Ceramics International xx (xxxx) xxxx–xxxx

Contents lists available at ScienceDirect

Ceramics International

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

Amino-functionalization of colloidal alumina particles for enhancement of the infiltration behavior in a silica-based ceramic core

Gye Seok An[a], Soo Wan Choi[a], Tae Gyun Kim[a], Jae Rok Shin[a], Yong-In Kim[a], Sung-Churl Choi[a,**], Young-Gil Jung[b,*]

[a] Division of Materials Science and Engineering, Hanyang University, 222 Wangsimni-ro, Seongdong-gu, Seoul 04763, Republic of Korea
[b] School of Materials Science and Engineeing, Changwon National University, 20 Changwondaehak-ro, Changwon, Gyeongnam 51140, Republic of Korea

ARTICLE INFO

Keywords:
Amine-functionalization
Precursor
Colloidal alumina
Ceramic core
Infiltration

ABSTRACT

The surface characteristics of nano-sized alumina particles were modified to include amine functional groups to enhance the infiltration behavior in a silica-based ceramic core, inhibiting crystallization of the cristobalite phase in order to improve mechanical properties. In this study, polymer-based polyethylenimine (PEI) or silane-based (3-Aminopropyl)triethoxysilane (APTES) were used as precursors in amine group addition. The specimens were characterized by the C-H stretching vibration and N-H bending vibration or the symmetric stretching vibration of siloxane group (Si-O-Si) in the FT-IR spectra, depending on the nature of the molecular structure of the precursor molecule. The dispersion properties were also improved through electrostatic repulsion or steric hindrance. These modifications resulted in a PEI-grafted colloidal alumina particle that induced a more rapid infiltration process with greater efficiency than did the APTES-treated particle. The rapid infiltration of the ceramic core suppressed the formation of the cristobalite phase, improved the flexural strength from 3.2 to 10.2 MPa, and reduced the linear shrinkage rate from 1.91–1.05%.

1. Introduction

Ceramic cores produced by injection molding have been extensively used in hollow gas turbine blades and vanes as part of the internal cooling path [1–5]. The ceramic cores are typically based on fused silica (SiO_2) and zircon ($ZrSiO_4$) materials, which are able to endure thermal stress induced by the high temperature investment casting process. These substances are selected due to their thermal shock resistance and chemical inertness against molten metal [6,7]. In order to endure these extreme conditions, the ceramic core must possess appropriate mechanical properties. High-temperature sintering is essential in the production of ceramic cores, but the fused silica is also converted to the phase at temperatures greater than 1300 °C. The volume reduction that results from the β to α phase transformation of cristobalite is the main cause of the shrinkage of the ceramic cores. Additionally, the microcrack produced in this phase transformation reduces the flexural strength. At temperatures near 350 °C, when the β phase of cristobalite is present, the flexural strength increased; it is decreased at room temperature. The changes in these mechanical properties are caused by the microcrack formed in the phase transformation, as described above [8–11]. A way to prevent the crystallization of the fused silica is needed in order to suppress the degradation of the silica-based ceramic core.

In our previous work, we introduced a method to prevent the crystallization of fused silica via infiltration with a colloidal alumina particle. The crystallization of cristobalite was limited due to the mullite phase formed by the reaction between the surface of fused silica and the absorbed alumina particle. The obtained infiltrated fused silica with 2.54% alumina content had a relatively high physical property. Even with this improvement to physical properties of the ceramic core, the efficiency is to still insufficient to apply to the production process due to relatively long infiltration time (150 min) [12].

In this study, the surface of alumina particles was modified in order to enhance the performance of the infiltration process. The silica surface is negatively charged due to the large amount of hydroxyl groups (-OH); therefore, it is advantageous to introduce a positive charge to improve the adsorption of the alumina particles through electrostatic attraction. The positively charged functional group most often utilized is the amine group. Polymer-based polyethylenimine or silane-based (3-Aminopropyl)triethoxysilane were used as precursors in the modification of the alumina particle surface. Infiltration effi-

* Corresponding author.
** Co-corresponding author.
E-mail addresses: choi0505@hanyang.ac.kr (S.-C. Choi), jungyg@changwon.ac.kr (Y.-G. Jung).

