The Effect of Nickel Oxide (NiO) Compositions in Consolidation of Silica-Nickel Oxide (SiO_2-NiO) Foams Using Replication Method

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ABSTRACT

Ceramic foams contribute to wide range of engineering application such as filter, thermal insulator, biomedical implant and catalytic reaction. There are many methods can be used found to be fabricating ceramic foams, however, replication method is the most effective method in producing ceramic foams. Fabrication of Silicon Dioxide (SiO_2) foam with the addition of Nickel Oxide (NiO) by using replication method at different compositions was studied. The composition of SiO_2 applied was at 55 wt. % while the compositions of NiO were 0, 5 and 7 wt. %. Polyethylene Glycol (PEG) and Carboxymethyl Cellulose (CMC) were used as binders while Polyurethane (PU) foam in cylindrical form was applied as space holder. Upon impregnation of the PU foams with SiO_2-NiO slurries, drying process was performed at 100℃ for 24 hours. Then, the sample was further sintered at 600℃ for an hour and 1300℃ for two hours with heating rate at 2℃/min and natural cooling in the furnace. Morphological analyses were performed by using Scanning Electron

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Microscopy (SEM). Density and porosity of the fabricated foams were performed as according to ASTM C20. The compressive strength analyses of foams were obtained based on ASTM C733-88. The morphological analyses showed that the pore size of the SiO_2-7wt% NiO (248 μm) smaller than SiO_2-5wt% NiO (272 μm). The increment of density was affected by the increase of weight percentage composition of NiO. Additionally, it was also depicted from compressive strength analysis that the higher NiO composition increased the compressive strength of the SiO_2-NiO foams. Thus, it is evidently clear from the findings that the replication method is indeed suitable to be applied for SiO_2-NiO porous foam with good mechanical strength.

Keywords: *Ceramic foam, Silica, Nickel Oxide, Replication method.*

Introduction

Porous body also known as foams is an important classification of lightweight cellular engineering material structures [1,2]. The classification depends on whether the individual or not individual cells have a solid face. It is called open cell if the solid face of the foam is found only in cell edges. If the faces of cells are present, it is called as closed-cell foam and the individual cells are separated from each other. An open cell structure of ceramic foam has a low mass, low density and, low thermal conductivity but different in terms of permeability that is higher permeability [3,4]. The specific surface area of foams normally has porosity percentage of no less than 50% while the pore sizes are usually between $50\mu m$ and $1000\mu m$ in diameter [5,6]. There are possibilities that the foam can be partly open and closed which depends on the fabrication technique applied.

Silica known as silicon dioxide (SiO_2) is commonly used in high-temperature structural applications as owing to its low density, high melting point, and excellent thermal shock resistance [7]. It is also widely available in forms of raw materials on the earth surface and therefore is a fundamental constituent of a wide range of ceramic products and glasses [8].

There are several methods that can be used for the fabrication of ceramic foam including replication method [9], high-temperature recrystallization [10], direct foaming method [11] and gel-casting of foam [12]. The replication method is the most suitable method for ceramic foam fabrication. This is due to ability of controlling the pore size, which can be achieved by the usage of polymeric sponge as template or space holder [13,14]. The polymeric sponge method produces open cell structure by immersing a polymeric sponge (*i.e.* polyurethane and polyester) with ceramic slurry with the composition desired. Sintering process afterwards would be

required as to burn out the polymeric sponge and also as to densify and strengthen the structure.

Nickel Oxide (NiO) addition to SiO_2 is possible to be applied in sensing devices, solar energy conversion, photovoltaic devices, electrochromic films, and as a catalyst material for wastewater pollution, due to the presence of excess oxygen [15]. Besides, recent developments in the field of using NiO have led to a renewed interest in producing ceramic foam catalyst with excellent abilities for tar reforming [16]. In particular, the effects of NiO composition in the fabrication of SiO_2-NiO foam by using replication method are highlighted and also show its characteristics by physical and mechanical testing analysis. Thus, the study highlights the fabrication of SiO_2-NiO foam by application of replication method. Moreover, the properties of the SiO_2-NiO foams with varied NiO addition were also explained.

Experimental Setup

Raw materials

Raw material includes the SiO_2 (Mechatech Solution, Malaysia) and NiO (Sigma-Aldrich, Germany) were the main component of raw material. Carboxymethyl Cellulose (CMC) (Multifilla, Malaysia) was used as thickener agent while Polyethylene Glycol-6000 grade (PEG) (United Kingdom) was chosen as binder. PU foam was applied as space holder or template in the form of cylindrical shape with size of 12.5 mm diameter and 26.0 mm length.

