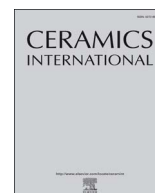




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Effects of water vapor on the crystallization and microstructure manipulation of MgO ceramic fibers

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ABSTRACT

Magnesium oxide (MgO) ceramic fibers are a promising candidate material in high temperature insulating area, supporting area, adsorption area and catalytic area, and most of the properties are determined by the microstructure. In the present work, MgO precursor fibers were fabricated by the centrifugal-spinning combined with sol-gel method. The thermal decomposition and crystallization process of the ceramic fibers were fully characterized. Different atmosphere preheat treatment results suggested that water vapor promoted the thermal decomposition and crystallization of precursor fibers at a lower temperature. Three kinds of particles, including round particles, polyhedron particles to plate particles in the microstructure of the fibers could be manipulated by adjusting the water vapor preheat treatment. The change in textural properties (BET surface area, pore size and pore volume) of the MgO fibers, heated at different temperatures in air after pre-heated treatment in water vapor, was analyzed. Furthermore, the formation mechanisms of the microstructures of the fibers were also presented. The easy manipulation of the microstructures of MgO ceramic fibers may make it a promising material in various areas.

1. Introduction

Magnesium oxide (MgO), a homomorphous compound with cubic structure, has been extensively studied for its functional and structural properties in the past decades. Combined with the stable oxidation states, high melting point ($\sim 2850^\circ\text{C}$), low heat capacity, high corrosion resistance and high geological abundance make MgO a promising refractory material [1–3]. Due to its non-toxic, strong affinity, high isoelectric point and high adsorption capacity, the unique physical properties of the nanostructured MgO with high surface area make it an excellent adsorbent for the removal of toxic metal ions [4] and fluoride ions [5] from water. In the catalytic area, MgO is used as the basic catalyst in organic reactions, such as ozonation of acetaminophen [6] and transesterification of dimethyl carbonate with glycerol [7], and the catalyst support in Ni/MgO catalyst for guaiacol conversion [8].

Ceramic fibers have been gained extensively research since the first energy crisis for its low thermal conductivity, light weight and high thermal stability. With the development of dry-spinning and wet-spinning, ceramic fibers in the range of nanometer to micrometer with various microstructures are successfully fabricated, thus leading to the application of ceramic fibers not only in the refractory area but also in

the supporting, catalytic and adsorbent areas. For various applications of MgO, the microstructure has an apparent influence on the performance. For example, mesoporous structure with larger surface area and narrow pore size distribution could improve the adsorbent and catalytic performance. Thermal decomposition of magnesium oxide precursors [4], sol-gel method [9], hydrothermal method [10] and solvothermal-annealing method [11] have been used for the preparation of MgO with different microstructures to promote its performance. Compared with other methods, the sol-gel method could be simple and convenient, with homogeneous solution operated at lower temperatures. In recent years, both centrifugal-spinning [12] and electrospinning techniques [13] have been used for the fabrication of dense magnesium oxide ceramic fibers. Nevertheless, seldom has manipulated the microstructure of the MgO ceramic fibers, which constrained the application of MgO ceramic fibers to refractory area. It is necessary for exploring new route to prepare MgO fibers and manipulate the microstructure of the fibers to widen its applications.

In the present study, MgO ceramic fibers were successfully fabricated by the centrifugal-spinning method. The decomposition process and crystallization of the precursor fibers in air and water vapor were fully discussed. Water vapor pre-heat treatment was effective in

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manipulating the microstructure of the MgO ceramic fibers, and the formation mechanism was presented. In the future, one would use the water vapor pre-heat treatment to obtain multi-functional MgO ceramic fibers.

2. Experimental

2.1. Materials

Magnesium oxide (MgO, $\geq 99\%$, Tianjin Fuyu Fine Chemical Co., Ltd.), citric acid (CA, $C_6H_8O_7$, $\geq 99\%$, Tianjin Fuyu Fine Chemical Co., Ltd.), and deionized water were used as starting materials without further purification.

2.2. Preparation of the precursor fibers

The spinning solution was prepared by the following procedure. Appropriate amount of MgO and CA (mol ratio MgO:CA = 2:1) were mixed in water with continuous magnetic stirring at 65°C until the mixture was transparent, and then cooled to room temperature. The solution was adjusted to a suitable viscosity (40–100 Pa s) by evaporating solvent to obtain a good spinnability. The MgO precursor fibers were prepared via centrifugal-spinning method using the laboratory-made centrifugal spinning machine with the hole diameter of 0.2 mm and a rotating speed of 23,000–25,000 r/min. The precursor fibers were put into a furnace for heat treatment at a rate of $1^\circ\text{C}/\text{min}$ to 600°C and $2^\circ\text{C}/\text{min}$ to higher temperatures in air, and held for 2 h, then cooled to room temperature in the furnace to obtain final samples. For comparison, it was the same with the heat treatment procedure in water vapor. The precursor fibers were put into an atmosphere programming furnace, first preheated at different temperatures (350°C , 450°C , 600°C) with a heating rate of $1^\circ\text{C}/\text{min}$ in water vapor atmosphere, and heat-treated to a desired temperature in air and held for 2 h, and finally cooled to room temperature in the furnace to obtain MgO fibers. Photographs of the MgO precursor and the MgO fibers heat-treated at 1000°C in air after different pre-heat treatment were shown in Fig. 1. The MgO fibers were given as AT and WT₁-T₂ for the MgO precursor fibers heat-treated at different temperatures in air and water vapor, respectively, where T was assigned as the heat-treated temperature in centigrade in air, T₁ was assigned in water vapor, T₂ was assigned in air after precursor fibers were pre-heated in water vapor.

