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Fabrication and Characterization of SiO_2·RO/SUS Porous Composite Body

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ABSTRACT

Porous materials exhibit various unique physical and mechanical properties, such as low density, low thermal conductivity, high temperature stability, high surface area and high permeability. Besides that, there is strong demand for practical usage of porous composites that can endure severe environments such as high temperature and highly corrosive atmospheres. In this study, porous glass/metal composites of SiO_2·RO/SUS system that can be served as a soot particulate filter have been fabricated through sponge replica method. Microstructure, compression strength, microwave heating ability and heat transfer coefficient of the porous composite were evaluated. Compression strength measurements depicted that, strength of the porous glass composite body was dependent on the stainless steel volume fraction in the samples. No significant effect of gas flow rate on the heating ability was observed. Owing to difference in microwave selectivity and absorbability, samples with higher stainless steel fraction exhibited better heating as compared with those in lower stainless steel fraction.

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INTRODUCTION

Porous materials which exhibit various unique physical and mechanical properties, such as low density, low thermal conductivity, high temperature stability, high surface area and high permeability (Innocentini *et al*., 1999)have attracted a great attention as new kinds of materials with a wide range of technical and engineering applications.There is a strong demand for practical usage of porous composites that can endure severe environments such as high temperature and highly corrosive atmospheres (Zhao *et al*., 2005). Such applications include, for example, high temperature thermal insulation, support for catalytic reactions, filtration of particulates from diesel engine exhaust gases and filtration of hot corrosive gases in various industrial processes (Schefler and Colombo, 2005).

Various process routes have been applied to fabricate the porous ceramic composites, including the combustion synthesis (CS) method (Dong at al., 2004; Menon *et al*., 2004: Lee *et al*., 2011), sponge replica method (SRM) (Dressler, 2009: Angela *et al*., 2009), foaming agent (Sepulveda and Binner, 1999) or space holder method (Yu *et al*., 2008). Out of these, CS and sponge replica method are two of most attractive techniques. The CS method uses the exothermic reaction of a starting compound, ignited to its ignition temperature to spontaneously transform into product. On the other hand, polyurethane (PU) sponge replica methodwas patented by Schwartzwalder and Somers(1963) which utilize polymeric sponge as the structure template or so called precursor to obtained open cell porous ceramic structures with a controllable pore size, interconnected pores and desired geometry.

Although the fabrication of porous composite of various materials have been widely reported in the literature, resources on fabricating of porous composite consisting of well distributed metal particle within the ceramic and/ or glass matrix which can be heated up easily by utilizing microwave power was very limited.In the present work, porous glass/metal composites consisting of well distributed stainless steel particles that can be served as a diesel particulate filter have been fabricated. Particulate matter generated from diesel engine can be trapped on the filter and subsequently combusted during the regeneration step. As a consequence,characterization of the materials' properties is essential and important to determine the applicability of the materials to its corresponding environment. Besides that, it is also important to ensure the filter heating capability, especially during cold-start phase of the engine, whereas exhaust gas temperature was

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insufficient for regeneration. Hence, active regeneration utilizing microwave power was introduced to overcome this problem. Microwave heating were conducted to study the heating ability of the fabricated filter materials.

MATERIALS AND METHODS

Preparation of porous glass composite body:

Commercial polyurethane (PU) sponge, with a cell size ~ 45 ppi (pore per inch), were chosen in this study. SiO_2·RO glass powder (D50 = 5 µm) from AGC (Asahi Glass, Japan) and 303L stainless steel powder (140 mesh) from Alfa Aesar (Wall Hill, MA, United State) were used as the raw materials. PU sponges are cut into cylindrical shape with an approximate diameter of 25 mm. SiO_2·RO glass powder was blended with 10 – 30 vol% stainless steel powder in an agitate mortal to obtained a homogeneous mixture.

