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Large scale fabrication of magnesium oxide fibers for high temperature thermal structure applications

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ABSTRACT

Magnesium oxide (MgO) fibers were prepared through centrifugal spinning combined with a citrate precursor method. The strategy is quite efficient that the fibers could be obtained in large scale. The morphology, microstructure and crystal phase of the obtained MgO fibers were characterized by digital camera, scanning electron microscopy (SEM), transmission electron microscopy (TEM) and X-ray diffraction (XRD). The results showed that the fibers present continuous morphology with an average diameter of 8 μm. Cubic MgO grains with the size of ca. 50 nm were closely packed in the body of the fibers. MgO fiber-board and fiber-cylinder were fabricated through injection molding of fiber slurry and exhibited promised mechanical performance investigated by compressive strength measurement.

1. Introduction

Metal oxide fibers [1–10] represent one of the most important and widely employed categories of refractory materials. These fibers feature light weight, high melting point, high thermal shock resistance and extraordinary oxidation resistance [11,12], having important applications in many fields including aviation, space flight, military and civil industry, etc [13–16]. Magnesium oxide (MgO) is an alkaline metal oxide with an ultra-high melting point of 2850 °C which is the highest in the family of metal oxide refractory. Furthermore, MgO possesses quite a few advantages, such as lighter molecular weight (40.3 g/mol), theoretical lower density (3.58 g/cm^3) and no phase transition with the changing of temperature. These excellent physicochemical properties indicate MgO fibers would be an excellent high temperature structure material. However, no MgO fiber products are available for factual application till now. The fibers only could be obtained in lab scale.

The fabrication of MgO fibers could be classified into two steps. The first step is the spinning of Mg-contained organic precursor fibers. The second step is the heat treatment of the precursor fibers to burn out the organic component. The precursor spinning dope has been synthesized through hydrolysis and condensation of magnesium methoxide which is so called sol-gel process [17]. However, metal alkoxides are quite sensitive to humidity and difficult to handle. As a result, the method is not appropriate for mass production. The reaction between Mg^{2+} ions and polycarboxylic acids produced much more stable precursor spinning dope with good spinnability [17]. Till now, the reported spinning technologies for MgO fibers were hand drawing and electrospinning [17,18]. Both of them are relatively low-efficiency. Centrifugal spinning which uses high speed centrifugal force to shape fibers allows for a significant increase in yield of production. The fiber production rate has been shown to be over 1 g/min per nozzle in the lab scale units [19].

The purpose of the present work is to introduce an efficient way for the large scale fabrication of MgO fibers. Centrifugal spinning combined a citrate precursor method was developed to fabricate MgO fibers. The morphology, microstructure and crystal phase of the obtained MgO fibers were characterized by digital camera, scanning electron microscopy (SEM), transmission electron microscopy (TEM) and X-ray diffraction (XRD). With the enough MgO fibers, MgO fiber-board and fiber-cylinder were fabricated and the compressive strength was also studied.

2. Experimental procedure

2.1. Chemicals

Magnesium oxide (MgO, 98.0%, Kermel), citric acid monohydrate (CA, 99.5%, Kermel), deionized water (18.2 MΩ).

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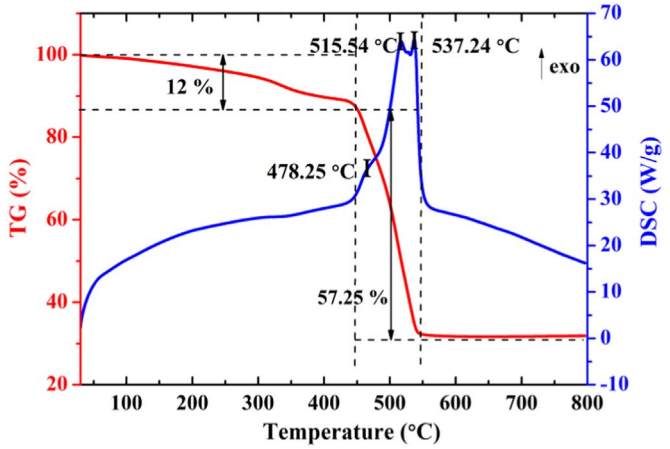

Fig. 1. TG-DSC profiles of the precursor fibers.

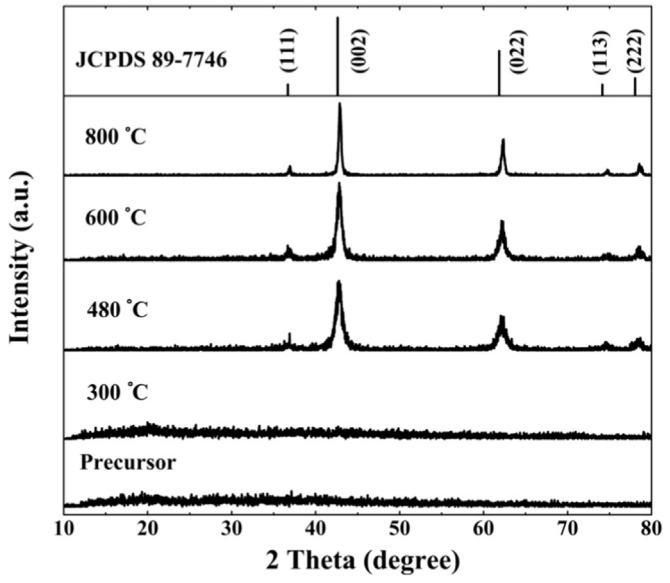

Fig. 2. XRD patterns of precursor fibers and the fibers sintered at different temperatures. The pattern of JCPDS 89–7746 (cubic MgO) was used as reference.

