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Fabrication of Fiber-Reinforced Porous Ceramics of Al_2O_3–Mullite and SiC–Mullite Systems

Byung-Hoon Kim & Yong-Han Na

Department of Inorganic Material Engineering, Chonnam, National University, 300 Yong Bong Dong, Bukku Kwang-Ju City, South Korea

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Abstract: Ceramic fibers and whiskers have been used for modification of toughness because they increase fracture energy due to their high elastic modulus and strength. The mechanism of this increase is well known to be pull-out, crack deflection and a bridging mechanism.

This study focused on the increase in toughness, strength and thermal shock resistance when using ceramic fibers in a gas filter for high temperatures. The basic systems studied were clay bonded SiC–mullite and Al_2O_3–mullite with carbon black (to improve filtering efficiency).

The Al_2O_3–mullite system showed lower porosity (27·45%) and higher strength (131 kg/cm^2) than the same system fired in air. The strength was higher than for the SiC–mullite system with the same treatment because of the high sintering rate.

Also, the hot strength at 1000°C was higher than for the SiC–mullite system. Strength degradation was only 14% and 5% after thermal shock tests.

Resistance to thermal shock was better than the Al_2O_3–mullite system owing to better thermal shock resistant raw materials (like SiC) and the fibrous microstructure. Both systems fired in air showed broader pore size distributions than when fired in a coke bed.

1 INTRODUCTION

Ceramics have wide applications in structural, heat resistant, chemical components because of their properties such as low density, stability at high temperature and chemical resistance. But their low toughness limits applications, so many researches to improve their toughness reliability have been undertaken.[1–3] Ceramic fibers and whiskers have been used for this purpose because they increase fracture energy due to their high elastic modulus and strength. The mechanism by which they increase fracture energy is well known to be pull-out, crack deflection and a bridging mechanism.[4]

This study focused on increasing toughness, strength and thermal spalling resistance using ceramic fibers in a porous body to use in gas filters for high temperature. The basic systems of this study were clay bonded SiC–mullite and Al_2O_3–mullite with carbon black (to improve filtering efficiency).

2 EXPERIMENTAL

The properties of the ceramic fiber (average length = 120 mm) are shown in Table 1. The chemical compositions of clay, Al_2O_3 and SiC are shown in Table 2, and their particle size distributions are plotted in Fig. 1.

The experimental procedure and batch composition are shown in Fig. 2 and Table 3, respectively. Dispersion of the ceramic fibers was thought to be the most important factor in this experimental procedure. Silica sol was used to disperse the ceramic fibers and to strengthen the body. The ceramic fibers were dispersed in water (100 times its weight) with silica sol and 10 wt% of 10% PVA aqueous solution with a high speed mixer at

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Table 1. Typical characteristics of ceramic fiber

Chemical composition (%)	Al_2O_3	47·3
	SiO_2	52·3
	Fe_2O_3	0·1
	$(Na_2O + K_2O)$	0·2
Maximum service temperature (°C)		1260
Fiber diameter (average) (μm)		5
Density (g/cm^3)		2·7
Tensile strength (kg/cm^2)		17250·0
Young's modulus (GPa)		105

Table 2. Chemical compositions of the raw materials

Component \ Raw materials	Clay	Alumina	SiC
Al_2O_3	55·8	95·0	—
SiO_2	29·8	1·0	—
SiC	—	—	98·7
Fe_2O_3	1·3	0·15	0·2
Others	$Na_2O + K_2O$ 0·8	TiO_2 2·7	Free C 0·5

380 rpm. This slip was dried at 100°C for 2 days and mixed with other raw materials and shaped at 500 kg/cm^2 pressure to a size of 4 × 8 cm. The specimens were fired in two atmospheres (air and coke bed) at 1250°C, because above this temperature the ceramic fibers become mullite by crystallization. Properties, such as porosity, strength (M.O.R, 3 point bending method) and resistance to thermal shock (strength change after 10 cycles when preheated to 1000°C in a furnace and cooled in air), were measured for both systems.

The microstructure was observed using SEM (JSM-840, Japan), the mineral phase was determined using XRD (Rigaku, Japan) and the pore size distribution was determined using a mercury porometer (Quanthachrome, USA).

