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The Effect of Sintering Temperature on Silica Derived from Rice Husk Ash - Nickel Oxide (SiO2-NiO) Foam Fabrication via Slurry Technique

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Abstract. Fabrication of ceramic foam has been an interesting study in the field of a porous material due to the excellent mechanical and physical properties. This study presents an approach for the fabrication of Silica (SiO2) derived from rice husk ash (RHA) and Nickel Oxide (NiO) foams using the slurry technique, highlighting the sintering temperature affects. Polyvinyl alcohol (PVA) was used as binder and Polyurethane (PU) foam was applied as the space holder. The composition of SiO2 applied in this study was 20wt%. The PU foams dipped into SiO2-NiO slurry were dried and further sintered at three different sintering temperatures of 1050˚C, 1150˚C and 1250˚C. The morphologies of SiO2-NiO foams were observed by using the Scanning Electron Microscopy (SEM) and physical properties were determined by using Archimedes method for investigating the total porosity and bulk density. The identification of phases of SiO2-NiO foams were analysed by using X-Ray Diffraction (XRD). The XRD analyses indicated that there were only SiO2 and NiO present and no additional phases detected after sintering which implied the compatibility of SiO2 derived from RHA and NiO even temperatures up to 1250˚C. The density values of the SiO2-NiO foams were found to increase with increasing of sintering temperature. The densities were found to be in the range 0.5239g/cm³ to 0.6210g/cm³ and the percentage of the foams porosity were in the range of 69.71% to 75.19%. Thus it is concluded that the slurry technique is found to be successful to fabricate the SiO2 as derived from RHA and NiO foams. The sintering temperatures was found to affect the SiO2-NiO foams in terms of the density and porosity of the foam.

1. Introduction

Ceramic foams are known as porous and brittle materials with pores of commonly open cell, closed cell and interconnect porosity[1–3]. Ceramic foams have been widely applied in many fields including thermal insulation, catalyst support, filter and medical implant thanks to their cellular microstructure[4]. Open cell foams are applied in metal melt filtration and also as filter in diesel engine exhaust and industrial hot gases [5–8]. Moreover, the closed cells in ceramic foam usually used in wide range of application for thermal insulation and fire protection. It is known that the ceramic foam with high porosities up to 95% exhibit a
Reticulate (open pores)[6]. The porosity percentage, pore size distribution, and pore morphology will affect most the material’s properties. As an example, the presence of cell walls influences both permeability and strength, while compositional purity will affect the chemical and oxidation resistance, as well as high-temperature creep, electrical resistivity and thermal properties[9]. Several techniques have been developed for the production of ceramic foams; which include replication technique, sacrificial templating and direct foaming[10–12]. The replication technique consists in the impregnation of a natural or a synthetic template polymer foam with a ceramic suspension [11]. 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 PU 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 pressure less sintering at designed temperatures[13].

Polyurethane (PU) foam used as a space holder or skeleton in fabrication of ceramic foam. Polyurethanes are the most versatile materials, which can be formed into rigid and flexible, foams, elastomers, adhesives, sealants, etc and are now widely used widely utilized as engineering materials in many industries [32]. PU foam are stiffness, hardness and density value was high. Among other advantage of using PU foam is a form of hollow structure and interconnect pores.

Silica (SiO₂) is an oxide of the element of silicon which is the second most abundant element found on earth. Silica can be obtain from natural resources including rice husk ash (RHA). Rice husk, a form of agricultural biomass, is generated in large quantities as a major by-product in the rice milling industry [14]. Rice husk ash (RHA) is released during rice processing; due to its high caloric value (16,720 kJ/kg). RHA has great potential as a thermal energy source. Due to its high silicon content, rice husk has become a source for preparation of elementary silicon and a number of silicon compounds, especially silica, silicon carbide and silicon nitride [15]. SiO₂ as derived from RHA usually constitutes three main phases which are quartz, cristobalite and tridymite depending on the firing temperature[16,17].

Silica is attractive as a catalyst support since has strong structural robustness, stable at elevated temperature and chemically inert. The application of silica in industry included of abrasive, building materials, fillers, electronics and water filtration [18].

Silica based foams are ideally suited for a wide variety of applications in light weight thermal protection systems. These foams are often fabricated by incorporation of air bubbles in an aqueous suspension of silica powders by direct foaming, which is well-recognized as a simple and effective process [19].

Porous silica have been proven invaluable for use as adsorbents, catalyst supports, and porous electrodes thanks to their low density, favorable thermal and mechanical stability, promising bioactivity, super paramagnetic, and chemical inertia[20].

Nickel is well known as non-noble catalyst materials with having large specific surface area and have gained tremendous attention in catalyst because they are active in hydrogenation, hydro treating, and steam-reforming reactions[21,22]. Nickel oxide (NiO) appears to be a promising alternative because of its high theoretical specific capacitance, low fabrication cost[23] and environmental impact [24].

In the present study, the feasibility of slurry technique as to fabricate the SiO₂ as derived from RHA and NiO foams was determined. The effect of sintering temperatures to the physical properties of the fabricated foams.

