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Letter

Preparation of partially stabilized zirconia from fused zirconia using roasting

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ABSTRACT

The present study attempts to utilize fused zirconia to prepare partially stabilized zirconia using roasting process. Effects of roasting temperature and holding time on the specific polymorphic phase transition of fused zirconia were systematically analyzed. Crystalline compound and microstructure of fused zirconia before and after roasting process were obtained and characterized by XRD and SEM, respectively. The characterization results show that untreated fused zirconia mainly consists of crystalline compounds of cubic ZrO_2 phase; while the roasted one mainly composed of crystalline compounds of cubic ZrO_2 phase and monoclinic ZrO_2 phase. Partially stabilized zirconia had to have a big acicular pattern crystal grains and finely ground particles gathering at the crystal boundary. Cubic ZrO_2 phase of fused zirconia is partially converted into monoclinic ZrO_2 phase at roasting temperature of 1723 K for 240 min.

1. Introduction

It is well documented that zirconia (ZrO_2) exists in three main polymorphic phases: tetragonal phase, monoclinic phase and cubic phase [1]. The tetragonal zirconia transformation to the monoclinic zirconia in the region of a propagating crack can be utilized to increase the fracture toughness of partially stabilized zirconia and composites containing ZrO_2 particles [2].

Partial stabilized zirconia shows obvious advantages in high temperature industrial fields, especially under the special operating conditions of against high temperature and erosion [3,4]. It finds application as high temperature refractory materials, grinding materials and high temperature insulation materials [5]. Partial stabilized zirconia also has a characteristic of martensitic transformation, which is an important character for improving the toughness of ceramic materials and thermal shock resistance of refractory materials [6]. The application of partially stabilized zirconia doped with various stabilizing oxides such as MgO, CaO, CeO and Y_2O_3 is widely used [7,8]. ZrO_2 doped with CaO could form solid solution and complex. The internal structure of crystals would be changed from a monoclinic phase to a mixed phase of quartet phase and cubic phase in stable double-crystal structure.

Partial stabilized zirconia are obtained from fused zirconia by roasting as partially stabilized zirconia has superior mechanical properties, such as anti-heat, low thermal conductivity, large coefficient of thermal expansion and stability, as well as good wear resistance [9]. Thus, in the present work, the partial stabilized zirconia is prepared by roasting process. Effect of the roasting temperature and holding time on crystalline compound and microstructure is mainly analyzed. The experimental conditions for producing partially stabilized zirconia from fused zirconia are obtained. The polymorphic phase transition and microstructure of fused zirconia before and after roasting are also analyzed.

2. Experimental

2.1. Materials

In the present study, fused zirconia is obtained from Yingkou City, Liaoning Province, China. Fused zirconia containing more than 92% ZrO_2 is prepared by electric melting method. The chemical composition of fused zirconia is listed in Table 1.

2.2. Characterization and apparatus

X-ray diffraction (D/Max 2500, Rigaku, Japan) with Cu Kα radiation analysis is performed to assess the changes in composition of samples. Scanning electron microscope (XL30ESEM-TMP, Philips, Holland) analysis of the sample is utilized to assess the microstructure morphology of sample before and after roasting.

2.3. Methods

100 g of fused zirconia is weighed and placed inside a quartz sample holder of 40 mm diameter. The sample holder is placed inside the muffle furnace (YFX12/16Q-YC, Shanghai, China) and heated to 1723 K at a heating rate of 288 K/min and held at this temperature for 240 min. The temperature of the raw materials during roasting process is monitored using a thermocouple inserted into the raw materials. After finishing the roasting process, the roasted raw materials are naturally cooled to room temperature in the furnace.

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Table 1
Chemical composition of raw materials (mass percentages).

Material	ZrO_2	CaO	SiO_2	TiO_2	Al_2O_3	Fe_2O_3
Mass %	92.1	7.1	0.3	0.2	0.2	0.1

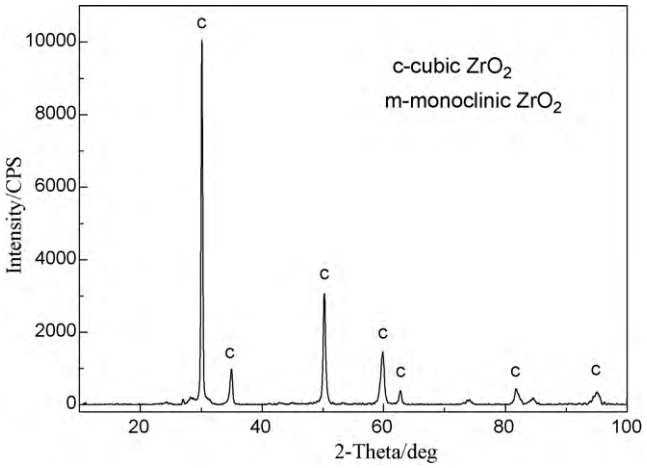

Fig. 1. XRD patterns of fused zirconia.