3 RESULTS AND DISCUSSION

Non-oxide phases, like β-SiC, sialon, Si_2ON_2, were not detected at low firing temperature as

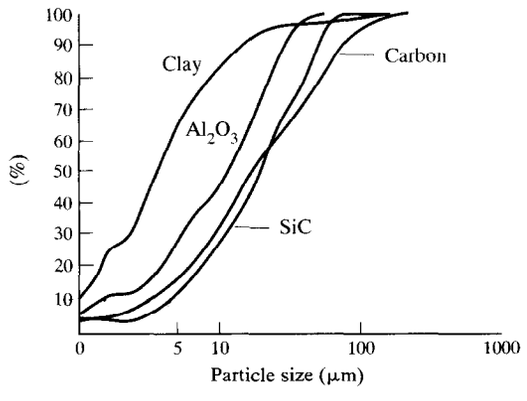

Fig. 1. Particle size distribution of raw materials.

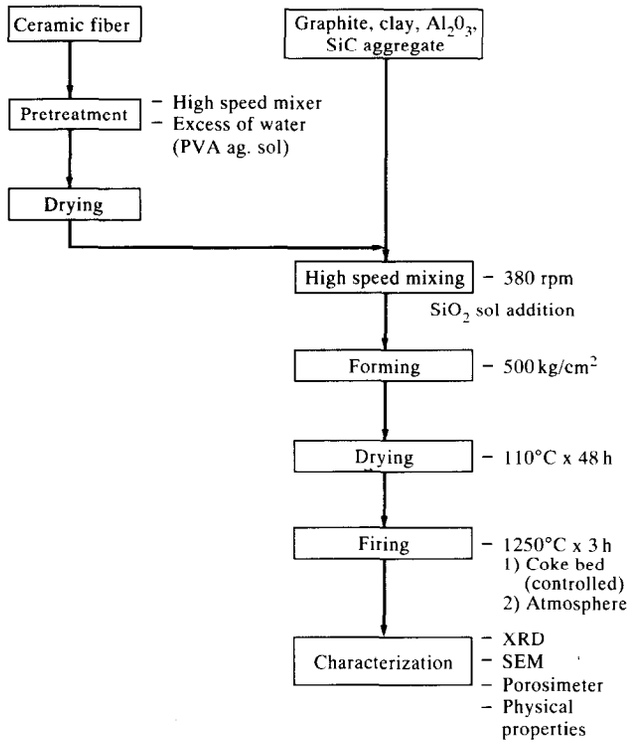

Fig. 2. Experimental procedure.

shown in the XRD patterns in Figs 3 and 4. Figure 3 shows that less mullite phase was synthesized after firing in an air atmosphere than in a coke bed atmosphere. Figure 4 shows the XRD patterns of the SiC–mullite system–less mullite phase was synthesized than in the Al_2O_3–mullite system (shown in Fig. 3) because of the lower content of Al_2O_3 and SiO_2 components. The Al_2O_3–mullite system fired in coke bed had a denser microstructure, shown in SEM photographs of Fig. 5. The properties of each system are shown in Table 4.

The Al_2O_3–mullite system showed lower porosity (27·45%) and higher strength (131 kg/cm^2) than the same system fired in air. The strength was higher than for the SiC–mullite system with the same treatment because of the high sintering rate. Also, the hot strength at 1000°C was higher than for the SiC–mullite system. Strength degradation was only 14% and 5%, respectively, after thermal

Table 3. Batch composition of the system

Raw materials \ System	Al_2O_3–mullite	SiC–mullite
Carbon	20	20
Clay	30	30
Al_2O_3	20	—
SiC	—	20
Ceramic fiber	10	10
SiO_2 sol	20	20

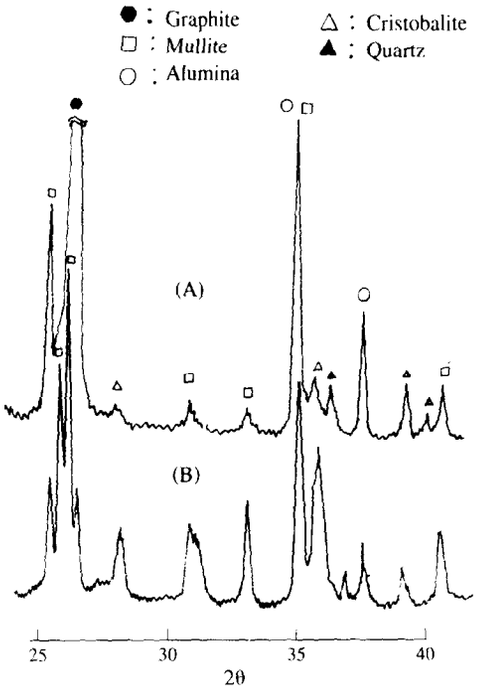

Fig. 3. XRD patterns of the Al_2O_3–mullite system after firing under (a) coke bed atmosphere and (b) air atmosphere.

