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Characterization of forsterite ceramics

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In the present study, phase pure forsterite was obtained in sintered bodies from powder mixtures that did not undergo a heat treatment process prior to sintering. This is contrary to the practice reported in the literature where the powder mixture is normally subjected to a heat treatment at about 1200 °C prior to sintering process. The present results revealed that pure forsterite could be obtained after sintering the powder mixture above 1300 °C. The study found that the mechanical properties of forsterite increased with increasing sintering temperature. A maximum Vickers hardness and fracture toughness of 7.68 GPa and 5.16 MPam$^{1/2}$ were measured for samples sintered at 1500 °C.

Key words: Forsterite, Heat treatment, Densification, Ceramics.

Introduction

Forsterite (Mg_2SiO_4) has found numerous applications in the electronics, communications and refractory industries due mainly to its low electrical conductivity and high melting temperature [1-3]. There are various techniques employed by researchers to prepare forsterite powder including a sol-gel method, mechanical activation, a citrate-nitrate route and a polymer matrix method. The use of a heat treatment, typically in the temperature range of 1000 to 1500 °C has been employed as part of the synthesis process to obtain phase pure forsterite [4-7].

In recent years, forsterite has been reported as a potential bioceramic that possess good biocompatibility and mechanical properties [6, 8-9]. Ni and co-workers [6] synthesized forsterite by a sol-gel method and heat treated it at 1200 °C for 3 hours to obtain phase pure forsterite. After conventional sintering for 8 hours at 1450 °C, a fracture toughness of 2.4 MPam$^{1/2}$ was achieved. This is indeed a good improvement as compared to hydroxyapatite (HA) which has toughness of about 1 MPam$^{1/2}$ [10-12]. Kharaziha and Fathi [13] synthesized forsterite by ball milling for 10 hours and a heat treatment at 1200 °C. These authors found that fracture toughnesses as high as 4.3 MPam$^{1/2}$ was obtained for the sintered ceramics.

In the present study, the main objective was to evaluate the sinterability of forsterite ceramics prepared using the conventional technique without undergoing any heat treatment prior to sintering.

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Experimental Procedures

Forsterite ceramics was prepared using magnesium oxide (MgO; Merck, 97%) and talc ($Mg_3Si_4O_{10}(OH)_2$; Sigma-Aldrich, 99%) as the starting precursors. The talc powder was mixed with the MgO powder according to the weight ratio of 1.88:1. The mixed powders were then ball milled for 3 hours in ethanol using zirconia balls as the milling medium. The slurry was dried in a box oven prior to sieving to obtain ready-to-press powder.

The as-prepared powder was uniaxially pressed at about 2.5-3.0 MPa pressure to form disc and bar samples, followed by cold isostatically pressing at 200 MPa. The compacted green samples were then sintered directly in an air atmosphere without undergoing any heat treatment process as normally reported in the literature. The sintering was carried out in an electric box furnace (LT Furnace) at temperatures ranging from 1200 °C to 1500 °C with a ramp rate of 10 °Kminute^{-1} for 2 hours holding time prior to cooling to room temperature. The phase compositions of sintered samples were characterized using a Shimadzu X-ray diffractometer (XRD), which operated at 40 kV and 30 mA with Cu-Kα as the source of radiation. XRD patterns were recorded in the two theta range of 10-60 ° with a step size of 0.02 ° and a scan speed of 6 ° minute^{-1}.

The bulk densities of samples were obtained by the Archimedes principle using a water immersion technique. Relative densities of the samples were determined by comparing the theoretical density (3.221 g/cm^3) and the bulk densities [14]. Hardness (H_v) of the forsterite samples was determined using a Vickers micro-hardness tester (HMV, Shimadzu, Japan). The indentations were made by a pyramidal diamond indenter with a loading

of 20 g for 20 s according to ASTM E384 standard. Five indentations were made for each polished sample and the average value was recorded. The fracture toughness was evaluated according to the crack measurement method derived by Niihara *et al.* [15]:

$$K_{Ic} = 0.203(c/a)^{-3/2} H_v \cdot a^{1/2} \quad (1)$$

where K_{Ic} is the fracture toughness ($MPam^{1/2}$); c is the crack length; a is the half diagonal of the indent; and H_v is the Vickers hardness.

Results and Discussion

Fig. 1 presents the phase compositions of forsterite samples that were sintered from 1200 °C to 1500 °C. It was found that the forsterite phase was detected at all sintering temperatures, which corresponded to the JCPDS card number 34-0189. However, referring to Fig. 1(a), a minute MgO peak was also present for the sample sintered at 1200 °C. This signified that a low sintering temperature was insufficient to eliminate a secondary phase, which implied that the formation of forsterite was not completed. When the sintering temperature was raised to 1300 °C, pure forsterite phase was obtained.

On the other hand, Ni and co-workers [6] also found that pure forsterite could be acquired through heat treatment at 1200 °C for 3 hours during synthesis. In their experiment the heat treated samples was sintered at higher temperatures (i.e. 1350-1550 °C) to obtain a high density and mechanical properties [16]. In contrast to the present study, the mixed powders were not heat treated prior to sintering and yet phase pure forsterite was obtained when sintered above 1300 °C.

