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Microwave sintering and kinetic analysis of Y_2O_3–MgO composites

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Abstract

To obtain fine grained Y_2O_3–MgO composites for infrared application, the kinetics of Y_2O_3–MgO nanopowders (~50 nm) during microwave sintering process was analyzed to track details of densification evolution. Finer structure and higher density were exhibited during microwave sintering process compared with conventional sintering process. The values of grain growth exponent n indicate that grain boundary diffusion is the main migration mechanism for microwave sintering when sintering temperature is below 1300 °C, while volume diffusion and grain boundary diffusion coexist at higher temperature (1400 °C). The calculated grain growth activation energy of microwave sintered samples (108.22 kJ/mol) is much lower than that of conventional sintered ones (160.42 kJ/mol), indicating that microwave sintering process can effectively promote the densification. Based on the kinetic analysis data, microwave sintering parameters were optimized, and Y_2O_3–MgO composites with an average grain size of ~300 nm and Vickers hardness of 11.2 ± 0.3 GPa were obtained.

Keywords: Y_2O_3–MgO composites; Microwave sintering; Kinetic analysis; Densification behavior

1. Introduction

Y_2O_3–MgO composites have recently acquired a high degree of research interest for infrared window materials due to the advantages of low cost and increased damage threshold compared with single-crystal infrared transparent materials. To obtain high infrared transmittance, a requirement for low optical scatter in a composite material is that grain size must be substantially smaller than the wavelength of light to reduce optical scatter to tolerable levels [1]. For instance, the Y_2O_3–MgO composites with grain size of ~310 nm can exhibit a high infrared transmittance of 80%, which is very close to the theoretical value [2]. However, the sintering of nanopowders to full or nearly full density without appreciable grain growth continues to present a significant practical challenge [3].

In view of this, a sintering process which can effectively densify materials without inducing the grain growth is thus demanded [4]. Previously, many materials have been successfully sintered, and fine grain size, uniform microstructure and significant energy saving were achieved by microwave sintering [5–8]. As reported [9], the advantages of microwave sintering were found to include not only higher energy efficiency, higher post sintering density and lower sintering temperature, but also reduced activation energy compared with those of conventional sintering. No report on microwave sintering of Y_2O_3–MgO composites has been undertaken to this date.

In this study, the kinetics of the microwave sintering process of Y_2O_3–MgO composites was analyzed to track the details of densification evolution. Conventional sintering process was also analyzed as a comparison. Furthermore, microwave sintering parameters were optimized, and submicron-grained Y_2O_3–MgO composites were achieved.

2. Experimental procedures

The as-received raw material used in this study was 50:50 vol% Y_2O_3–MgO nanopowders, synthesized previously by sol–gel combustion technique using yttrium acetate and magnesium nitrate [10]. To improve the phase homogeneity, a

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ball-milling process for 12 h with ZrO_2 ball media was applied to the fluffy powder. The milled powders were pressed into green compacts of 30 mm diameter by uniaxial pressing at 30 MPa, followed by isostatic cold pressing at 200 MPa. At this stage, samples had a green density of around 38% of the theoretical density.

Y_2O_3–MgO composites were sintered by microwave sintering and conventional sintering process. Microwave sintering was realized in an automated microwave laboratory furnace (SLW-SQF14X10-1800B). During Microwave sintering, two magnetrons were used with the operating frequency of 2.45 GHz $\pm$ 50 MHz. In all cases, the samples were placed at the same position in the microwave furnace in order to avoid the influence of geometric factor. The sintering temperatures of samples were monitored with an thermocouple at the back of the furnace.

For nonisothermal experiments, the compacted samples were heated to 800 °C, 900 °C, 1000 °C, 1100 °C, 1150 °C, 1200 °C, 1300 °C, 1350 °C, 1400 °C and 1500 °C, with a constant heating rate 10 °C/min, and the furnace was shut down without any holding when each temperature was reached. For isothermal experiments, the samples were held at 1100 °C, 1200 °C, 1300 °C and 1400 °C for 15 min, 30 min, 45 min, and 60 min, respectively.

