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Characteristic of Silica Xerogel from Rice Husk Ash Wastes Sintered by Microwave and Conventional

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Abstract

An attempt was made to produce silica from an agricultural waste, rice husks ash (RHA), as well as studied them. The high percentage silica content from ash of rice husks was used as silica source for sodium silica solution. Different heating temperature, time and acid treatments was studied for silica extraction. The composite is characterized and its microwave properties is investigated after sintering from 400 to 1200 ^oC. The results show the existing phases crystoballite at 1100 ^oC in microwave sintering. Preliminary experiment for reflection losses of silica alumina composite show that there are relation between degree of crystallinity and their reflection loss.

Keywords: Silica xerogel, alumina, rice husk ash, sintering, microwave

1 Introduction

Silica (SiO_2) is well recognized as the most applied oxide materials.

Silica is also well known that its structure shrinks considerably during drying, because the aqueous phase in the pores is removed by evaporation, capillary contraction, condensation – polymerization reactions, structural relaxation, and viscous flow [1, 2]. The silica xerogel ceramic are also extensively studied because of their potential application in industry [1–5]. The silica-alumina composites are considered as the leading attenuation technologies of the microwave reflection that have a potential to finally replace the ferrites and carbon that are traditionally used as attenuation media. The silica material has highly porous, lightweight, and a very high external surface area [3, 5]. These characteristics make it possible to become a good alternative candidate of microwave absorbing material. Unfortunately, commercial silica material is expensive. In the study, we propose to replace commercial silica commonly used in microwave absorber industries as filler in ceramic matrix of alumina with high purity silica prepared from rice husks ash (RHA). Each material is characterized carefully before applied as a microwave absorber.

Rice husk are hard protecting covering grains of rice. Combustion of rice hulls affords rice husk ash (acronym RHA). This ash is a potential source of amorphous reactive silica, which has a variety of applications in materials science. Chemical composition of rice husk is shown in Table 1 [6].

Table 1. Chemical composition of rice husk

Element	%
Silica	22.24
carbon	35.77
Hydrogen	5.06
Oxygen	36.59
Nitrogen	0.32
Sulphur	0.02

Method for preparation of silica has been reported by previous researchers in investigating silica from rice hull/husk [6-7]. An alkali extraction method can be used to recover silica from rice hull using low temperature reaction. This method was developed based on the unique solubility properties of an amorphous silica. The high solubility of amorphous silica under basic conditions enables silica to be extracted from amorphous silica containing materials. The extraction of silica from rice hull ash yields a high concentration of silica. The structure, density and mechanical strength of silica are affected by gelatin pH and silica concentration. They reported that high purity silica xerogel (99%) from bagasse waste was obtained through a sodium silicate process with subsequent acid treatment for gelation. The characterization of the silica from RHA was also successfully performed and reported [8-9].

2 Experimental Setup

Materials are prepared for manufacturing microwave absorber materials, they are silica, and alumina (Al_2O_3) materials. The silica xerogel from rice husk ash (RHA) is as filler and alumina (Al_2O_3) are as matrix. Al_2O_3 are obtained from Sumitomo AES-11C with purity ≅ 99.8%. One of microwave absorber material is only prepared from silica ceramic and the other is prepared from silica filled inside Al_2O_3 ceramic matrix (silica-alumina composite). The detailed methods of preparation for both microwave absorber materials are described as follows. The silica-Al_2O_3 composites are prepared from silica of the RHA with the maximum silica content and Al_2O_3 powder. They are prepared by using different level of silica content and thickness. The composites are molded to shape of rectangular slabs. The molded composites are sintered at temperature up to 1200 ^oC using microwave. The composites are characterized using XRD and SEM. The relationship between their microstructure, mechanical, porous and microwave properties (permittivity ε and loss tangent, and reflectivity) with silica content, thickness and sintering temperature is studied by using a Vector Network Analyzer. The procedure of research is shown in Fig. 1.

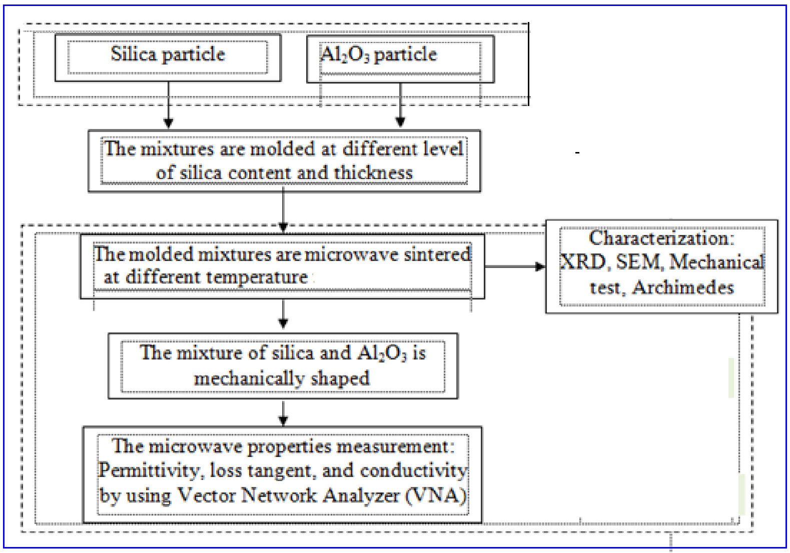

Fig. 1 Research procedure

3 Experimental Results and Discussion

Figure 2 shows XRD patterns of Al_2O_3 amorphous silica xerogel, and 65 mol% Al_2O_3 loaded silica xerogel. The labels S and A are for amorphous silica and Al_2O_3, respectively. The patterns show that a broaden peak of silica xerogel mean that silica is in amorphous phase. The amorphous phase is also shown in XRD pattern of their silica-alumina composite (Fig. 2)