The slurries are prepared by dispersing the powder mixtures with distilled water without any further additives to give a suspension with approximately 40 wt% solids loading. According to Sifontes *et al*. (2010), this weight allowed obtaining adequate viscosities of slurries and proper mechanical strength for ceramic foam. PU sponge which had been cut into cylinder was immersed into this suspension and was compressed while submerged in order to fill all the pores. At the same time, stirring was carrying out to avoid segregation in the slurries due to powder mixtures density different. After a minute, the impregnated sponge was removed from the slurries and excess slurries were removing by blowing with air followed by drying at 60 ^oC for 12 h in a convection oven. Finally, the coated cylinder was heat-treated to burn out the sponge and sintered at 940 ^oC with a heating rate of 1 ^oC/ min for 1.5 hrs holding time in air.

Physical and Mechanical Properties Evaluation:

In order to study themicrostructure of the porous bodies, a scanning electron microscope equipped with a Field-Emission Gun (FEG-SEM), Philips XL-30FEG was used. Samples are prepared by attaching the polished porous body to an aluminum sample holder with conductive tape. Carbon coating was performed to prevent charging of the samples. Compression test was carrying out using a Shimazu Autograph AG-X universal testing machine with a crosshead speed of 0.5 mm/min at ambient temperature. Microwave heating ability was conducted in the H maximum field in a 2.45 GHz, 2 kW, single mode applicator with an input power of 300 – 400 W. Detail of the experimental set-up is given elsewhere (Lee *et al*., 2011). Heat transfer coefficient of the heated sample was then calculated by employing Newton's law of cooling; referring to the recorded temperature after microwave power is turned off.

RESULTS AND DISCUSSION

Microstructure evaluation:

Scanning electron microscope (SEM) micrographs of the sintered porous glass-stainless steel composite is shown in Figure 1. It can be observed that the sintered composite sample maintained the pore structure of the original PU template. The skeletons of cells are uniform and free of defects. The pores are mostly interconnected, some macrospores on the skeletons surface can also be observed. Porosity around 65-70% can be obtained.

Figure 2 shows the microstructure of the skeleton surface with different stainless steel fraction. The SiO_2·RO matrix had a grey appearance whereas the stainless steel particles were in light grey. No obvious remnants of the PU sponge were detectable. Stainless steel particles were well dispersed and distributed within the glass matrix. Increase in the stainless steel fraction led to higher allocation of stainless steel particles at the skeleton. Some particles were interconnected to from a cluster, but most of the particles were well distributed and isolated within the skeleton. This microstructure effect was generated during the impregnation process. Since the stainless steel powder has a higher density compared to SiO_2·RO glass powder, segregation might occur in the slurries which could draw in non-homogeneity that will influence the uniformity of the stainless steel particle distribution within the glass matrix. Hence, uninterrupted stirring is needed during the impregnation step, to minimize the occurrence of segregation.

Compression strength:

Compression strength has been evaluated for the porous composites of different stainless steel volume fraction, fabricated at the same sintering temperature. As depicted in Fig. 3, compressive strength of samples decreased with increasing of stainless steel volume fraction in the samples. Since, stainless steel powder and glass power has a large particle size difference, 5 µm and 105 µm respectively, and higher sintering point of stainless steel, grains of these two powder were loosely stacked. Beside that, small openings within grains may be occurred.

For the sample with lower stainless steel volume fraction, bonding within the glass powder grains itself resulting from neck growing give a higher strength between particles which result in higher strength of the

sample because the fracture of porous foam mainly happen at the particles boundary. As the stainless steel volume fraction increased, bonding among grains become weaker, thus, lowering the material strength. Hence, the increase of grains bonding area has an important effect on the improvemnet of sample strength. This can be done by increasing the sintering temperature as indicated in Fig. 3.

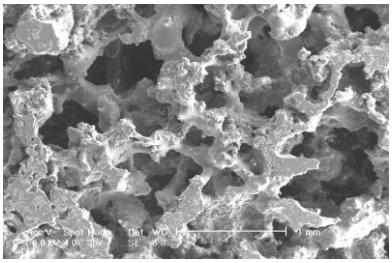

Fig. 1: SEM micrograph of the sintered glass-stainless steel porous composite.

