

Title: Preparation and characterization of forsterite (Mg_2SiO_4) from SiO_2 and MgO nanopowders

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ABSTRACT

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The focus of this work is on the synthesis of forsterite (Mg_2SiO_4) bioceramics from SiO_2 and MgO nanopowders. The used raw materials powders (initial powders) were mixed, ball milled and sintered within the temperature range of 1100–1500 °C for 2 h. Differential thermal analysis, dilatometry, Thermogravimetry, thermodilatometric analysis and x-ray diffraction, complementary techniques were used to study the sintering behavior and The crystalized phase during the thermal treatment. X-ray diffraction method was used to characterize the specimens heat-treated in both the dilatometer and furnace. The results shows that the forsterite bioceramic synthesized from pure nano-oxide powders begin to crystallize at 900 °C and completed at 1400 °C for the heat-treated specimens by furnace.

1. Introduction

forsterite is an intermediate compound in the MgO - SiO_2 phase diagram with chemical formula $2MgO \cdot SiO_2$ [1], Pbnm as space group, orthorhombic crystal system, and structure parameters $a=5.19$, $b=4.754$, and $c=10.19$ Å. Forsterite has many attractive properties, such as: melting point (1910 °C), high creep resistance at high temperatures, low thermal/electric conductivity, and high corrosion resistance against molten iron and neutral slag, therefor making it an excellent candidate for high-temperature applications such as refractory materials.

To synthesize forsterite powders, various preparation methods have been devised, including coprecipitation, sol-gel, solid-state reaction, self-spread synthesizing method, and so on. [1-3], Traditional solid state reaction processes are more convenient and desirable for large-scale manufacturing comparing to the techniques listed above. However, the generated $MgSiO_3$ (secondary phase) and residual MgO in the prepared Mg_2SiO_4 directly result in low density, high porosity, poor sinterability, and other inferior qualities during the calcination process.

2. Materials and Procedures

high purity nano powders of MgO and SiO_2 were used to synthesize forsterite bioceramic. yield stoichiometric forsterite content of oxides was selected to (42.70 wt % SiO_2 and 57.29 wt % MgO). The SiO_2 and MgO powders mixture, distilled water, PVA at 1 %, and ZrO_2 balls were emptied in ZrO_2 vials. The

experiments were follow through using a Fritsch P6 planetary ball mill for 3 h at 180 rpm, the furnace condition for drying the slurry are 120°C for 1 day. The LABSYS EVO DTA/DSC-TG SETARAM equipment was used obtain the TG and DTA studies. The samples were heated to 1300 °C under 40 cm^3/min of argon gas flow. The heating rates were 20 °C/min. NETZSCH (Dil 402 C) equipment was used in the dilatometry studies up to 1500 °C, at heating rate of 5 °C/min. Identical dimensions of a reference sample manufactured from Alumina were utilized. The raw powders and sintered samples were characterized using XPERT-Pro diffractometer equipment with Cu-K radiation of wavelength 0.15418 nm. Applying the water immersion procedure, the bulk density of samples was determined using a KERN densitometer.

3. Results and discussion

3.1 Phase formation analysis

Forsterite formation is a multistage process, which is mainly influenced by temperature and time. Therefore, it is essential to analyse the kinetics of forsterite formation using DTA and TDA methods. The phase transformations leading to forsterite formation for the samples heated in the DTA equipment and dilatometer were characterized by XRD, then, the kinetic analysis was performed.

Figure 1 shows DTA/TG/DTG curves of the sample heated from room temperature to 1200 °C at 20 °C/min. based on the figure, it can be observed that there are two endothermic peaks in the DTA curve accompanied by a decrease in mass in the TG curve.

For the first endothermic shift, the percentage of mass decrease is 26.04 %, it temperature ranging from 58°C to 291.5 °C and reaching the maximum 148° C in the DTG curve, which is known for absorbing water. As for the second endothermic transformation, a relative decrease in mass was estimated to be 36.01 % of the total mass within the temperature range of 300 °C to 676.82 °C with maximum temperature at 447.23 °C. The second mass loss is due the dehydroxylation Mg(OH)₂ formed during milling. We also notice that there is another exothermic shift in the DTA curve that is not accompanied with mass loss in the TG curve at 830.33°C.

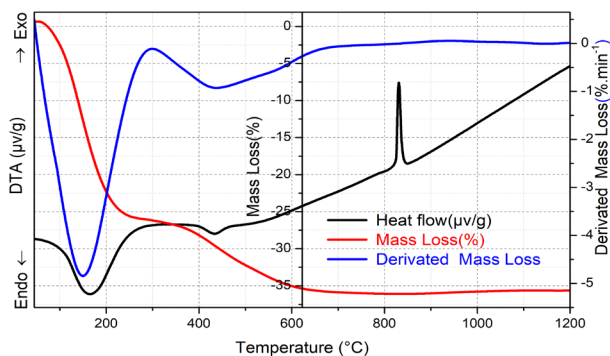


Figure 1 DTA/TG/DTG curves of the sample heated to 1300 °C at 20 °C/min.

