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Bi-phase ceramic composite using an interpenetrating network

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ABSTRACT

A secondary phase has been reinforced into a porous alumina (Al_2O_3) matrix using an interpenetrating network (IPN) method to enhance the mechanical properties of the porous matrix. To increase the addition effect of the secondary phase into the Al_2O_3 matrix, two types of SiO_2 precursor were used: tetraethylorthosilicate (TEOS) of the silicate type and polydimethylsiloxane (PDMS) of the siloxane type. The PDMS does not undergo a sol–gel reaction, whereas the TEOS is converted into glass-phase SiO_2 by a sol–gel reaction. This means that the siloxane type has a higher conversion ratio of precursor into the glass phase of SiO_2 than does the silicate type. The mechanical properties of the composites prepared using TEOS and PDMS without sodium methoxide (NaOMe) have been improved, showing 12.9 ± 5.9 and 9.9 ± 6.4 MPa in fracture strength, respectively, and 17.2 ± 4.6 and 15.5 ± 6.9 GPa in elastic modulus, respectively, while the mechanical properties of the porous Al_2O_3 matrix were 6.0 ± 1.2 MPa and 8.0 ± 4.0 GPa in fracture strength and elastic modulus, respectively. However, the mechanical properties of the composite prepared using PDMS with NaOMe were higher than those of the composite prepared using TEOS with NaOMe, showing 25.3 ± 7.3 MPa and 26.3 ± 5.5 GPa in fracture strength and elastic modulus, respectively. The increase in mechanical properties was caused by the enhancement of glassification owing to the high conversion ratio of the SiO_2 precursor, in the absence of a sol–gel reaction. Consequently, bi-phase composites with reasonable properties have been successfully prepared through the IPN method using inorganic precursors.

1. Introduction

Porous alumina (Al_2O_3) has been widely utilized for hot gas filtration, catalyst support, filtration of heavy metal ions (Cr and Ni) in water, chromatography, and fine or microchannels for electrophoresis, because of its excellent physical and chemical properties such as acid resistance and chemical stability [1,2]. However, its use in practical applications has been impeded by its naturally low mechanical properties, such as fracture strength, elastic modulus, and hardness, which have been a bottleneck from the scientific and technological points of view. Therefore, a hybrid composite reinforced by a secondary phase has been introduced for functional materials for industrial applications [3–6]. The secondary phase used as a reinforcing material should be homogeneously and uniformly dispersed into the ceramic matrix for maximization of the addition efficiency of the secondary phase. Moreover, the addition of a reinforcing phase should not affect the original shape of the porous ceramic.

Therefore, in this work, a bi-phase composite has been fabricated using the interpenetrating network (IPN) method and a liquid-phase inorganic precursor to increase the mechanical properties of the matrix and induce uniform infiltration of the secondary phase into the matrix. This inorganic precursor is converted into the glass phase, which will increase the mechanical properties of the porous ceramic. Therefore, the conversion ratio of precursor to glass phase, which is called the glassification efficiency, is an important factor in this work. Two types of SiO_2 precursor were used to investigate the glassification efficiency according to the type of precursor: tetraethylorthosilicate (TEOS) of the silicate type and polydimethylsiloxane (PDMS) of the siloxane type. The reaction phenomena of each precursor and the mechanical properties of the prepared bi-phase composites were analyzed and measured, respectively, using various analytical techniques.

2. Experimental procedure

Inorganic precursors used as a secondary phase were prepared using TEOS (bp 168 °C, Sigma-Aldrich Korea, Yongin, Korea) of silicate type or PDMS (bp 182 °C, Sigma-Aldrich Korea, Yongin, Korea) of siloxane type as a SiO_2 precursor, and sodium methoxide (NaOMe, Sigma-Aldrich Korea, Yongin, Korea). The green body of the ceramic matrix was prepared by a uniaxial pressing process with Al_2O_3 powder, and then the prepared green body was calcined at 1500 °C for 1 h. At room temperature, the inorganic precursor mixed to a specific composition was infiltrated into the porous ceramic matrix. The precursor-infiltrated matrix was dried at 80 °C for 24 h and then heat treated at 1000 °C for 1 h. The synthetic scheme for the bi-composite is given in Fig. 1, and the basic formulations employed to prepare the composites are shown in Table 1.

The structural transition of the precursors employed and the crystal phase of the composites prepared were analyzed with heat treatment, using a Fourier transform infrared (FT-IR) spectrometer (Nicolet, Thermo

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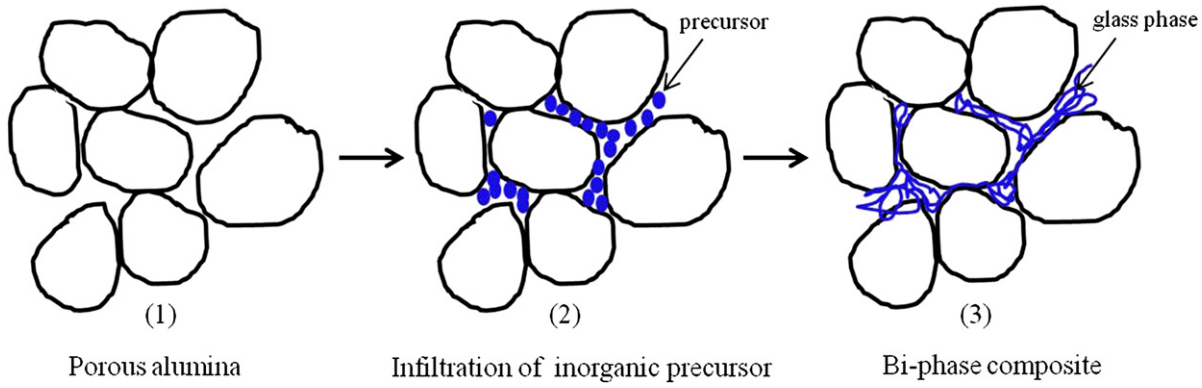

Fig. 1. Schematic diagram for fabricating a bi-composite.