The effect of the sintering temperature on the relative density of sintered forsterite is shown in Fig. 2. The relative density of forsterite increased gradually with an increase in the sintering temperature. A maximum relative density of 90.7% was achieved at a sintering

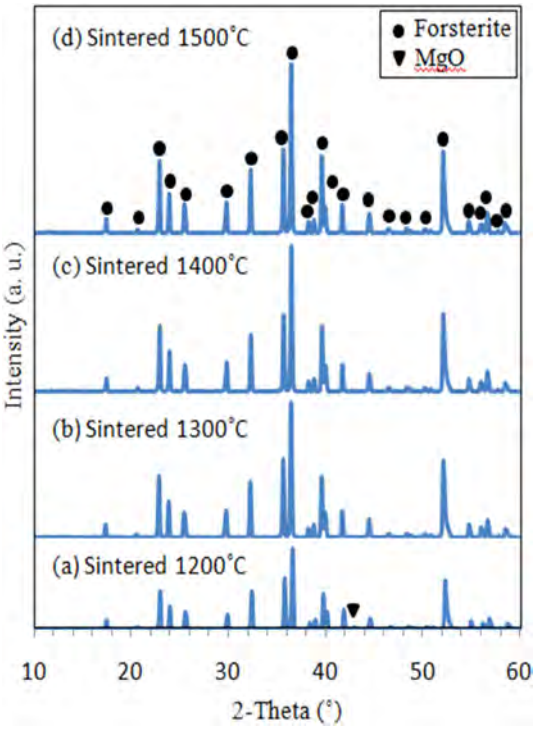

Fig. 1. X-ray diffraction patterns of forsterite samples sintered at different temperatures.

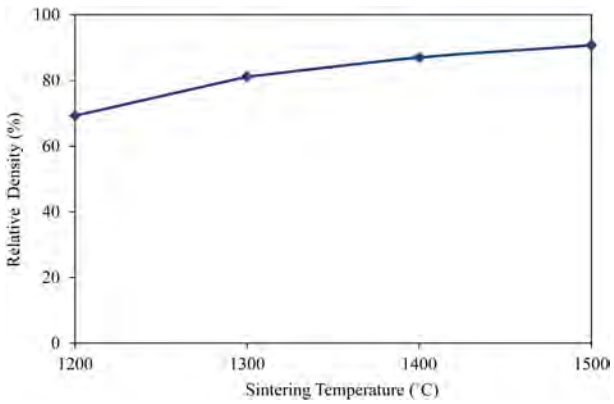

Fig. 2. Relative density of forsterite when sintered at different temperatures.

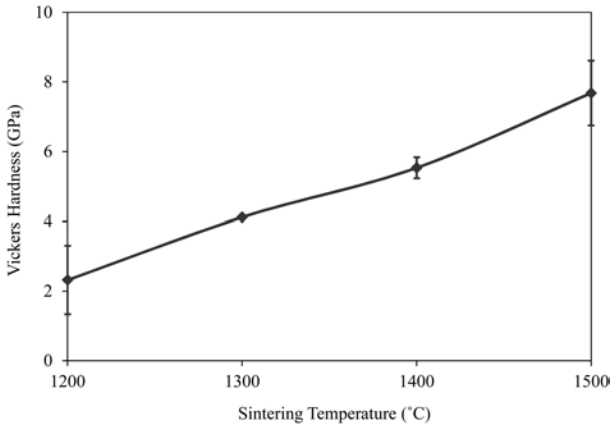

Fig. 3. Vickers hardness variation with sintering temperature for forsterite ceramic.

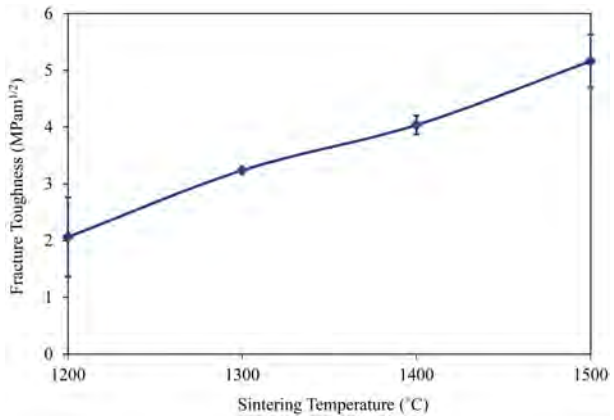

Fig. 4. Effect of sintering temperature on the fracture toughness of forsterite ceramic.

temperature of 1500 °C. This result is encouraging when compared to that reported by Mustafa et al. [17] who acquired low densification (77.6%) for their forsterite after sintering at 1500 °C.

Fig. 3 shows that Vickers hardness variation with the sintering temperature. It was found that the hardness increased linearly from 2.32 GPa to 7.68 GPa when the sintering temperature was increased from 1200 °C to 1500 °C. The highest hardness value was favorable as it was higher than that reported for HA (about 3-7 GPa) and exceeded the lower limit of cortical bone (about 0.24 GPa) [10-12, 18].

The fracture toughness variation with sintering temperature is shown in Fig. 4. The trend is in good agreement with that observed for the Vickers hardness. The highest fracture toughness of 5.15 $MPam^{1/2}$ was achieved at 1500 °C, which is 115% higher than the toughness reported by Ni and co-workers [6]. In another study, Fathi and Kharaziha [13] achieved a fracture toughness of 4.3 $MPam^{1/2}$ i.e. about 20% lower than the current result. Most importantly, the fracture toughness of 5.15 $MPam^{1/2}$ is higher by two fold if compared with the lower limit toughness of typical cortical bone (i.e. about 2 $MPam^{1/2}$) [19].

Conclusions

In the present study, forsterite ceramic was prepared by the conventional ball milling process, without undergoing any heat treatment process and yet the composition was able to be sintered to produce phase pure forsterite in the sintered body. Based on the XRD results, pure forsterite could be obtained during sintering at above 1300 °C. Besides, 90.7% of densification was attained with superior mechanical properties at a sintering temperature of 1500 °C. A maximum Vickers hardness and fracture toughness of 7.68 GPa and 5.15 $MPam^{1/2}$, respectively was achieved.

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