Fabrication of SiO_2-NiO by replication method

The mixing process of raw materials as specified in Table 1 was performed using a mechanical stirrer (RWA 20 digital, IKA®, Germany) at 500 rpm. The composition of all the raw materials is also shown in Table 1.

Table 1: The composition of raw materials

SiO_2 (wt. %)	NiO (wt. %)	PEG (wt. %)	CMC (wt. %)	Distilled water (wt. %)
55	0	2.5	2.5	40
	5			35
	7			33

The process started by mixing distilled water and PEG for 15 minutes. Then, CMC was added and stirred until the homogenous mixtures are obtained. Next, the SiO_2 powder was added and continued to stir for an hour. NiO was then added and stirring continuously for another one hour to produce completely homogenous mixtures.

The PU foams in cylindrical shape were immersed into the slurry solution for soaking and pressed slowly for five minutes to remove excess slurry. Immersions were performed for three times to ensure the mixture are able to absorb over the whole space and pores in the PU foams. The drying process was afterwards conducted as to remove water from the slurry by using drying oven (UM500, Memmert, Germany) at the temperature of 100℃ for 24 hours. Sintering process was conducted at a temperature of 600℃ for one hour to ensure that the PU foam was burnt completely and later increased to 1300℃ in a muffle furnace (Protherm, Turkey).

SiO_2-NiO foam characterisation

The characterisations involved in this study were morphology analyses, density and porosity analyses and compression strength analyses. The morphology analyses were performed by using Scanning Electron Microscopy (SEM) (Hitachi, Japan).

The density and porosity analyses were performed based on ASTM C20 which is standard for apparent porosity, water absorption, apparent specific gravity and bulk density. The test was conducted by using (Mettler Toledo, Switzerland). The calculation for density and porosity analyses had shown in the equation (1) and (2).

$$\text{Density} = \frac{W_d}{W_w - W_s} \quad (1)$$

Where,
W_d = Dry weight, g
W_w = Wet weight, g
W_s = Saturated weight, g

$$\text{Porosity percentage} = \frac{W_w - W_d}{W_w - W_s} \times 100\% \quad (2)$$

The mechanical properties of the SiO_2-NiO foams was performed in terms of the compressive strength test based on ASTM C733-88 by using Autograph AG-10kN ISMS Universal Testing Machine (Shimadzu, North America). The compressive strength was calculated in the equation (3).

$$\text{Compressive strength, S} = P \Big/ \left(\frac{\pi d^2}{4}\right) \quad (3)$$

Where,
P = Force, N
d = Diameter of sample, mm

Results and Discussion

Foam morphology analysis

The microstructure of SiO_2 foams added with 0, 5 and 7 wt. % of NiO can be seen in **Figure 1**. The addition of 7% NiO produced the smaller pore sizes compared to the microstructure of SiO_2 foam without any NiO addition. This may be attributed to the increment of slurry viscosity due to NiO addition. This was observed by the slurry thickening increment during the mixing process. This eventually leads to formation of the strong bonds in between the SiO_2 and NiO particles development during sintering which later caused smaller size of pores formation.

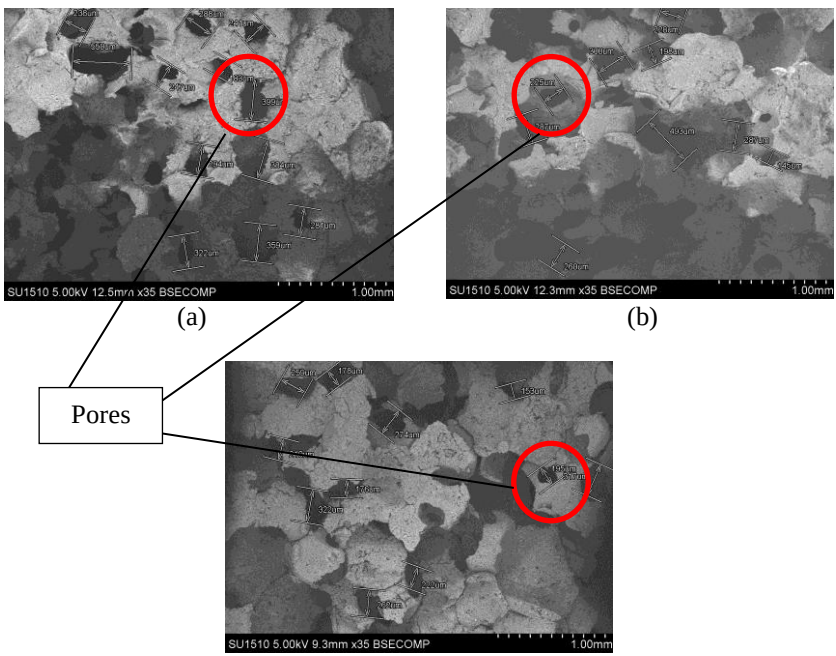


Figure 1: Microstructure of Silica foam with temperature 1300℃ (a) without NiO, (b) 5% NiO and (c) 7% NiO