2. Materials and Method

In this study, the SiO₂ as derived from RHA local rice mill (Muar,Johor) with the average size of 63μm was used as a raw material. Slurry of SiO₂ (20wt%) and NiO (5wt%) composition was applied throughout the study.5wt% Polyvinyl alcohol (PVA) was used as a binder and Polyurethane (PU) polymeric sponge in cylindrical shape of 125mm x 260mm was applied as the template slurry of SiO₂-NiO mixtures was first
prepared in an aqueous media. The dipped PU foams with SiO$_2$-NiO slurry were dried in the drying oven at 110°C for 24 hours. The SiO$_2$-NiO foams were sintered at 1050°C, 1150°C and 1250°C with ramp rate of 2°C/min. The morphologies of the SiO$_2$-NiO foams was observed by using Scanning Electron Microscopy (SEM) (JEOL, JSM- 6700F, JAPAN). Phase analyses of the SiO$_2$-NiO foams were performed by using the X-Ray Diffraction technique (XRD) Bruker D8 Advance, German at the range of 10-100° (2θ). The density and porosity test was carried out using the Archimedes’ method by Mettler Toledo Density Determination Kit model XS64, Switzerland to identify the bulk density and the total porosity of porous foams by using distilled water as the immersion medium.

3. Results and Discussion

3.1 Phase Identification of SiO$_2$-NiO Foams

Figure 1 shows XRD pattern SiO$_2$ and NiO foams sintered at temperatures of 1050°C, 1150°C and 1250°C. Both SiO$_2$ and NiO phases were identified in all green before sintering and after sintered foam. It was also clear that there was no secondary phases (apart from SiO$_2$ and NiO) identified in SiO$_2$-NiO foams sintered at all temperatures and there do not have any new phase will form after sintering process. The SiO$_2$ was identified as the phases of cristobalite (JCPDS 39-1425) and tridymite (JCPDS 42-1401) were present in green and all sintered foams. The peaks positions appearing at 2θ of 36°, 43°, 62°, 76° and 80° were identified as NiO, as perfectly matched to JCPDS 47-1049. The sharpness and the intensity of the peaks indicate the well crystalline nature of the prepared sample[25].

Figure 1. XRD patterns of SiO$_2$-NiO foams sintered at 1050°C, 1150°C and 1250°C.

3.2 Morphologies of SiO$_2$-NiO Foams

Figure 2 shows the image of sintered SiO$_2$-NiO foams surface before and after sintering at 1050°C, 1150°C and 1250°C.

Figure 2. SiO$_2$-NiO foams a) before and b) after sintered 1050°C, 1150°C and 1250°C.
From Figure 3, it is observed that all foam were black in colour due to raw SiO$_2$ and NiO before sintering due to the colour of the slurry. The change in colour of SiO$_2$-NiO foams was observed after sintering process from black to green colour. The colour changed from dark grey to green was due to the presence of NiO [26]. The changed in colour observed may be caused by oxidation of NiO during sintering[27].

As seen in Figure 3, all SiO$_2$-NiO foams fabricated shows appearance of the open and closed pores. The higher temperature would cause the gradual formation of the pores which yielded dense and pores with decreasing porosity.

3.3 Porosity and Density of SiO$_2$-NiO Foams

It was observed that good porosity percentages of more than 50% achieved in all samples indicated that the SiO$_2$-NiO foams were indeed successfully fabricated using the slurry technique. The properties of porous ceramic should consist of > 70 %[28,29]. The total porosity was found to decrease when the increasing of sintering temperature which can be seen in the Figure 4.

![Figure 3. SEM micrographs of SiO$_2$-NiO foam after sintering a) 1050°C b) 1150°C c) 1250°C](image)

![Figure 4. Porosity and density of SiO$_2$-NiO foam at three different sintering temperatures](image)
Figure 4 shows porosity percentage SiO$_2$-NiO foams was 74% at 1050°C and decreased to 71.00% when temperature is increase to 1150°C and decreased to 69.00% when temperature change to 1250˚C. Thus high temperature causes the gradual occurrence formation of the pores where the microstructure will be denser thus with decreasing porosity [30].

Additionally, the density of SiO$_2$-NiO foams formed at 1050°C (0.5239g/cm$^3$) were increased to 0.5547g/cm$^3$ at temperature 1150°C and further increased to 0.6210 g/cm$^3$ with sintering temperature of 1250˚C like in Figure 5. Thus, it is clear that when the higher sintering temperature is set, higher densities were achieved. This is may be due to the reducing pore size and decreasing number of porosity because of migration of the massive atoms towards the connective surfaces which promotes the sintering necks grown and the inter-particle spacing become shorten as the sintering temperature increase [31].

![Figure 5. Density of SiO$_2$-NiO Foams](image)

4. Conclusions

SiO$_2$-NiO foams were successfully fabricated by using the slurry technique at different sintering temperature. XRD analyses signifies that SiO$_2$ as derived from RHA is indeed compatible with NiO, as there were no additional phases detected in foams sintered at 1050˚C to 1250˚C. SEM micrographs showed formation of open pores and closed pores which supports the findings of decreasing porosity percentages and increasing density. Sintering at lower temperature of 1050˚C produced highest porosity (75.19%) in the SiO$_2$-NiO foam. However, although lower porosity (70.95% and 69.72%) were yielded from sintering temperatures of 1150˚C and 1250˚C percentage. The values are still in acceptable range for foams. Moreover the density of the SiO$_2$-NiO foams was found to increase proportionally with the sintering temperature at temperature 1250˚C to 0.6210g/cm$^3$.

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