Table 1
Formulations and conditions used to prepare bi-phase comosites.

Run number	Matrix	Inorganic binder precursor				Hardness
		TEOS (wt.% (mol%))	PDMS (wt.% (mol%))	NaOMe (wt.% (mol%))	Isobutyl alcohol (wt.% (mol%))	
Run-1	Al_2O_3		–			1.35 ± 0.10
Run-2-1		100 (0.47)	–	–	–	2.01 ± 0.19
Run-2-2		38 (0.18)	–	56 (1.5)	6 (0.08)	2.50 ± 0.25
Run-3-1		–	100 (0.184)	–	–	2.10 ± 0.14
Run-3-2		–	38 (0.07)	56 (1.5)	6 (0.08)	2.31 ± 0.24

Fisher Scientific, Waltham, MA, USA) and an X-ray diffractometer (XRD; Philips X-pert MPD, Model PW3040, Eindhoven, Netherlands). The microstructure of the bi-phase composite was observed using a scanning electron microscope (SEM, JEOL Model JSM-5610, Tokyo, Japan). The fracture strength of the samples was measured using a universal testing machine (UTM, Instron 5566, Instron Corp., Norwood, MA, USA) in the 4-point bending mode at a rate of 0.5 mm min^{-1}. The support spans of the low and upper noses were 2.5 cm and 1.0 cm, respectively. Tests were carried out at room temperature, and ten runs were performed to determine the standard deviation of the strength. The hardness value was measured using a Vickers indenter (HV-114, Mitutoyo Korea, Seoul,

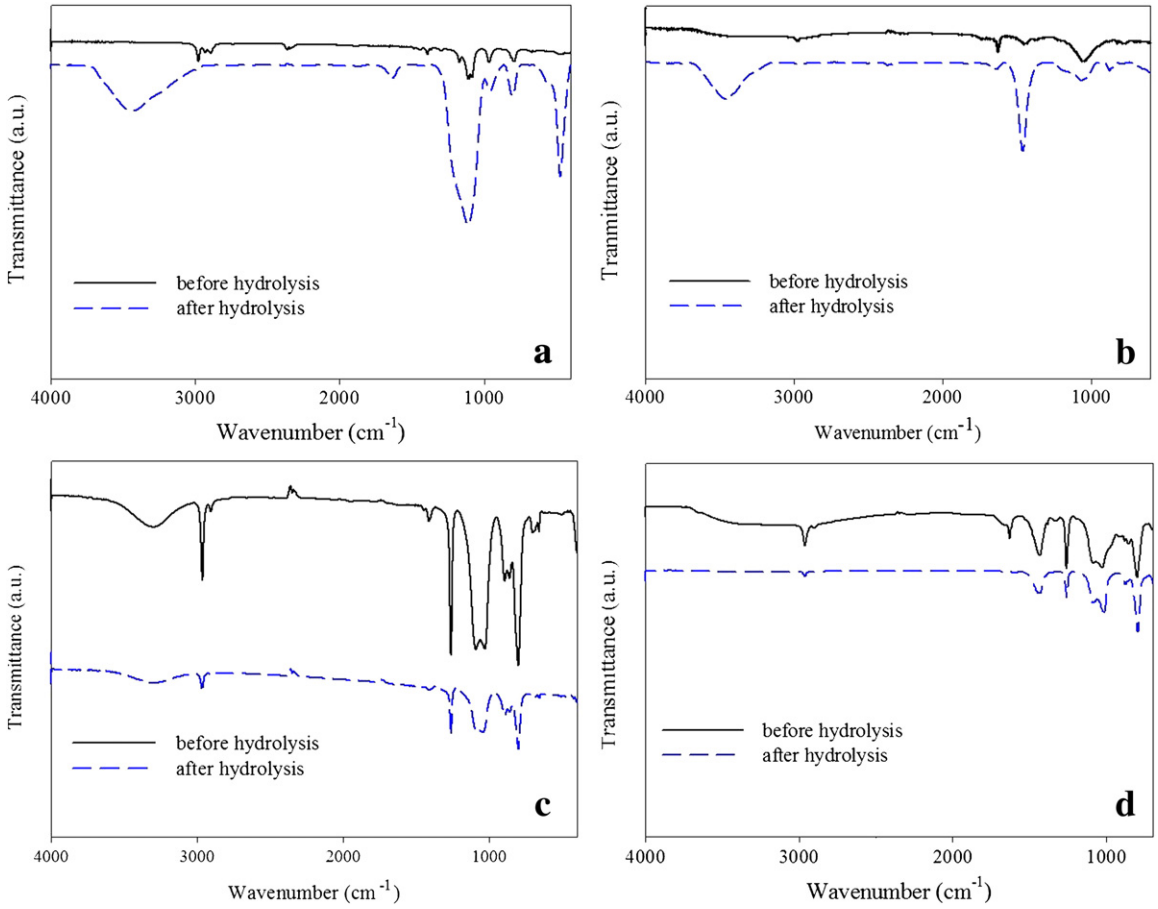

Fig. 2. FT-IR analysis of inorganic precursors before and after the hydrolysis reaction: (a) TEOS, (b) TEOS with NaOMe, (c) PDMS, and (d) PDMS with NaOMe.