Figure 2 showed the average pore size as according to different percentages of SiO_2 foams with and without NiO addition. Meanwhile, the increased addition of NiO would lower the pore sizes produced due to the increased viscosity of the SiO_2-NiO slurry which further impedes the absorbance of slurry in PU foams. This was similarly reported by Fadli

(2011) which observes that low viscosity of slurry increased the pore size [17].

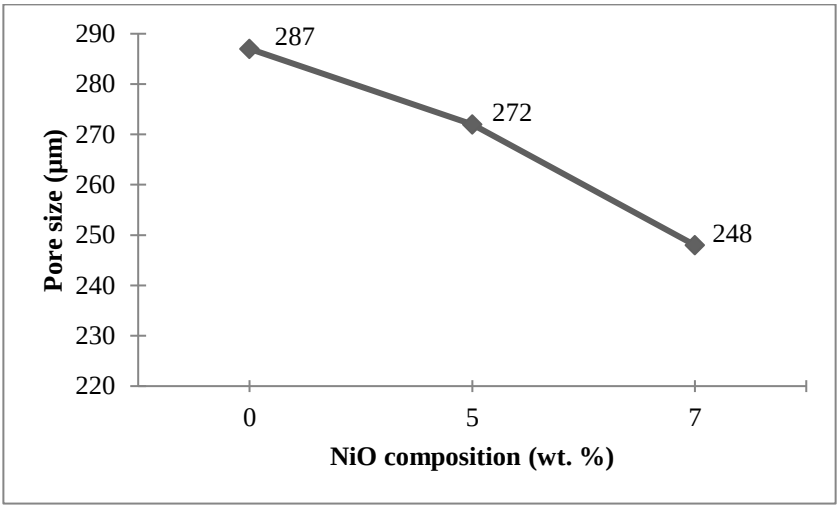
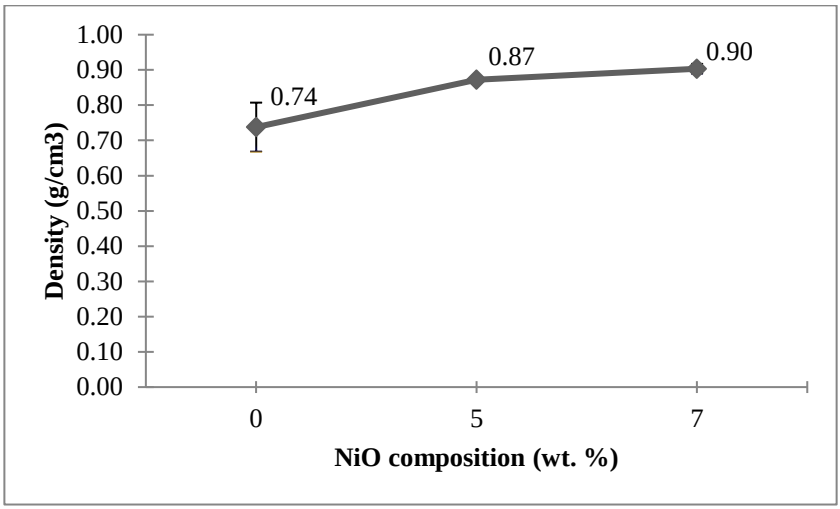