3. Results and discussion

3.1. Characterization by XRD

The XRD characterization for the fused zirconia before roasting is shown in Fig. 1. It can be seen that the cubic ZrO_2 is the main crystalline compound in the fused zirconia. In addition, all the X-ray diffraction peaks of the samples matches well with the standard XRD pattern of cubic ZrO_2 phase.Partially stabilized zirconia after roasting at 1723 K for 240 min is also characterized by XRD, which is shown in Fig. 2. It can be found that the diffraction peaks of cubic ZrO_2 phase gradually are broadened and their intensities are decreased under roasting process. Compared with fused zirconia before roasting, the partially stabilized zirconia has peaks at $2\theta = 28.2^\circ$ and 31.5°, which are the strongest and the second strongest peaks of monoclinic ZrO_2, while the third strongest peak is peak is seen at $2\theta = 24.0^\circ$. The result shows the cubic fused zirconia transforms from single pure cube ZrO_2 phase into the mixture of the cubic ZrO_2 phase and the monoclinic ZrO_2 phase. It can be inferred from XRD data that cubic ZrO_2 phase is partially converted to monoclinic ZrO_2 phase.

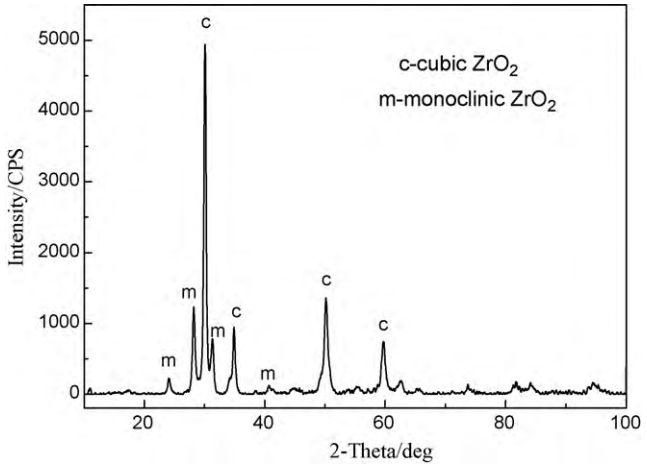

Fig. 2. XRD patterns of partially stabilized zirconia obtained at roasting temperature 1723 K for 240 min.

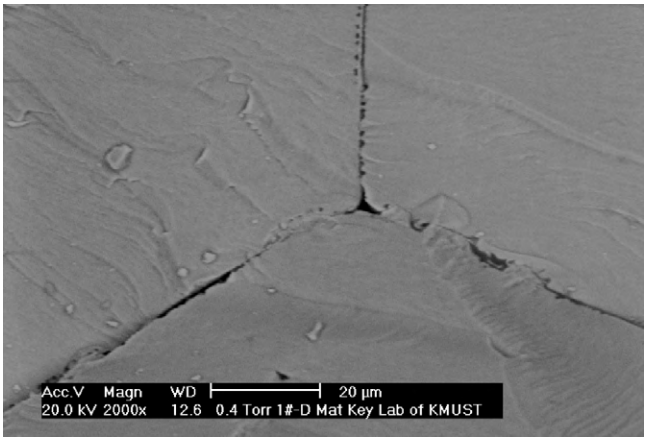

Fig. 3. SEM photograph of fused zirconia.

Fig. 4. SEM photograph of partially stabilized zirconia obtained at roasting temperature 1723 K for 240 min.

3.2. Characterization by SEM

The SEM analysis of raw material is shown in Fig. 3 which reveals the regular crystal boundaries. The crystal boundary intersection points are almost very balanced 120° angles, which indicate that the raw materials are mainly cubic ZrO_2 phase.