http://dx.doi.org/10.1016/j.ceramint.2016.09.127
Received 8 September 2016; Received in revised form 13 September 2016; Accepted 19 September 2016
Available online xxxx
0272-8842/ © 2016 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

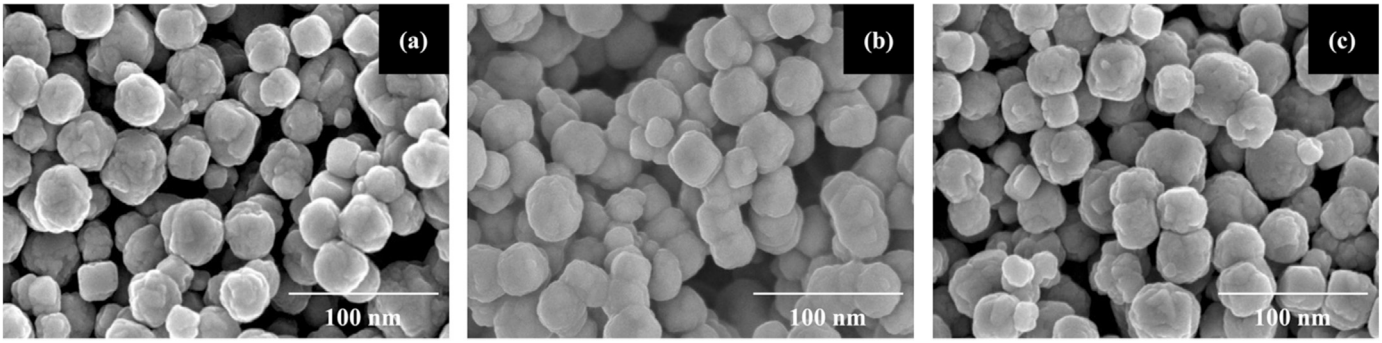

Fig. 1. High resolution SEM images of (a) as-prepared alumina, (b) PEI grafted alumina, and (c) APTES treated alumina.

ciency, the mechanical properties, and the crystal structure were improved through this modified preparation of colloidal alumina particles.

2. Experimental process

Commercially available nano-sized aluminum oxide (Al_2O_3, APS 40–50 nm, 99.5%, Alfa-Aesar, United States) was surface-modified through hyper-branched polyethylenimine (PEI, Mw 60,000, Sigma-Aldrich, United States), and (3-Aminopropyl)triethoxysilane (APTES, 99%, Sigma-Aldrich, United States). Alumina powder (5*g*) was suspended in 500 ml of deionized water and stirred at 400 RPM at room temperature for 1 h. 3 wt% of PEI in deionized water was added to the well-dispersed alumina solution and mixed at 80 °C for 3 h. The resulting colloidal particles were centrifuged at 10,000 RPM for 1 h. The refined particles were rinsed with deionized water several times and collected.

To modify the surface by silane grafting, alumina powder (5 g) in 500 ml of deionized water was mixed with 100 ml of 1N ammonia solution at 250 RPM at 80 °C for 3 h. The sample was then cooled by the addition of 200 ml of deionized water and mixed with 150 ml of ethanol at 250 RPM at 60 °C for 12 h. The silane precursor solution, composed of 2 ml of APTES, 1 ml of ethanol, and 1 N of ammonia solution, was added 30 min into the procedure. After heating was complete, this product was washed as previously described for PEI.

The silica-based matrix of the ceramic core was prepared by ceramic injection molding. 63 wt% of fused silica (amorphous SiO_2, 325 mesh, IMERYS, United States) was used as the base material and was modified by addition of 21 wt% of zircon ($ZrSiO_4$, 1 μm, Cenotec, Korea). A thermoplastic binder (15 wt%) composed of paraffin wax (Nippon-seiro, Japan), microcrystalline wax (Nippon-Seiro, Japan), stearic acid ($C_{19}H_{36}O_2$, Samchun Pure Chemical, Korea) and oleic acid ($C_{19}H_{34}O_2$, 97%, Samchun Pure Chemical, Korea) was used as feedstock for injection molding. The feedstocks were injection molded using a C-frame ceramic injection molding machine (CTM-CI-CF-35-100HT, Cleveland Tools and Machines, United States) with 6 mm x 8 mm x 90 mm green bodies in accordance with ASTM C 1161-13. After calcination of the thermoplastic binders at 0.2 K/min, the specimens were pre-sintered at 1100 °C for 2 h with a heating rate of 5 K/min using a box furnace (UAF-15–27-LHE, Lenton, United Kingdom).