Figure 2 shows the linear shrinkage/expansion curve and its first derivative obtained from the preformed experiment on a compact sample heated by 5 °C/min. to 1500°C. Between 25 and 200 °C (stage A) with maximum rate at 75 °C is due to the evaporation of water. The first shrinkage (around 3 %) started at 300 °C and ended at 600 °C (stage B), with maximum rate at 508.5 °C. A second shrinkage (around 17.43 %) in the temperature range 623-823 °C (stage C) with a maximum rate at 763 °C. A second expansion (around 28.69 %) between 1105 and 1500 °C (stage E). The cooling curve does not show any expansions or contractions, which means that the heating led to the formation of stable phase.

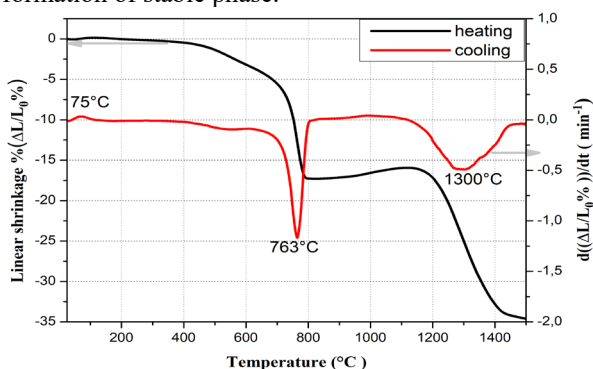


Figure 2 Linear shrinkage curves of the sample.

3.2. Densification

The change in the bulk density of samples is shown in Fig. 3. The density increased from 1.8 to 2.7 g/cm³ with the increase in sintering temperature from 1100 to 1300 °C, then keep increasing from 2.80 to 2.95 g/cm³ with further increase in sintering temperature to 1400

and 1500 °C, respectively. The increase in bulk density is due to the decrease in the amount of pores during sintering. It is believed that the small size and the rounded shape of particles of the milled powder had enhanced sintering and improved densification as a result of the small diffusion path and the large surface area associated with the fine particles. The bulk density value of 2.7 g/cm³ obtained at 1300 °C is very close to the bulk density value of Forsterite, which suggests that the formation of highly dense Forsterite (relative density of almost 90 %) was complete at 1300 °C

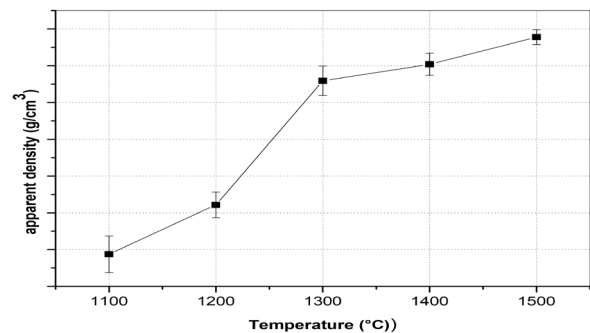


Figure 3 Bulk density of sintered samples

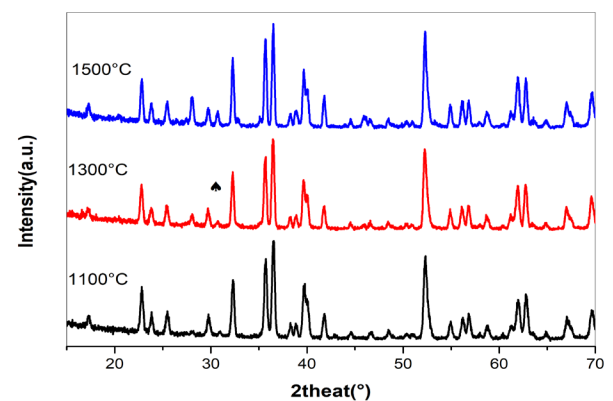


Figure 4 XRD patterns of powder treated

4. Conclusion

Phase transformations leading to the formation of forsterite were characterized using DTA, TDA and XRD. Analysis of samples heated in either the DTA equipment or the dilatometer showed that the reaction of MgO and SiO₂ nano-powders involves the formation of forsterite. The results obtained from DTA and dilatometry experiments, under non-isothermal conditions, showed that the formation of forsterite from pure nano-oxides started at 800 and completed at 1300 °C

5. References

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