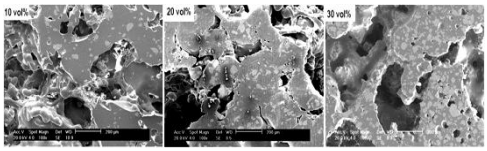

Fig. 2: SEM micrograph of sintered glass-stainless steel porous composite with different stainless steel fraction.

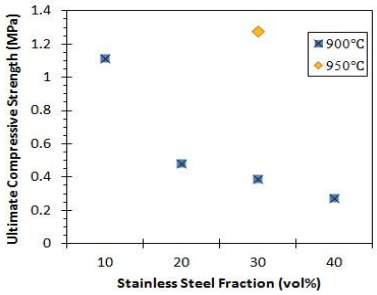

Fig. 3: Influence of stainless steel fraction and temperature on the compressive strength.

Microwave heating ability:

Heating ability of the SiO_2·RO/SUS porous composite in H maximum field is illustrated in Fig. 4. Temperature of 600 ^oC was chosen as the target, referring to the combustion temperature of diesel soot, as reported by Xu *et al*. (2000). It is clearly shown that, heating rate of these porous composites was fully associated with the amount of stainless steel particles in the samples. Regardless of microwave input power, the heating rate increased with increasing of stainless steel fraction in the samples. Samples with higher metal fraction can be heated up rapidly compared with those of lower metal fraction. At a lower input power, heating of samples with lower stainless steel fraction were unable to reach the target temperature. Hence, those data were not included in the figure. Owing to difference in microwave absorbability and heating selectivity, these allow the microwave to penetrate through the glass matrix, heated up the metal part and subsequently transferred the heat energy throughout the whole samples. Since, stainless steel particles in the samples played an important role as the microwave absorber, considerable amount of stainless steel particles are needed to achieve a higher heating rate

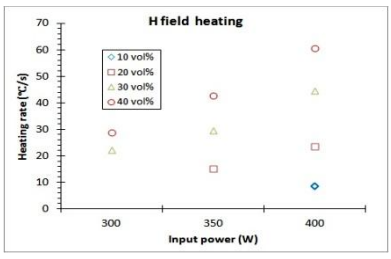

Fig. 4: Effect of stainless steel volume fraction and MW input power on the heating rate of SiO_2·RO/SUS. porous composite.

Heat transfer coefficient:

Heat transfer coefficient (hs) of the samples, obtained by employing Newton's Law of Cooling is shown in Fig. 5. It was calculated based on the temperature difference after the MW power was turned off. During the heating, it is assumed that, no energy dissipation was accured, all the energy was absorbed by the sample and convert into heat. Thus, heat transfer characterization is carrying out after the MW power was turned off, in which heat from the sample is allowed to dissipate to the ambient for the cooling purpose. Clearly seen form the figure, for a higher input power, heat transfer coefficient of the samples was slightly higher due to higher final heating temperature.

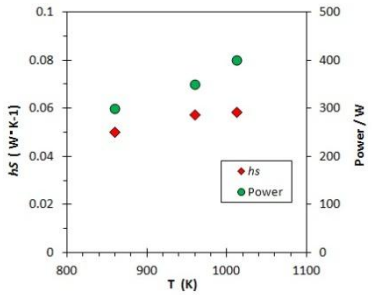

Fig. 5: Input power dependent heat transfer coeefficient.

Conclusion:

Porous glass-stainless steel composite (SiO_2·RO/SUS) with interconnected pores and high porosity (65-70%) which consist of well dispersed and distributed stainless steel particles within the SiO_2·RO glass matrix were obtained by the sponge replica method. Samples with higher stainless steel volume fraction shows a better and faster heating by microwave. Owing to difference in microwave absorbability and heating selectivity, heating rate of the porous samples increased with the increase of stainless steel fraction. However, strength of the porous sample exhibit a reversed behaviour, decreased with the increase of stainless steel fraction. A higher sintering temperature is recommended for the achievement of a higher strength.

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