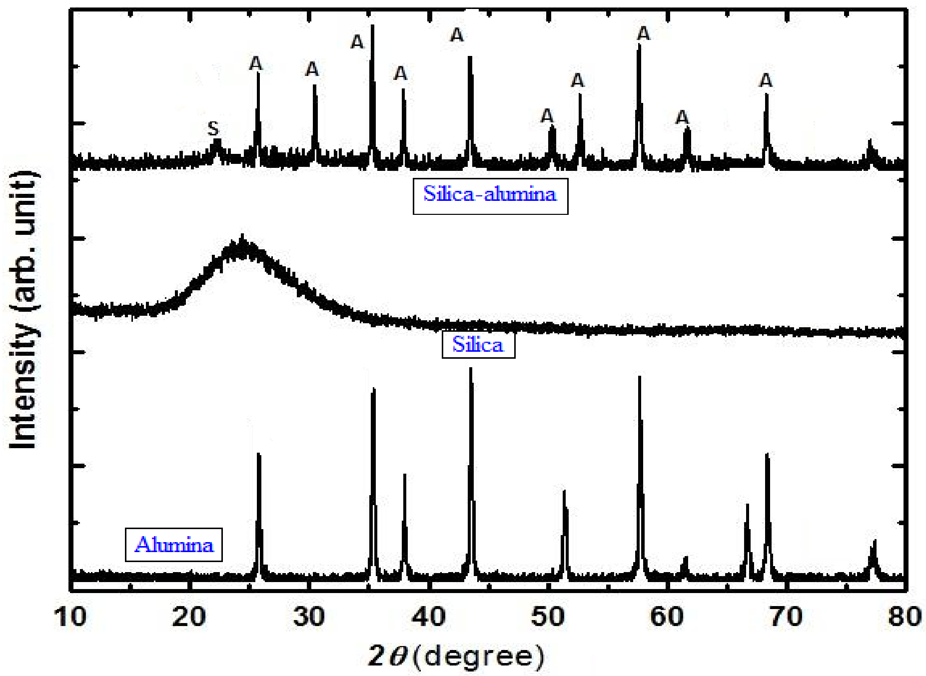

Fig. 2 XRD patterns of alumina, amorphous silica xerogel, and alumina loaded silica xerogel.

Figure 3 shows the XRD patterns of conventionally and microwave annealed 65 mol% Al_2O_3 loaded silica xerogel powders at 1000^{o}C. At about 22^o, the XRD pattern that corresponds to the conventional sintering of 65 mol% Al_2O_3 loaded silica xerogel indicates a weak peak of crystalline crystobalite (C) in composite particle, whereas the pattern for the microwave sintering 65 mol% loaded silica xerogel shows a presence the sharp peak of crystalline crystobalite. This differences in the XRD patterns demonstrates a microwave effect on crystallization. It also demonstrated the advantages of the microwave sintering for production of particle composite with high degree of crystallinity. The difference might be due to the enhancement of mass transport caused by additional driving forces during microwave heating. The driving forces could be triggered by the presence of electric fields resulted in a ponderomotive force and by a thermal stress [8, 10]. Advantages of microwaves on crystallization of silica was also reported previously [9, 11].

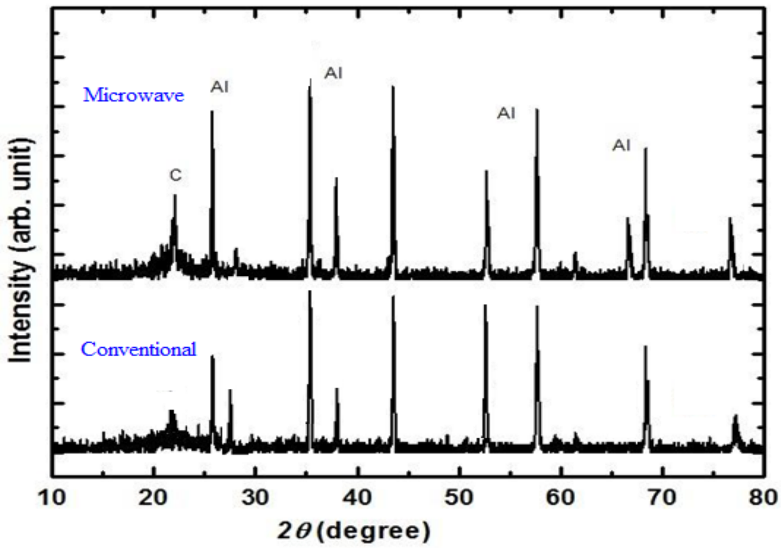

Fig. 3 X-ray diffraction graph of silica-alumina composite sintered by microwave and conventional at 1100^{o}C

In the case of high frequency microwave such as millimeter wave processing, the temperature for the crystallization phase of silica xerogel was about 200^{o}C lower than that observed in the conventional heating [10]. A correlation between the structural changes and the microwave properties of the samples in the frequency range from 8.2 to 12.4 GHz has been also investigated. The increase of the degree of crystallinity was identified as the main reason for the decrease of the reflection losses since the silica xerogel becomes more transparent to the microwaves. The details of experimental results will be reported in separated paper.

4 Conclusion

A series of experiments to investigate of ceramics produced from Al_2O_3 and silica xerogel derived from rice husk ash for microwave absorber by controlling both the content of Al_2O_3 and the sintering temperature was performed. The results presented in this work show the existing phases of crystoballite at microwave sintered samples at 1100^{o}C which is not shown in conventional sintering. Preliminary experiment for reflection losses of silica alumina composite show that there are relation between degree of crystallinity and their reflection loss.

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