Microstructure Development in Silica Glass Ceramic by Rapid Sintering of Submillimeter Wave Gyrotron

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Abstract - In this paper, we present and discuss experimental results on sintering of silica-glass ceramic, produced from a silica xerogel extracted from sago waste ash. The pressed silica xerogels were sintered using submillimeter wave processing of a 300 GHz CW gyrotron at the temperature ranging from 300°C to 1500°C. The microstructural changes are observed using SEM. The results show that the presence of dense layer on the sample surface increases while the presence of pore acts in the opposite direction with increasing temperature.

Keywords: silica xerogel, submillimeter wave gyroton, silica glass ceramic, porosity

1. Introduction

The production of silica glass ceramic at higher sintering temperature by conventional heating needs longer time, which promotes grain growth. In contrast to the conventional (surface) heating the microwave heating is volumetric, since the electromagnetic energy is dissipated simultaneously in the whole irradiated volume. Additionally, the microwave treatment offers higher densities of the heating power, better precision in controlling the process of energy deposition etc. A successful use of millimeter wave heating to sinter materials such as boron carbide [1] and alumina [2] has been reported recently. It has been found that the irradiation by millimeter waves enables very high heating rates to be achieved and reduces the time for grain growth. The smaller grains make the microstructure more uniform and increase the strength of the material. In this paper we present the results of our study and discuss the influence of the sintering temperature using a 300 GHz CW gyrotron on the microstructure of silica glass ceramic.

2. Description of the experimental procedure

Silica xerogel was extracted from a sago waste (solid residue, which is left behind after the starch has been washed out) obtained from the sago processing plant in Kendari, Indonesia. The detailed extraction procedures are described elsewhere [3]. The molding of sample and sintering procedures using a sub-millimeter wave gyrotron (Gyrotron FU CW I) are described in the literatures [4, 5]. Furthermore, the microstructure of the sintered samples was characterized by the scanning electron microscope (SEM).

3. Experimental results and discussion

Figure 1 shows SEM image of the silica xerogel samples without calcination and such sintered at different temperatures using gyrotron heating. In the sample without sintering, both rough surfaces and uniform pores can be observed. With increasing the sintering temperature up to 362°C, the roughness and pores appear to be reduced from the sample surfaces. These pores are ascribed to the empty sites left by evaporated water and to some residues of poly vinyl alcohol [4]. The incorporation of the particles and the reduction of porosity in the sintered samples can be observed at 800°C. Thus it is likely that at this temperature, in the condensation reactions, more hydroxyls on the pore walls are activated and condensate simultaneously. During sintering process, silica begins to melt into pockets of a viscous liquid phase within the pores. It was confirmed from the measured XRD pattern that the silica xerogel starts to crystallize at the temperature [4]. At temperature from 1200°C to 1500°C, the large amount of liquid phase fills more pores, and therefore, the dense layer on the sample surface was observed. The appearance of pores on the sample surfaces sintered at 1200°C and

1500^{o}C is not distinguishable. This can be due to the very low rate of densification and pore removal as a result of the transformation of the amorphous materials into crystalline phase.

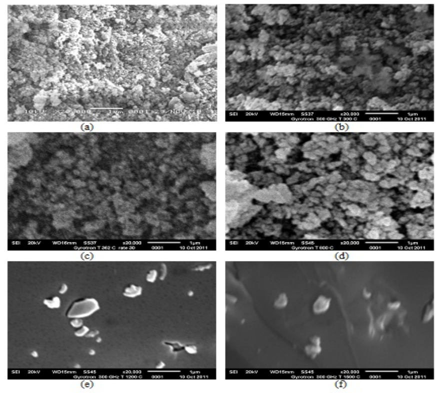

Figure 1. SEM picture of the silica xerogel samples without sintering (a) and sintered at (b) 300^{o}C, (c) 362^{o}C, (d) 800^{o}C, (e) 1200^{o}C, and (f) 1500^{o}C.

4. Conclusions

Xerogel powders have been used successfully for producing glass silica ceramics controlling accurately the sintering temperature. From observed SEM image, an increase the dense layer and pore removal on the glass silica ceramic surface was attributed to three different mechanisms. They are the empty sites left by evaporated water and to some residues of PVA as well as to combusted residual organics at 300^{o}C, partially crystallized sample at 800^{o}C, and fully crystallized sample at temperatures greater than or equal to 1200^{o}C.

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