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Preparation and properties of Al_2O_3–$MgAl_2O_4$ ceramic foams

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Abstract

In this work, a series of alumina (Al_2O_3)–magnesium aluminate ($MgAl_2O_4$) ceramic foams (AMCFs) were fabricated from powders of fused white corundum and magnesia (MgO) by a polymeric foam replication method combined with a reaction sintering process. The effects of MgO content and sintering temperature on the sintering properties and post-sintering properties of the AMCFs such as phase composition, appearance, microstructure, cold compressive strength and thermal shock resistance have been investigated. During the sintering process, MgO reacted with Al_2O_3 to form $MgAl_2O_4$ ceramic phase, which contributed to the improvement in sintering properties, cold compressive strength and thermal shock resistance of the AMCFs. The AMCFs doped with 8 wt% MgO and sintered at 1450 °C for 2 h displayed high cold compressive strength of 0.89 MPa, good sintering properties and thermal shock resistance. Although their apparent porosities were about 87%, the cold compressive strength of as-prepared foam was about 1.3 times higher than that of commercial Al_2O_3 foam.

Keywords: A. Sintering; B. Porosity; C. Strength; D. Al_2O_3; D. MgO

1. Introduction

Due to many attractive properties such as unique three-dimensional skeleton structure, low weight and thermal conductivity, high porosity and permeability, good corrosion resistance and chemical stability, ceramic foams have been found in many applications, including molten metals and hot gas filters, catalyst supports, thermal insulation, chemical sensors, biomedical materials [1–4], etc. There are a number of methods currently available to fabricate ceramic foams such as gelcasting [2,5–7], directly foaming [4,8,9], coating particle stabilized foam [10], high-temperature recrystallization [11] and polymeric foam replication [1,3,4,7,12–15], etc. Among these methods, the polymeric foam replication process is regarded as a promising one because it is cost-effective and easy to operate. This method involves i) soaking of polyurethane foams with slurries containing ceramic particles and some appropriate binders, ii) removal of excess slurries until a thin ceramic coating forms over the struts of reticulated structure, and iii) drying and pyrolysis of the organic matter and pressureless sintering at designed temperatures [4,15]. Many ceramic foams such as Ti_3AlC_2 [1], SiC [13,14], cordierite [3], Si_3N_4 [4], Al_2O_3 [4], ZrO_2 [4], MgO [4] and $Al_6Si_2O_{13}$ [4] have been produced by this process. In order to improve the mechanical properties such as strength and fracture toughness of the ceramic foams to meet the demand of structural applications, most common ways are to introduce reinforcing additives, including particles and fibers, to develop composite foams, such as Al_2O_3–SiC [15], Al_2O_3–ZrO_2 [16], Al_2O_3–Fe [17], Al_2O_3–V_2O_5 [18], SiC–Si_3N_4 [19], SiC–$Al_6Si_2O_{13}$ [20], SiC–TiC [21], and so on.

In this study, fused white corundum powder was chosen as the main raw material, and MgO was utilized as the reinforcing additive to fabricate the Al_2O_3–$MgAl_2O_4$ ceramic foams (AMCFs) by a polymeric foam replication method combined with the reaction sintering process. The AMCFs with various MgO contents ranging from 0 to 12 wt% were sintered at different temperatures from 1450 to 1550 °C for 2 h. The formation of $MgAl_2O_4$ during the sintering process and its effects on the phase composition, appearance, microstructure, linear shrinkage ratio,

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apparent porosity, cold compressive strength and thermal shock resistance of the AMCFs were investigated in detail. Introducing a small amount of MgO (8 wt%) could increase sintering properties, mechanical properties and thermal shock resistance of Al_2O_3 ceramic foam, and the cold compressive strength of as-prepared foam prepared at 1450 °C for 2 h was about 2.3 times as high as that of commercial Al_2O_3 ceramic foam.

2. Experimental procedure

2.1. Raw materials

In the present work, commercially available powders of fused white corundum (Al_2O_3 content more than 99.55 wt%, median diameter of 0.85 μm) and magnesia (MgO content more than 98.0 wt%, median diameter of 1.92 μm) were used as the raw materials. Introduction of MgO serves to form $MgAl_2O_4$ reinforcement phase via the reaction between MgO and Al_2O_3.