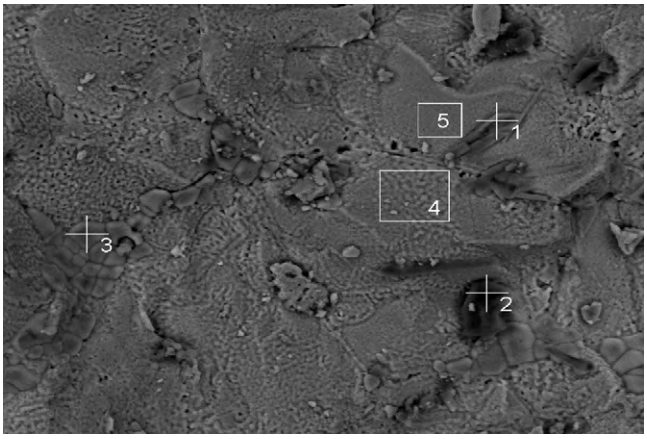

Fig. 5. District of EDAX analysis.

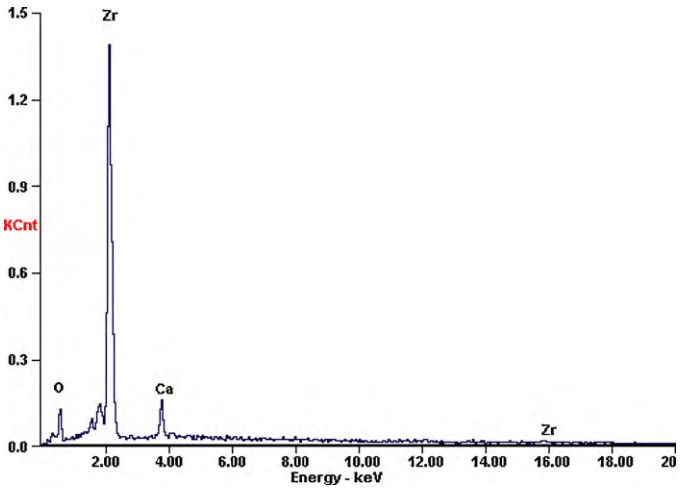

Fig. 6. EDAX analysis results of Spot 3.

Microstructures of partially stabilized zirconia obtained at roasting temperature 1723 K for 240 min, are characterized by SEM is shown in Fig. 4. It can be seen that big acicular pattern crystal grains and gathering of circular finely ground particles at the crystal boundary, indicating stabilizer CaO of fused zirconia separated during the roasting process.

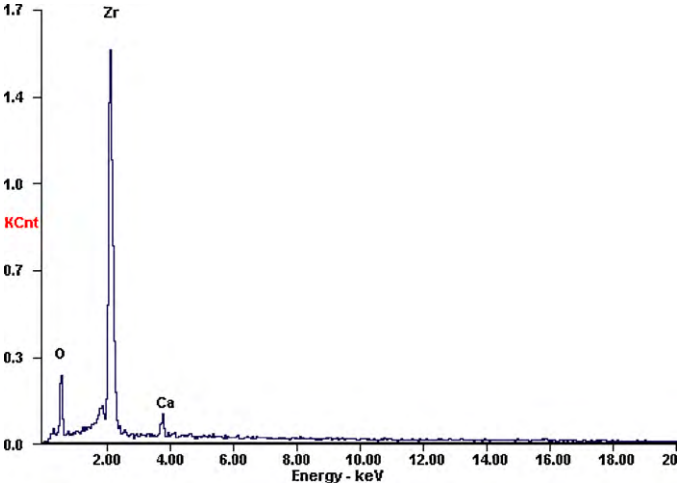

Fig. 7. EDAX analysis results of District 4.

Fig. 5 shows the SEM photograph of the phases (Points 3 and District 4 as indicated by the marks) in partially stabilized zirconia. The EDAX analysis is performed to assess the changes in the separation in crystal the crystal boundary, the results are presented in Figs. 6 and 7. It can be found from Figs. 6 and 7 that the CaO content of stabilizer in crystal grain is 3.26%, but in the granulated structure of crystal boundary the CaO stabilizer content is 6.35%. The content of stabilizer CaO in the crystal boundary is bigger than that in the crystal grain. This clearly shows that the stabilizer CaO separated in the crystal boundary during roasting process.

4. Conclusions

Fused zirconia is the most popular and industrially widely adopted precursor for the preparation of partially stabilized zirconia. The present study utilizes fused zirconia to prepare partially stabilized zirconia using roasting, in order to obtain the better crystalline compound and good microstructure. The experiment parameters were obtained with roasting temperature of 1723 K and holding time of 240 min. The partially stabilized zirconia prepared under the experimental conditions was characterized by XRD and SEM, from which the diffraction pattern satisfactorily matched with that of the standard cubic ZrO_2 phase and monoclinic ZrO_2 phase.

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