Pre-sintered specimens were infiltrated by 20% supensions of the prepared surface-modified colloidal alumina in deionized water under vacuum. The length of reaction was varied to achieve different levels of the alumina content. Resulting specimens were dried at 110 °C for 12 h to evaporate residual solvent and sintered at 1300 °C for 2 h.

The surface functional groups of the modified alumina particles were determined by Fourier transform infrared spectroscopy (FT-IR, IRAffinity-1S, Shimadzu, Japan). The zeta-potential value at various pH was measured using 0.1 M HNO_3 or 0.1 M NaOH in deionized water by electrokinetic sonic amplitude measurements (ESA, Zeta Finder, Matec Applied Sciences, United States). The particle size distributions were measured using dynamic light scattering (DLS) (Zetasizer Nano ZSP, Malvern, United States) in order to estimate the dispersion behavior for different functionalizing methods. The weight loss of infiltrated specimens was measured using a thermogravity/differential thermal analyzer (TG/DTA, SDT 2960 Simultaneous, TA instruments, United States) and particle images were obtained by field emission scanning electron microscopy (FE-SEM, S-4800, Hitachi, Japan) and EDX mapping (Link Pentafet, Oxford Instruments, United Kingdom). The crystal structure of sintered specimens was determined using an X-ray diffractometer (XRD, D/Max-220, Rigaku, Japan) under Cu Kα radiation (λ=1.54178 Å). The flexural strength and shrinkage rate were estimated based on 3-point bending test with a universal testing machine with a span size of 80 mm and crosshead speed of 1.0 mm/min (UTM, H10SK, Hounsfield, United Kingdom).

3. Results and discussion

The morphology of as-prepared and each surface modified alumina particles were examined by SEM in Fig. 1. The as-prepared alumina particles have slightly keen edged quasi-spherical shape with about 40 nm of average diameter approximately. However, the morphology of PEI grafted alumina particles exhibit rugged and distorted surface compared with the raw alumina particles due to grafting of polymer, which has randomly arranged polymeric structure. On the other hand, the morphology of APTES treated alumina particles are similar with the as-prepared one. It is possibly considered that silane layer formed around the surface of alumina particles by condensation of silanol group is homogeneous and very thin which cannot identify with naked eye, thus the morphology of both as-prepared and APTES treated alumina shows no significant difference..

The identity of the surface functional groups of the as-prepared alumina particles and PEI or APTES surface-modified particles through PEI or APTES was verified from the FT-IR spectra (Fig. 2). Absorption peaks for the Al-O stretching vibration were observed at 575 and 787 cm^{-1} and a broad absorption band of the surface hydroxyl group (Al-OH) in the range of 3700–3000 cm^{-1} due to the alumina base material [13,14]. Additionally, the N-H bending vibration peak at 1630 cm^{-1} from the functionalized amine group and the absorption bands between 3000 and 2700 cm^{-1} due to the C-H stretching vibration of alkyl chains of PEI and APTES were observed [15,16]. For the APETS-grafted specimen, the symmetric stretching vibration peaks of siloxane group (Si-O-Si) were observed at 1080 and 1120 cm^{-1}[16]. The broad absorption band between 3700–3000 cm^{-1} in both the surface modified alumina than as-prepared alumina particle is thought to be due to overlap of the –OH and N-H stretching vibrations [17]..

In Fig. 3, the zeta potential values at each pH were used to evaluate the level of surface charge. The fused silica has a negative zeta potential value (between −3 to −45 mV) between pH 2 and 11. In this same range, the zeta potential of as-prepared alumina particle was 5 to

Download English Version:

https://daneshyari.com/en/article/5438124

Download Persian Version:

https://daneshyari.com/article/5438124

Daneshyari.com