Available online at www.sciencedirect.com
ScienceDirect
Journal of the European Ceramic Society 34 (2014) 4077–4082
www.elsevier.com/locate/jeurceramsoc

Short Communication

Novel freeze-casting fabrication of aligned lamellar porous alumina with a centrosymmetric structure

Yufei Tang *, Qian Miao, Sha Qiu, Kang Zhao, Long Hu

Department of Materials Science and Engineering, Xi'an University of Technology, Xi'an 710048, PR China

Received 10 April 2014; received in revised form 25 May 2014; accepted 29 May 2014
Available online 21 June 2014

Abstract

A novel freeze-casting method is used to fabricate aligned lamellar porous alumina with a centrosymmetric structure from aqueous alumina slurries. Two cold fronts oriented perpendicularly to each other, originating from the bottom and side of the cylindrical copper mold, induce the growth of ice crystals in specific directions along the radius of the cylindrical mold. Lamellar channels of porous alumina are arranged centrosymmetrically along the radial axis. The pore distribution of the currently prepared porous ceramics is more regular when compared with that of porous ceramics prepared by conventional freeze casting. This affords porous ceramics with improved mechanical properties and stability. The current method addresses the issue of partial failure as induced by the randomly distributed channels in lamellar porous ceramics.

Keywords: Freeze casting; Porous ceramics; Alumina; Centrosymmetric structure; Mechanical properties

1. Introduction

Porous ceramics are widely used in areas of catalytic reaction carriers, biological implants, filters, and solid oxide fuel cells, owing to their chemical stability and high porosity.[1,2] However, the high brittleness of ceramic materials results in low mechanical properties, thereby limiting the applications of porous ceramics in many area.[3,4] Most research studies focus on the improvement of mechanical properties, such as compression strength, by developing new synthesis methods.[5–8] In 2004, Deville reported that the axial compressive strength of cylindrical porous ceramics prepared by directional freeze-casting methods is about four times higher than those prepared by other methods at a given material porosity.[9] Since then, many researchers have examined freeze casting to preparing porous ceramics with high mechanical properties. Alteration of the freezing processing conditions affords control over the pore size and pore distribution of the ceramics, and adjustment of the solid content enables control over the porosity of the final material.[10–12]

Porous ceramics prepared via freeze casting feature high compressive strength only in the axial direction that corresponds to the freezing direction because of the formation of lamellar channels as induced by the formation of ice crystals along the freezing direction.[13] In contrast, in the radial direction, random lamellar channels form owing to the random distribution of the ice crystals. Thus, the compressive strength of porous ceramics in the radial direction is anisotropic and unstable.[14] Areas featuring lamellar channels that are not parallel to the external force direction will collapse partially. Although the high strength in the axial direction meets the requirements of loading in practical applications,[15,16] such as bone implant scaffolds,[17] the instability of the mechanical properties in the radial direction is one of the causes of potential failure in porous ceramics. Thus, increasing the mechanical properties of cylindrical porous ceramics in the radial direction is important.

Directional freezing results in the anisotropic growth of ice crystals.[18] The velocity of the ice front parallel to the freezing direction is 10^2–10^3 times higher than the velocity perpendicular to the freezing direction, thereby generating ice platelets with a very large anisotropy and a small thickness.[19] Fig. 1a shows a schematic representation of lamellar porous ceramics prepared by freeze casting. Ice crystals in the *z*-axis direction, parallel

* Corresponding author. Tel.: +86 29 82312922; fax: +86 29 82312922.
E-mail address: yftang@xaut.edu.cn (Y. Tang).

http://dx.doi.org/10.1016/j.jeurceramsoc.2014.05.040

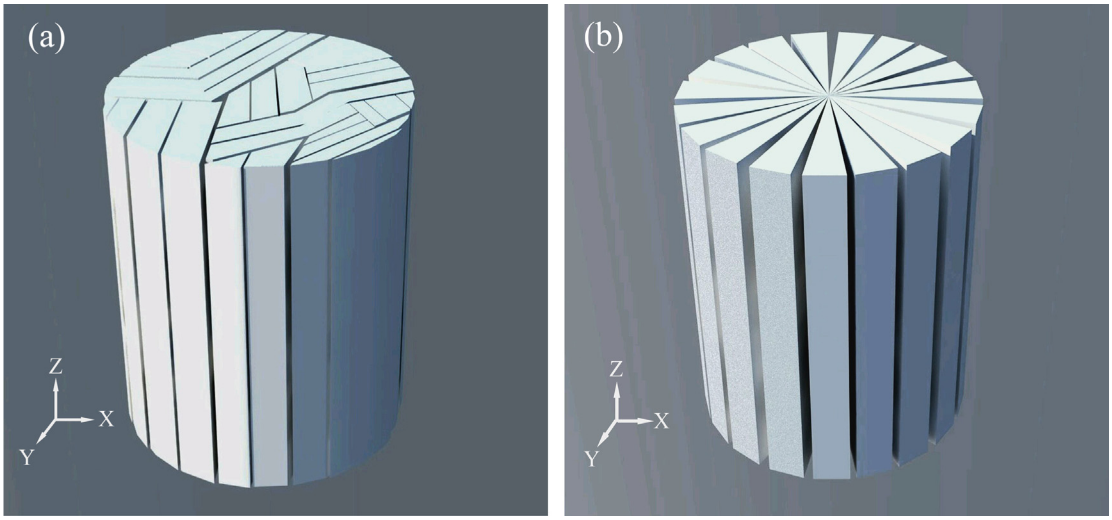

Fig. 1. Schematic representations of lamellar porous ceramics prepared by (a) conventional freeze casting and (b) novel freeze casting.