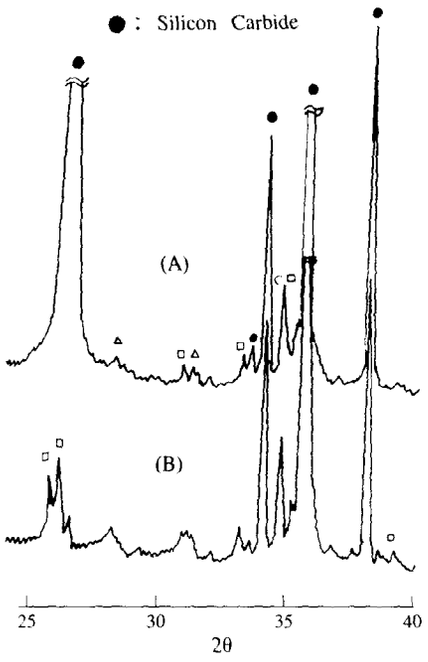

Fig. 4. XRD patterns of the SiC–mullite system after firing under (a) coke bed atmosphere and (b) air atmosphere.

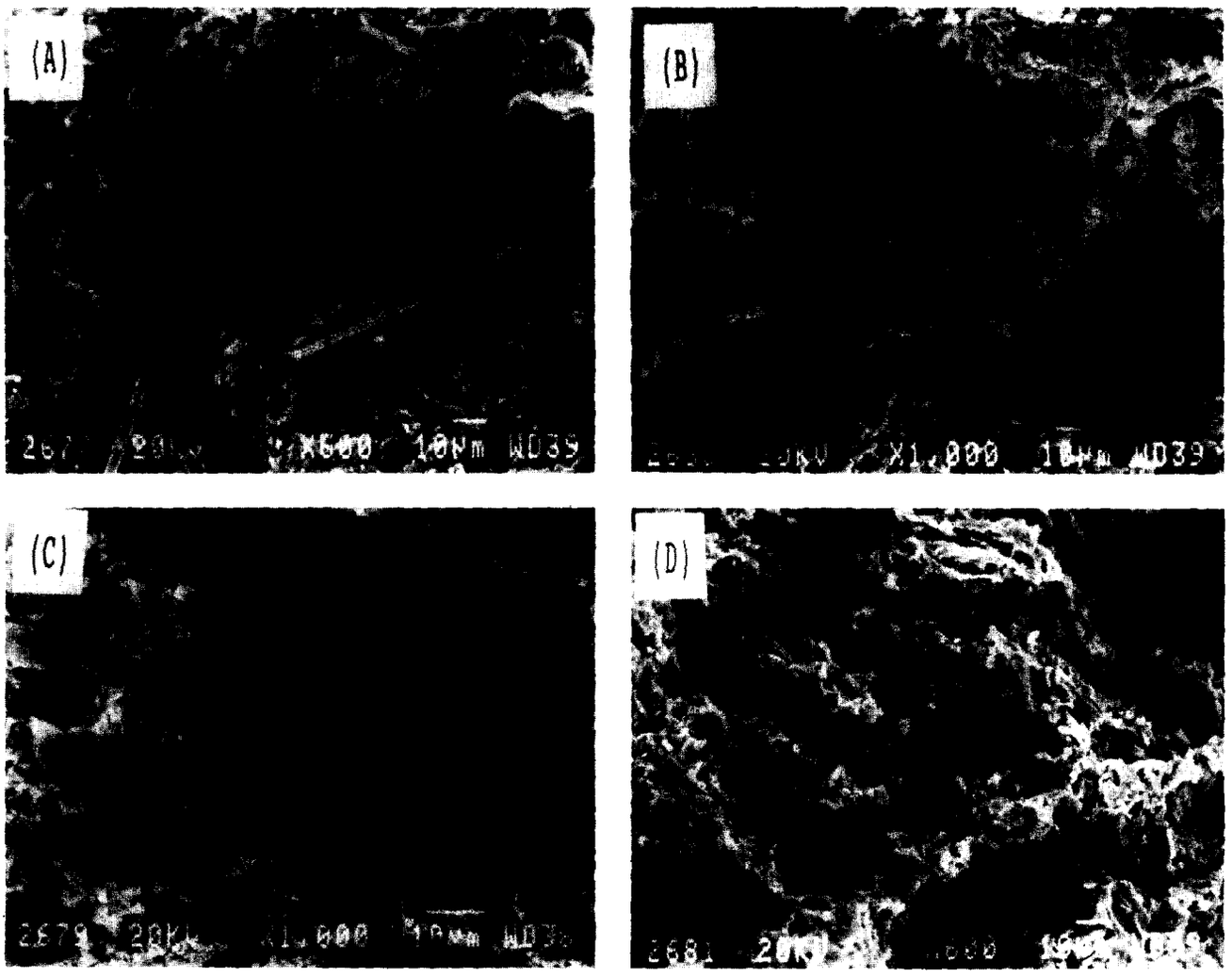

Fig. 5. SEM micrograph of the specimen fired at 1200°C for 3 h: Al_2O_3–mullite based system under (a) coke bed atmosphere and (b) air atmosphere; SiC–mullite based system under (c) coke bed atmosphere and (d) air atmosphere.

Table 4. Properties of the Al_2O_3–mullite and SiC–mullite systems after firing under coke bed atmosphere and air atmosphere

System		Al_2O_3–mullite		SiC–mullite	
Firing atmosphere		Coke bed	Air	Coke bed	Air
Properties					
Porosity (%)		27·45	38·40	33·33	54·56
Density (g/cm^3)		1·99	1·83	1·71	1·25
Apparent density (g/cm^2)		2·73	2·80	2·57	2·77
Bending strength (kg/cm^2)	Room temp.	131	112	81	73
	Hot M.O.R at 1000°C	—	68	—	21
Resistance to thermal spalling (kg/cm^3) (after 1000°C × 10 cycle air cooling)		—	96	—	69
Rate of strength degradation after thermal spalling test (%)		—	14	—	5

shock tests. The microstructure of the SiC–mullite system, shown in Fig 5, was more porous than that of the Al_2O_3–mullite system. In the SiC–mullite system ceramic fibers not bonded to the matrix could be found. The SiC–mullite system fired in air atmosphere has a very high porosity of 54·56% and strength of 73 kg/cm^2, which was reduced to 21 kg/cm^2 at 1000°C due to insufficient bonding between the ceramic