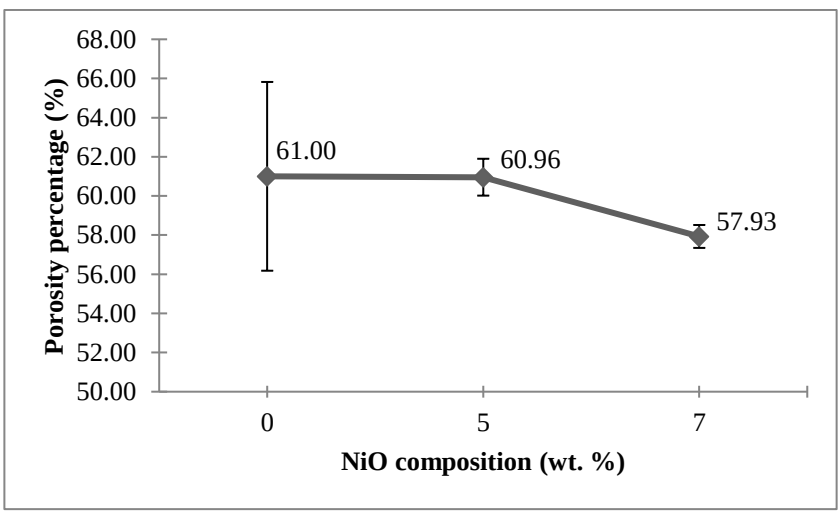
Figure 2: Average pore size of SiO_2 foam as a function of increasing of NiO compositions

Density and porosity analysis

Figure 3 (a) presents an overview of the average density of SiO_2 foam with different NiO compositions. It was shown that the density increased at the highest NiO addition. In contrary, the porosity percentage of SiO_2 foam was decreased by the increased NiO composition. Thus, it is clear that NiO composition affects density of foam in which increased the density. It is because the increased of density slurry will increase the densities of solid bodies [18].



(a)



(b)

Figure 3: (a) Density and (b) porosity percentage of SiO_2 foam at different NiO compositions

Compressive strength analysis

The effect of NiO addition to SiO_2 foam was also assessed in terms of the compressive strength. **Figure 5** showed the relations of compressive strength average to the NiO compositions. From the result, the compressive strength

was found to increase by the increased of NiO addition. This has been observed similar to work reported by Nor *et al*., (2008) in which when the composition increases, the compressive strength would also increase [19].

Theoretically, the strength of SiO_2 foam depends on the structure acting as strut loads. Moreover, the size and number of porosity also affect the compressive strength of a sample [20]. With respect to the density and porosity analysis, it was found that the increased NiO addition contributed to the decrement of porosity and increment of the density of the SiO_2 foam. In addition, the previous study by Baharom *et al*., (2017) indicated the increase in compressive strength can be interpreted as the effect of the slurry viscosity and density [21]. The highly porous material with high porosity is weak and easy to break.

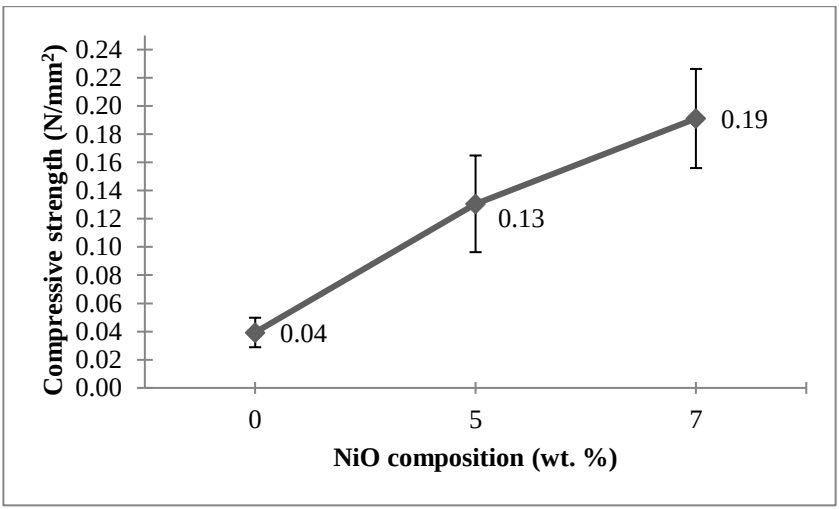


Figure 4: Compressive strength of SiO_2 foam at different NiO compositions

Conclusion

The SiO_2-NiO foam fabrication by replication method with different NiO composition was found to be successful. NiO compositions added were found to affect the physical and mechanical properties of the SiO_2 foam. The morphology analyses showed that the pore size of SiO_2 foam without the addition of NiO is larger than the SiO_2 foam with the addition of NiO. The density was increased by the increased NiO composition while the percentage of porosity was decreased by the increased of NiO composition. It is worth noting that even though the porosity percentage at 7% NiO composition highest was decreased, it was still in the range of 50% which indicated a

good percentage of porosity for foams. Moreover, the compressive strength analyses also showed that the compressive strength was increased by the increased of NiO. Thus it can be concluded that the replication method is indeed feasible to fabricate SiO_2-NiO foams with up to 7 wt% addition of NiO.

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