The densities of the sintered samples were determined based on Archimedes' principle and calculated using the theoretical Y_2O_3–MgO (50:50 vol%) density. Phase analysis was conducted in an X-ray diffraction technique (D/MAX-γA). Microstructural observation was carried out using a field emission high-resolution scanning electron microscope (JSM-6380LA). The average particle size and grain size were measured from SEM graphs using the line-intercept method, and taking into account at least 100 grains. The Vickers hardness measurement was performed using a hardness tester (LECO DM-400FT) with 300 g load and a dwell time of 15 s. The hardness value is the average of 10 measurements.

3. Results and discussion

3.1. Characterization of Y_2O_3–MgO nanopowder

The results from X-ray diffraction (Fig. 1) indicated that the Y_2O_3–MgO nanopowder was a two phase material consisting of cubic Y_2O_3 and cubic MgO phase. Most powders were agglomerated to ~1.5 μm size (Fig. 2(a)) and the particle size was normally below 70 nm, with the average value being ~50 nm (Fig. 2(b), (c)).

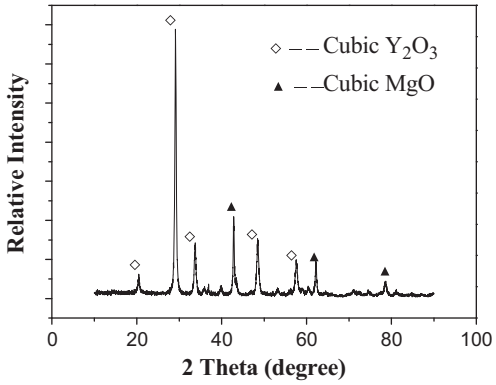

Fig. 1. XRD pattern of Y_2O_3–MgO nanopowders.

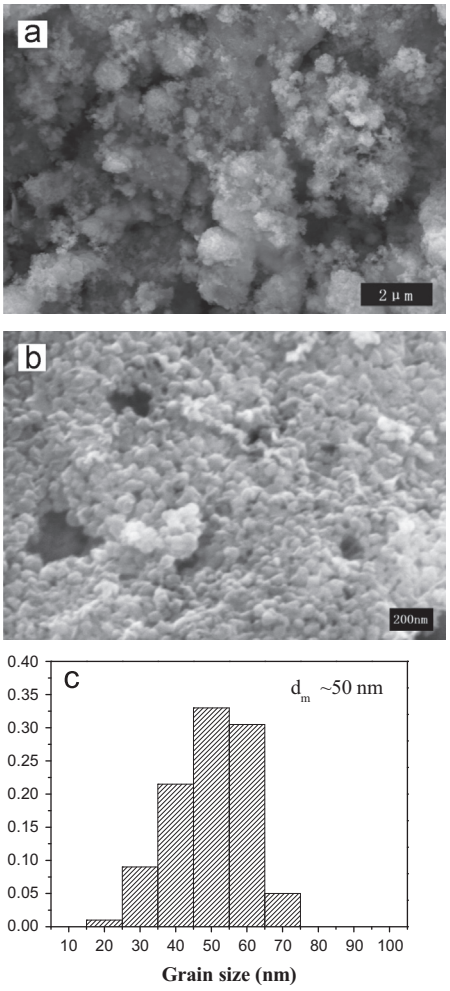

Fig. 2. (a) Low magnification SEM micrographs of Y_2O_3–MgO nanopowders, (b) high magnification SEM micrographs of Y_2O_3–MgO nanopowders and (c) particle size distributions of Y_2O_3–MgO nanopowders.

3.2. Densification evolution

Fig. 3 shows the change in the relative density of Y_2O_3–MgO composites upon heating from 800 °C to 1500 °C. Based on the typical "S" shape curve, the evolution of density during both microwave and conventional sintering can be both viewed as consisting of three stages: the initial, intermediate, and final stages. For microwave sintering, the initial stage of sintering refers to the slow densification at temperatures below 1150 °C, and corresponding density increases from 38% (green density) to 50%. The intermediate stage refers to the rapid densification between 1150 °C and 1400 °C, during which the majority of densification is completed and the density increased from 50% to 95%. The final stage of sintering involves the continued densification, with full or nearly full densification achieved during this stage. As a comparison, for a given temperature,