to the freezing direction (or temperature gradient), grow more easily. Thus, crystals will grow at the expense of other crystals and continue to grow in an upward direction. The temperature gradient along the horizontal planes that are parallel to the *x*–*y*-plane is the same, however, the distribution of the ice crystals in the horizontal plane is random. It is believed that if the distribution of ice crystals is regular along the radial direction of the cylindrical samples, aligned lamellar porous ceramics with a centrosymmetric structure (Fig. 1b) and a high radial compressive strength are expected to form following ice sublimation and sintering.

In the current study, a novel freeze-casting technique is proposed, using a cylindrical mold with sides and bottom made of conductive metal, which would promote the growth of ice crystals. Ice crystals are expected to be distributed along the radial direction of the cylindrical mold, as restricted by the two temperature gradients when ice crystals grow along the hexagonal plane. The growth behavior of ice crystals using a dual-cold source system was investigated. The pore distribution and mechanical properties of the porous ceramics in the axial and radial directions were characterized.

2. Experimental procedure

Alumina powder (GW-1; Dengfeng Smelting Materials Co., Ltd., Zhengzhou, China) with a median size (d_{50}) of 1 μm was used as ceramic material and deionized water was used as freezing medium. Polyvinyl alcohol (Yakuri Pure Chemicals Co., Ltd., Osaka, Japan) was used as binder. Dispersant polyacrylate sodium (Sinopharm Chemical Reagent Co., Ltd., Shanghai, China) was used to stabilize the slurry.

Alumina powder was mixed with 2 wt.% dispersant and 1 wt.% binder in deionized water, and ground with a ball mill for about 24 h. The prepared slurries (alumina contents: 20, 30, and 40 vol.%) were respectively poured into copper cylinders (diameter × length: 8 mm × 48 mm; 16 mm × 48 mm; and 24 mm × 48 mm), then placed on a pre-cooled plate at −40 °C in a freeze drier (Freezone 2.5 L Triad; Labconco, Kansas, USA) to ensure the top of the mold with a height of 48 mm get a temperature lower than the crystallization temperature of the slurry and completely freeze of the samples finally. The current freeze casting involves two cold sources at perpendicular directions to each other, i.e. originating from the bottom and side of the

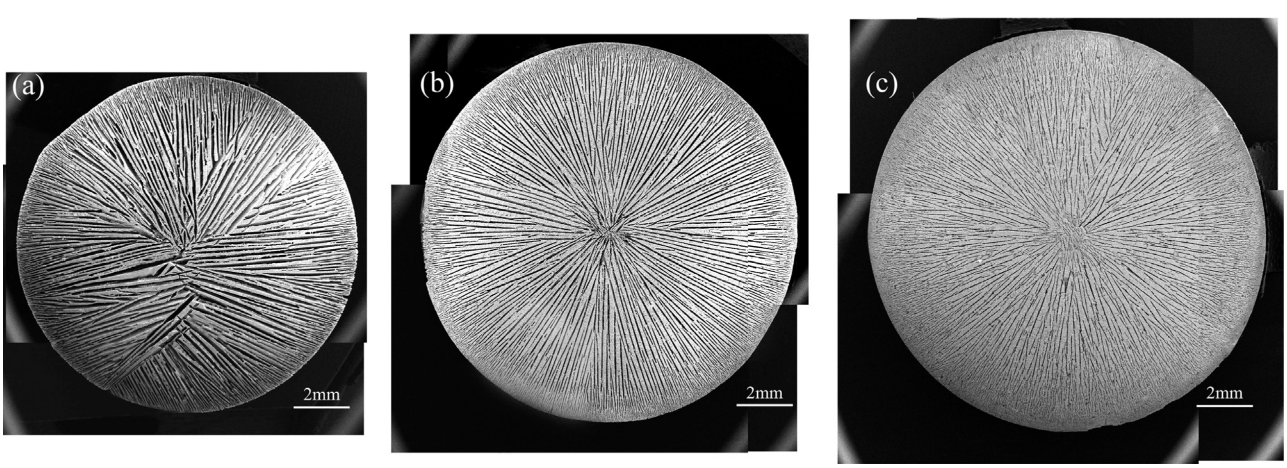

Fig. 2. Morphologies of lamellar porous alumina samples with centrosymmetric structures fabricated with different alumina contents: (a) 20, (b) 30, and (c) 40 vol.%.