016.05.046

2. Experimental procedure

The starting material used in this work was made from commercial high-purity (> 99.99%) Al_2O_3, Y_2O_3 and ZrO_2 powders of nano-spherical shape. The powders were taken at eutectic proportion [17] 65 mol%Al_2O_3 /16 mol%Y_2O_3/19 mol%ZrO_2, mixed in an agate mortar and sintered at 1400 °C for 10 h.

Eutectic ceramics plates were solidified by the micro-pulling down technique (μ-PD) and the experimental set- is described in our previous works [19–22]. An iridium crucible with plate shape geometry was coupled to a radio frequency (RF) induction heating source. To visualize the melt meniscus, the interface shape and all the phenomenon which can be involved in the melting zone a CCD camera was used to take inset a live picture through a small rectangular window made in the iridium after heater and alumina ceramic thermal insulation around the hot zone (Fig. 1). To protect iridium crucible and after heater form oxidation and damage argon atmosphere (2 L/min) was used during the solidification process.

At the extremity of the capillary die crucible a pendant drop flows and wet the lip of the die. A eutectic rod seed containing Al_2O_3, YAG and ZrO2 phases with diameter of 3 mm was used for seeding. After connection, the seed was pulled down with low rate to enlarge the plate up to its final shape (Fig. 1). The solidification process was controlled by manual adjustment of power generator. In this work, the solidification rate was varied from 0.1 to 1 mm/min. 100% of the melt was solidified. Depending on the initial

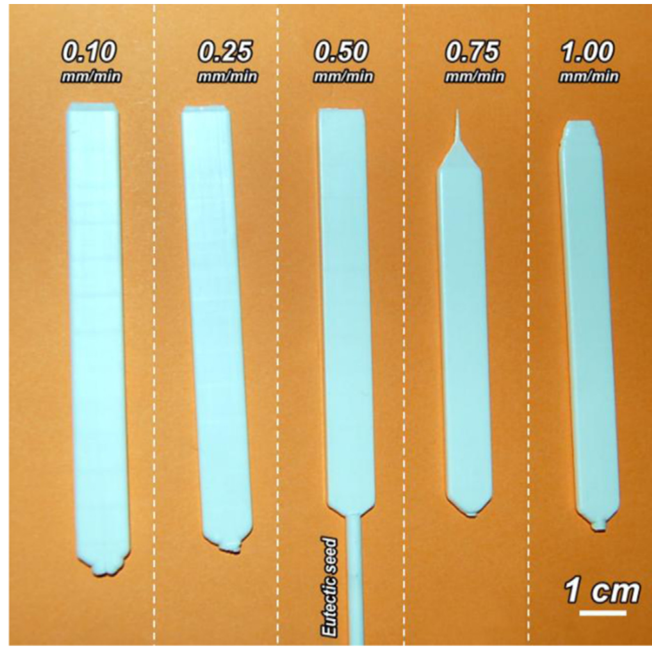

Fig. 2. Al_2O_3–YAG–ZrO_2 ternary eutectic plates pulled from the melt by μ-PD technique at different rates.

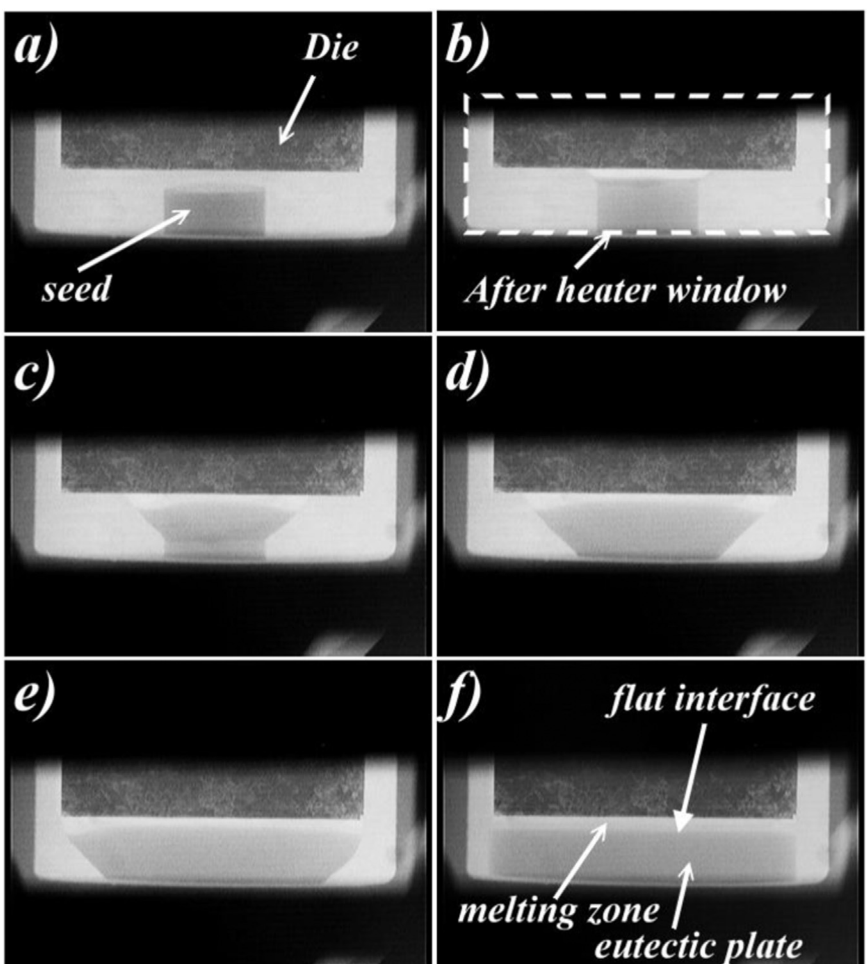

Fig. 1. Plate growth steps, (a) before connection, (b) connection, (c, d and e) pulling down and eutectic plate widening, (f) regular plate form after a few millimeters of pulling.