2.3. Characterization

Thermogravimetry and differential scanning calorimetry (TG/DSC, SDT Q600 v8.3 Build 101, TA, US) measurements of the precursors were performed at a heating rate of $10^\circ\text{C}/\text{min}$ from room temperature to 800°C in air. Fourier transformation infrared (FT-IR, ALPHA-T,

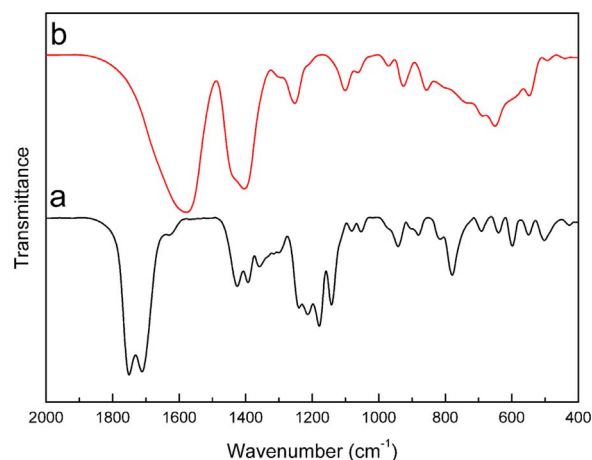


Fig. 2. IR spectra of the citric acid (a) and the precursor fibers (b).

Bruker, Germany) spectra recorded in the region $4000\text{--}400\text{ cm}^{-1}$ were measured with a RT-DLATGS spectrometer using the KBr pellet method. X-ray diffraction (XRD, D8 Advance, Bruker, Germany) data collection was performed with $\text{Cu-K}\alpha$ radiation using a graphite monochromator in the range of $10\text{--}90^\circ$. Specific surface area and pore size distribution analyzer (JW-BK 112, JWGB Sci.& Tech., Beijing, China) were used to measure the N_2 adsorption-desorption isotherms at 77 K after samples were evacuated at 150°C for 8 h under vacuum. The surface areas (S_{BET}) were calculated using the Brunauer-Emmett-Teller (BET) equation. The pore size distribution and pore volume were obtained with the Barret-Joyner-Halenda (BJH) method using the desorption branch of the isotherms with the relative pressure (P/P_0) from 0.05 to 0.3. Scanning electron microscope (SEM, S-4800, Hitachi, Japan) was used to observe the surface morphologies and microstructures of the fibers.

3. Results and discussion

3.1. Formation of the MgO precursor fibers

The IR spectra of citric acid and the precursor fibers were shown in Fig. 2a and b, respectively. For free anhydride citric acid in Fig. 2a, bands [14] at 1750 and 1711 cm^{-1} were correspondingly assigned to the antisymmetric and symmetric stretching modes of $-\text{COO}-$, respectively. While for MgO precursor fibers in Fig. 2b, the antisymmetric and symmetric stretching modes were at 1579 and 1404 cm^{-1} , which were the characterized vibrations of ionized carboxylate of citrate [15]. There were no carboxylate vibrations of free anhydride citric acid for the precursor fibers, which suggested that the hydrogen atoms of carboxyl groups of citric acid were completely replaced by Mg^{2+} to form a magnesium citrate complex.

3.2. Thermal decomposition process of the precursor fibers in air and water vapor

The thermal decomposition of MgO precursor fibers in air was characterized by TG/DSC and shown in Fig. 3. There were three stages of weight loss and two obvious exothermal peaks in the TG/DSC curves. The first stage was due to the volatilization of absorbed sorbents and the weight loss at the second state may attribute to the thermal pyrolysis of C-H for citrate. Above 400°C , apparent weight loss (41.54%) and two exothermal peaks (473 and 513°C) appeared at the last stage for the thermal decomposition of magnesium citrate. There was no intermediate formation such as carbonates but complete decomposition of the magnesium acetate from the previous study [16,17]. The last stage was mainly due to the thermal pyrolysis of magnesium carboxylate and crystallization of MgO. The percent of the remained weight was 33.97% (Seen from Fig. 3, and the calculated value was 34.05%),

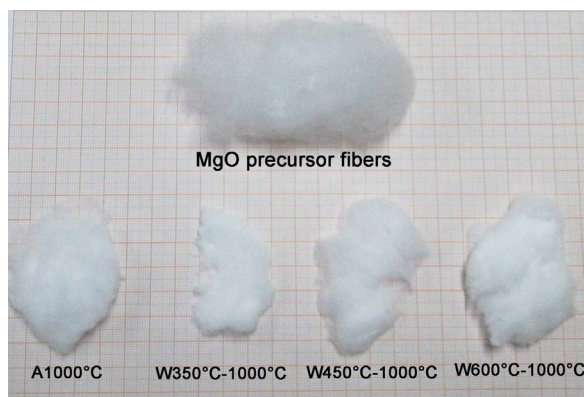


Fig. 1. Photographs of the MgO precursor fibers and the MgO fibers heat-treated at 1000°C after different pre-heat treatments.

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