fibers and the matrix. The resistance to thermal shock of SiC–mullite was better than that of the Al_2O_3–mullite system owing to the better thermal shock resistance of its raw materials (like SiC) and its fibrous microstructure.

In Fig. 6, macropores of ~10 μm in size in specimens (B) and (D) were induced by carbon oxidation and resulted in broad pore size distributions,

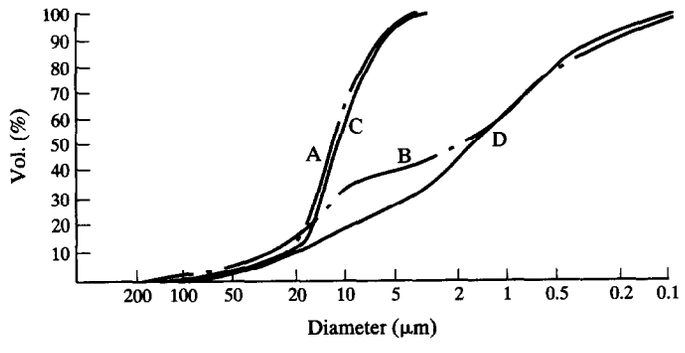

Fig. 6. Pore size distribution after firing at 1200°C under (a) coke bed atmosphere, (b) air atmosphere, (c) coke bed atmosphere and (d) air atmosphere. (—— - - —— Al_2O_3–mullite system; ———— SiC–mullite system).

compared to (A) and (C) which were fired in the coke bed atmosphere. Macropores, ranging from 10 to 20 μm, in the Al_2O_3–mullite system fired in air were induced from agglomeration of ceramic fibers due to irregular dispersion.

4 CONCLUSION

The purpose of this study was to improve the strength and toughness of clay bonded SiC–mullite and Al_2O_3–mullite ceramic filters for hot gas filtering. The Al_2O_3–mullite system showed good mechanical strength compared to the SiC–mullite system owing to high mullite formation and its microstructure. Both systems fired in air showed broader pore size distributions than when fired in coke bed. The SiC–mullite system was better in resistance to thermal shock than the Al_2O_3–mullite system.

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