Chapter V

Discussion

5.1 Powder Preparation

The SEM figures 4.2, 4.3 compare dispersed particles of OX-50 and rice husk ash. OX-50 had excellent dispersion, but rice husk ash had agglomerated particles. So the rice husk ash was classified by the particle classifier device. The figures 4.4, 4.5 illustrate the good result from using the particle classifier. The sequence shows the powder after pre-grinding, the fine fraction, and the coarse discard, respectively. The results of XRD diffraction patterns in figure 4.1 show the amorphous nature of both OX-50 and rice husk ash.

5.2 Gel Preparation

The gel formation step is most important and determines the quality of the product. It has the same significance as the packing and pressing procedure in a conventional sintering process. If the gel does not mix homogeneously, the product will not be glassy after sintering (see figure 4.8 of SEM). Table 4.1 shows that 1% ammonium fluoride condition results in rapid gelation of rice husk ash. So, the pH of the sol must be used to control gelation. The rice husk ash was mixed with 5% boric acid (HCl and NH_4OH to adjust pH): The SEM figure 4.8 shows the microstructure of the gel; some part are homogeneous and some are still heterogeneous. The SEM image in figure 4.9 shows the microstructure of a small part with poor mixing, which must be improved by more intense mixing. The XRD diffraction patterns in figure 4.7 shows that the gels are all amorphous materials.

5.3 Drying

The drying was studied in two types of drying method. One method used temperature control only and the other method used humidity control along with temperature control. The method of no humidity control yielded cracks in almost all products at 70, 90, 110, 200, 300 °C. The method of humidity control at 70, and 90 % relative humidity and 90°C yielded products which were crack free. The drying was studied in terms of shrinking, mass loss , and packing. The formular from chapter II shows that the most important factors are temperature and pressure. The figures 4.13-4.16 of humidity control, for 70 and 90% relative humidity explain the matter of cracking. The two reactions relevant to the drying process were found to be diffusion and evaporation. The first range of drying conditions was controlled by evaporation and the second range was controlled by diffusion. When the diagram shows that the rate depends on pressure, then evaporation is the rate controling step. Diffusion of water in the sample is faster. This region, tension causing crack are less likely to occur. When the rate becomes independent of pressure, then diffusion less likely to starts to take over as the slower step. Now, a humidity gradient exits in the samples, and cracking may occur.

5.4 Sintering

Sintering was investigated at variable temperatures, the chosen conditions for densification without crystallization. The XRD figures show the results of sintering at variable temperatures and times. The XRD figures 4.20 (4.22 at $1250^{\circ}C$ of OX-50 and the figures 4.29-4.31 at $1450^{\circ}C$ of rice husk ash) show some examples which are amorphous. The TTT curves of OX 50 show the appropriate range for complete sintering as amorphous phase. But the TTT curve of rice husk ash (approximately $1450^{\circ}C$)was higher than for OX 50. So higher temperatures and short times were required. The heating rate of sintering was approx. 50 K/min. The sintering rate was kept constant because the sintering process is a competition between crystallization reaction and densification, but the cooling rate did not have much effect.

5.5 Characterization

The characterization is an important part to demonstrate, which of the samples were glassy. The results in table 4.7 help to identify the appropriate sintering temperatures and times. X-ray diffraction can only demonstrate that a material has no long-range order from a certain scale upwards (approximately $>0.5\ \mu m$). The most stringent criterion used in this thesis is the thermal expansion which definitely shows the absence of crystalline silica modifications.