Preparation of CePO$_4$ Modified ZrO$_2$ Ceramics with Different Particle Sizes and Their Mechanical Behaviors

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1. Introduction

Zirconia is widely used in the fields of oxygen sensors, catalysts, solid oxide fuel cells, and structural engineering ceramic currently [1] because of its high temperature ionic conductivity, good mechanical strength and toughness, and corrosion prevention characteristics. However, its brittleness and hardness hinder the application in dentistry to a great extent [2]. Some studies have shown that adding an auxiliary phase [3], which has a low mechanical strength, or adding some rare earth phosphate [4] to zirconia can efficiently improve its machinability. We have previously studied the mechanical property of CePO$_4$ coated ZrO$_2$ and determined the optimum CePO$_4$ content and the best sintering temperature [5].

A finer grain size gives ceramic materials better mechanical properties [6], and when the size ranges from microns to the nanoscale, a series of groundbreaking new features such as low-temperature superplasticity and scalability are produced. We tried to control the grain size growth of zirconia by sparking plasma sintering and have made some progress [7]. However, a question arose about the effect of the grain size of zirconia on the coating process and the mechanical properties of the coated products. We reviewed the literature and found no research concerned with this question.

In this study, we prepared different particle size 3 mol% Y$_2$O$_3$-stabilized tetragonal zirconia polycrystalline (3Y-TZP) coated with CePO$_4$, and the effect of grain size on the microstructure and mechanical properties of the sintered samples was determined to further improve the machinability of the zirconia.

2. Materials and Methods

2.1. Materials. 3 mol% yttria-stabilized tetragonal zirconia (3Y-TZP; Hangzhou Wanjing Advanced Materials Co., Ltd., Hangzhou, China) was the main starting material used in this study. In accordance with the grain size, the zirconia powder was divided into four groups: group A (50–80 nm), group B (100–150 nm), group C (300–500 nm), and group D (500 nm–1 μm). Transmission electron microscopy (TEM) images of...
the four groups are shown in Figure 1. The other chemicals such as polyethylene glycol 20000 (AR) and cerous nitrate (AR) were all purchased from Beijing Chemical Plant.

2.2. Coating the Zirconia Powder. In this study, 3 wt% 3Y-TZP powders with different particle sizes were dispersed ultrasonically in distilled water for 10 minutes. PEG was added as a dispersant at 2% of the weight of 3Y-TZP, and ammonium citrate was added at 1.5% of the weight of 3Y-TZP as a release agent. They were dissolved in deionized water at a temperature of 50°C and then added to a suspension of 3Y-TZP. An as-prepared NH₄H₂PO₄ solution was added to the suspension after 10 minutes of ultrasonic treatment and then the pH of the solution was adjusted to 4.0–5.0 via adding H₃PO₄. Ce(NO₃)₃ was added drop by drop to the solution up to 1/3 of the mole ratio of 3Y-TZP and the mixture was stirred for 2 h at 50°C. Finally, the solid-phase content was separated by centrifugation and dried in an oven, milled, calcined at 800°C, and then milled again. The powder was analyzed using TEM.

2.3. Sintering Process and Microstructure Characterization. The coated and uncoated powders were produced in a steel mold, mashed, sieved to 50 meshes, and reformed. The formed body was pressed under a load of 200 MPa for 5 min in a cold isostatic press machine. Then the body was sintered as follows: the temperature was raised to 450°C at a rate of 100°C/h and maintained for 1 h, and then it was raised to 1200°C within 2.5 h to decrease the tetragonal to monoclinic phase transition. Finally, the temperature was raised to 1250°C, 1300°C, 1350°C, 1400°C, 1450°C, and 1500°C, respectively, at a rate of 200°C/h and maintained for 2.0 h. The Archimedes method was used to measure the density of the samples. An electronic vernier (accuracy: 0.01 mm) was used to determine the diameters of the samples before and after the sintering process and the values were \( l_0 \) and \( l \), respectively. The formula \([ (l_0 - l)/l_0 ] \times 100\% \) was used to determine the linear shrinkage.

The phase and the microstructure of the as-prepared ceramic samples were determined by using X-ray diffraction (XRD) (Rigaku D/MAX-2550V) and scanning electron microscopy (SEM). Besides, transmission electron microscopy (TEM) was used to determine the morphology of the particles.

2.4. Mechanical Testing. The sintered ceramics samples were shaped into cuboid with dimensions 3 mm × 4 mm × 36 mm and 4 mm × 6 mm × 30 mm, respectively. An EZ-100 Universal Test Instrument was used to measure the bending strength by means of the three-point bending method and
30–50 nm, uncoated
50–100 nm, uncoated
100–300 nm, uncoated
300–500 nm, uncoated
30–50 nm, coated
50–100 nm, coated
100–300 nm, coated
300–500 nm, coated

Figure 2: XRD patterns of the uncoated and coated 3Y-TZP powders.

Figure 3: Linear shrinkage of the samples at different temperatures
1: 30–50 nm, uncoated; 2: 50–100 nm, uncoated; 3: 100–300 nm, uncoated; 4: 300–500 nm, uncoated; 5: 30–50 nm, coated; 6: 50–100 nm, coated; 7: 100–300 nm, coated; 8: 300–500 nm, coated.

Figure 4: XRD patterns of the sintered 3Y-TZP samples.

3. Results and Discussion

The TEM images show that the particle sizes of the four kinds of 3Y-TZP powders that were purchased from Hangzhou Wanjing Advanced Materials Co., Ltd., are 30–50 nm, 50–100 nm, 100–300 nm, and 300–500 nm. The particles have a near ball-shaped morphology and the size uniformity decreases with an increase in the particle size.

XRD patterns of the uncoated and coated powders are shown in Figure 2. From these patterns, the content of the tetragonal phase of the two samples within 100 nm is similar and when the particle size exceeds 100 nm, the content of the tetragonal phase decreases rapidly and it is barely visible when the particle size is 300–500 nm. The characteristic peak for CePO₄ is present in the XRD patterns of the coated samples, which indicates that the coating process was successful.

The linear shrinkage of all the samples was evaluated, and from Figure 3, the optimum sintering temperature of the uncoated and coated samples was determined to be 1550°C and 1650°C, respectively. The particle size is not related to the sintering temperature and the coating process is responsible for the differences observed in the linear shrinkage.
Figure 5: SEM images of the fractured surfaces of the uncoated samples. (a) 30–50 nm, (b) 50–100 nm, (c) 100–300 nm, and (d) 300–500 nm.

Figure 6: SEM images of the fractured surfaces of the coated samples. (a) 30–50 nm, (b) 50–100 nm, (c) 100–300 nm, and (d) 300–500 nm.
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In summary, the grain size of zirconia before coating greatly influences the mechanical properties of the coated samples because different particle sizes result in different fracture styles. When the grain size is less than 100 nm, the differences are small, and when it is larger than 100 nm, the differences are very noticeable. At the experimental limits, the 30–40 nm samples have the best flexural strength and machinability rating.

References


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