Some chemical reagents including kaolinite (median diameter of 0.63 μm, its main compositions includes Al_2O_3 and SiO_2), sodium carboxymethylcellulose (CMC), sodium polyacrylate (PAAS), polyving akohol (POVAL), tributyl phosphate (TBP) and sodium hydroxide (NaOH) were also chosen as additives. Kaolinite with good plasticity and associativity can reduce the deformation fracture of ceramic slurries in the extruding process, and increase the strength of dried ceramic slurries. CMC was chosen as surface modification and rheological agents, PAAS, POVAL and TBP were used as a dispersing agent, for high temperature adhesive bonding and as a defoamer, respectively.

Polymeric foam (25 PPI, pores per inch) was used as the replica to produce the AMCFs. NaOH solution was used to pretreat the polymeric foam to improve the surface roughness and further increase the loading of slurry.

2.2. Pretreatment of polymeric sponge

The polymeric foam was tailored to size of $(50 \pm 1\,\text{mm}) \times (50 \pm 1\,\text{mm}) \times (10 \pm 1\,\text{mm})$, and then it was put into a NaOH solution (6.5 mol L^{-1}) and heated at 60 °C for 8 h. Next it was fully washed and dried in air. The surface modification treatment was further conducted in the CMC solution (1 wt%) for 12 h to remove the surface residual, and the polymeric foam was further dried fully in air.

2.3. Preparation of ceramic slurry

The qualities of as-prepared ceramic slurry, such as dispersion, slurry stability and solid content, are of great importance for preparing high performance ceramic [22]. In this study, the Al_2O_3–$MgAl_2O_4$ ceramic slurries (solid content of 50 wt%) were prepared by the following procedures. Firstly, fused white corundum powder, magnesia, kaolinite and other additives were weighed according to the raw materials ratios listed in Table 1. Secondly, the POVAL was heated to dissolve in a constant water bath pan, and it was poured into a beaker containing a certain amount of deionized water. Then the CMC and PAAS were added to the beaker to dissolve, and the TBP was also added to eliminate the bubbles generated during the solution preparation. Thirdly, the fused white corundum powder, magnesia and kaolinite were added into the as-prepared solution, and they were stirred for 10 h to form ceramic slurry. Finally, the ceramic slurry was milled for 12 h.

Table 1
Raw materials ratios for preparing ceramic slurry (wt%).

Sample nos.	Fused white corundum powder	Magnesia	Kaolinite	CMC, PAAS, POVAL and TBP
M0	87	0	10	3
M4	83	4	10	3
M8	79	8	10	3
M12	75	12	10	3

2.4. Preparation and sintering of green bodies

The polymeric foams after pretreatment were soaked into the as-prepared ceramic slurries (solid content of 50 wt%), and the excessive slurry was extruded. The as-prepared green bodies were fully dried for 12 h and sintered at 1450, 1500 and 1550 °C for 2 h.

2.5. Characterization of sample

2.5.1. XRD and SEM analyses

The phase compositions of the AMCFs were characterized by X-ray powder diffraction (XRD, Cu Kα radiation, 30 kV and 30 mA). The powders were obtained from AMCFs via crushing, fully milling and sieving (320 mesh), and their microstructures were observed by a scanning electron microscope (SEM).

2.5.2. Sintering properties

In the present work, sintering properties, including linear shrinkage ratio and apparent porosity, were studied. The linear shrinkage ratio was calculated according to Eq. (1), and the apparent porosity was measured in water under vacuum using Archimedes' principle and it was calculated according to Eq. (2) [23].

$$\Delta L = \frac{L_0 - L_1}{L_0} \times 100\% \quad (1)$$

$$P_a = \frac{m_3 - m_1}{m_3 - m_2} \times 100\% \quad (2)$$

where ΔL and P_a are the linear shrinkage ratios (%) and apparent porosity (%) of the sintered samples, L_0 and L_1 are the height of the samples before and after sintering (mm), respectively, m_1 is the mass of the dried sample in air (g), m_2 is the mass of the sample in water (g), and m_3 is the mass of the sample with free bubbles on the surface (g).

2.5.3. Cold compressive strength

The cold compressive strength was measured using a CMT5105 type universal tester with loading rate of 0.5 mm