2.2. Fabrication of MgO fibers, fiber-board and fiber-cylinder

2.2.1. Synthesis of spinning dope for precursor fibers

45.7 g CA was dissolved in 150 ml deionized water with vigorous stirring at 40 °C for 20 min. Then, 15.7 g MgO powders were added into the CA solution and magnetically stirred till the white powders dissolved completely. The obtained transparent solution was concentrated into some viscous by a vacuum rotary at 55 °C. The appropriate viscosity adaptive for spinning was about 35–55 Pa s measured by a rotary viscosimeter.

2.2.2. Centrifugal spinning of precursor fibers and heat treatment

The precursor fibers were obtained by centrifugal spinning the spinning dope into precursor fibers. The revolving speed was about 16000–24000 rpm and the aperture diameter was 0.2 mm. The obtained precursor fibers were dried in an oven at 50 °C for 0.5 h. Subsequently, the precursor fibers were heat-treated in steam atmosphere to remove the citrate group at a heating rate of 0.5 °C/min from 120 to 540 °C. The fibers were continued to sinter to desired temperatures in air with a heating rate of 0.8 °C/min. The fibers were held at the desired temperature for 2 h and then slowly cooled to room temperature in the furnace to obtain final products.

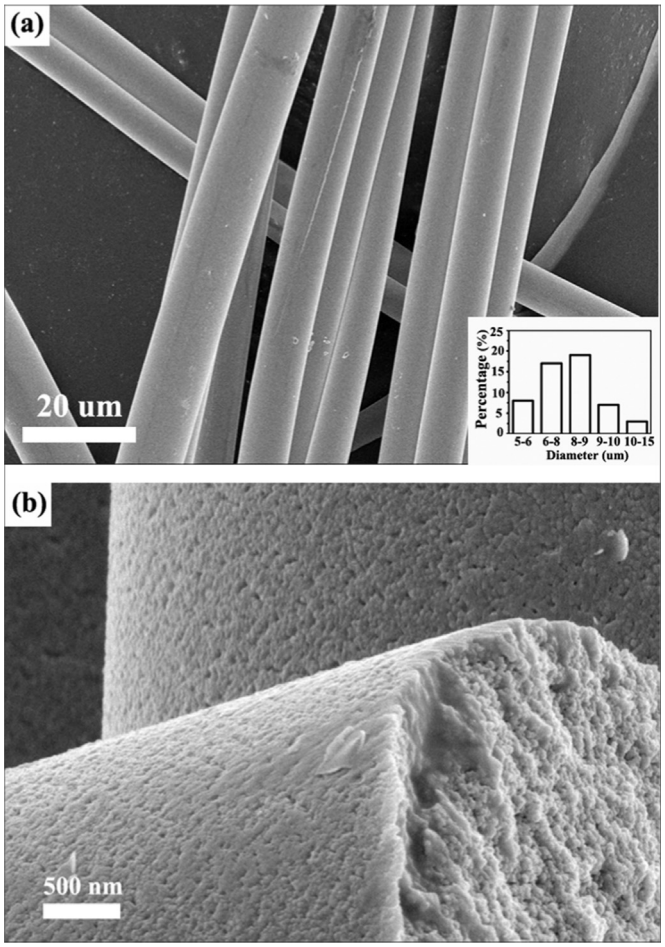

Fig. 3. (a) SEM image of the MgO fibers heat-treated at 800 °C, the inset is the diameter distribution of the MgO fibers. (b) SEM image of cross section of the individual fiber.

2.2.3. Fabrication of MgO fiber-board and fiber-cylinder

2 g MgO fibers were added into a beaker containing 200 ml PVA (0.1 wt%) aqueous solution. After stirring for several minutes, the suspension was injected into a mode with the sizes of 15 mm×15 mm×10 mm. Force was applied from one direction and lasted for about several minutes to obtain an original fiber-board. The fiber-board was sintered at the required temperatures (1200 and 1500 °C) for 1 h. Meanwhile, MgO fiber-cylinder was fabricated by using the similar method.

2.3. Characterization

The thermal behaviors of the precursor fibers were studied by thermogravimetric (TG) and differential scanning calorimetric (DSC) using an SDT Q600 V8.3 Build 101 thermal analyzer instrument, ranging from room temperature to 800 °C at a heating rate of 10 °C/min in air. The XRD was performed on a Bruker D8 advance X-ray diffractometer at 40 kV and 100 mA with CuK_α (λ=1.540598 Å) radiation, employing a scanning rate 5°min^{-1} in the 2θ ranging from 10° to 80°. The sizes and morphologies of the obtained MgO fibers were determined by a Hitachi S-4800 scanning electron microscope. Detailed structures were examined by high resolution transmission electron microscopy (HRTEM) with a JEOL JEM-2100F electron microscope operating at an acceleration voltage of 200 kV. The compressive strength of MgO fiber-board was measured by using a WDW-5 Electronic Tensile Testing Machine. Each sample was measured for 4 times and the average value was adopted as the result.