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Novel preparation of anti-static zirconia ceramic by iron infiltration at high temperature

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ABSTRACT

A simple and innovative processing method for fabricating anti-static ceramics has been developed based on Fe-infiltration in sintered tetragonal polycrystalline zirconia doped with 3 mol% yttria (3Y-TZP). The 3Y-TZP samples infiltrated by Fe in a reducing atmosphere showed both excellent anti-static and mechanical properties. Effects of infiltration temperature and time on the surface resistivity and Vickers hardness of the prepared samples were investigated. The surface resistivity and hardness were found to decrease with increases in the infiltration time and temperature. For a typical sample infiltrated at 1000 °C for 4 h, the surface resistivity decreased to 10^7 Ω/□, and the Vickers hardness was 11.2 GPa. The microstructure, chemical composition, and anti-static mechanism were investigated by XRD, SEM and XPS. The results indicated that there was a phase transformation from *t*-ZrO_2 to *m*-ZrO_2 during the infiltration process. It was also found that Fe, Fe_3O_4 and FeO were located in the infiltration layer, conferring an antistatic property to the ZrO_2 ceramics.

1. Introduction

The electrostatic phenomenon is very common in the fields of electronic information, aerospace, petrochemical, oil paint, textile, etc., leading to varying degrees of damage. According to U.S. statistics, in the electronics industry, the annual loss due to electrostatic hazards amounts to more than $10 billion; the annual loss for UK electronics caused by static electricity reaches as much as £2 billion; 45% of sub-quality products in Japanese electronic devices caused by static electricity. Therefore, there is a high demand for the development of anti-static materials. According to the related standard for anti-static materials, a material with a surface resistivity in the range of 10^5 Ω/□ to 10^{12} Ω/□ and a volume resistivity in the range of 10^4 Ω cm to 10^{11} Ω cm is defined as an anti-static material.

At present, anti-static materials applied in engineering could be divided into two categories: (1) Polymer-based composites. Such polymer-based anti-static materials or coatings are prepared by the addition of carbon nanotubes, graphite, carbon black, conductive fiber, or mica powder or by utilizing the conductive path of the polymer itself. Unfortunately, it is difficult to manipulate the dispersion and homogeneity of the electrically conductive phase in such materials, and they also exhibit poor durability, high temperature resistance, and low wear resistance [1]. (2) Anti-static glazes. The anti-static function is realized by adding conducting (or semi-conducting) oxide powders or conducting fiber into conventional anti-static glaze. Such anti-static materials are mainly applied in the fields of building materials, such as wall and floor tiles and anti-static platen [2]. The main disadvantages of such materials are poor mechanical properties and a narrow range of applicability.

The above materials could not meet the requirements of a harsh environment such as high temperature or high-frequency friction or in aerospace applications. High-performance structural ceramics are adopted to address the limitation in such harsh environments. However, most structural ceramics are insulators at room temperature, though they possess excellent mechanical properties. For example, the surface and volume resistivity of zirconia reach up to 10^{14} Ω/□ and 10^{12} Ω·cm, respectively. There are only few studies on both the electrical and mechanical properties of such ceramics. Nakayama et al. reduced the surface resistivity of a structural ceramic to 10^4 Ω/□ and the volume resistivity to 10^2 Ω cm by doping it with 40 wt% ZnO, but the bending strength was only 60 MPa [3]. Fan et al. reduced the volume resistivity of zirconia to 2.1 Ω cm via an iron implanting method, but the microstructure was easily damaged, and the manufacturing cost was high [4]. TiO_2 ceramic is a semi-conducting material with an electrostatic dissipative function that is widely applied in the textile industry, yet the abrasion resistance and compactness are poor [5]. Other structural ceramics such as SiC, ZrC, TiN, and ZrN are expensive and require complicated synthesis techniques [6–8].

In this study, 3Y-TZP with both good anti-static and mechanical properties was prepared via a simple high-temperature infiltration

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method. The effects of infiltration time and temperature on the surface resistivity and hardness were investigated. The anti-static mechanism was also studied preliminarily.

2. Experiment

2.1. Materials and experimental procedure

3Y-TZP ceramic pieces with dimensions of 23×23×5 mm (prepared in the lab, density was 6.02 g/cm^3) and 50 mL alumina crucibles were washed and dried. Graphite powder (Qingdao Chenyang Graphite, chemical purity) and reduced Fe (Xilong Chemical, analytical purity) were dried in a vacuum drying oven. The Fe powder was placed in the bottom of the alumina crucible to a thickness of 5 mm, and the 3Y-TZP ceramic piece was then placed onto the Fe powder, followed by another 5 mm layer of the reduced Fe powder. Finally, graphite powder was placed on top to a thickness of 30 mm. The crucible was then heated in a resistance furnace at temperatures ranging from 900 °C to 1300 °C for 4 h. For samples heated to 1000 °C, the heating time was varied from 2 h to 10 h. The resultant devices were allowed to cool down to room temperature naturally inside the furnace. Lastly, the Fe coating was removed, leading to the anti-static zirconia sample.

2.2. Characterization

The surface resistivity was measured using the device shown in Fig. 1 and calculated using Eq. (1).

$$\rho_s = R_s \times \frac{b}{l} \tag{1}$$

Where, ρ_s is the surface resistivity; R_s, the surface resistance obtained from the ohmmeter; b, the length of the rectangular electrodes; and l, the distance between the rectangular electrodes. Conductive silver pulp was used to improve the good contact between the rectangular electrodes and ceramic surface.

The hardness of the zirconia ceramic was measured by a digital Vickers hardness tester (401MVD, Wolpert, the Netherlands). The phase composition was analyzed by an X-Ray Diffractometer (XRD, Rigaku D/max 2200X, Rigaku, Japan). The microstructure was observed by scanning electron microscopy (SEM, JSM-6700F, JEOL, Japan). The element valence states and chemical composition were analyzed by X-ray photoelectron spectroscopy (XPS, K-Alpha 1063, England).

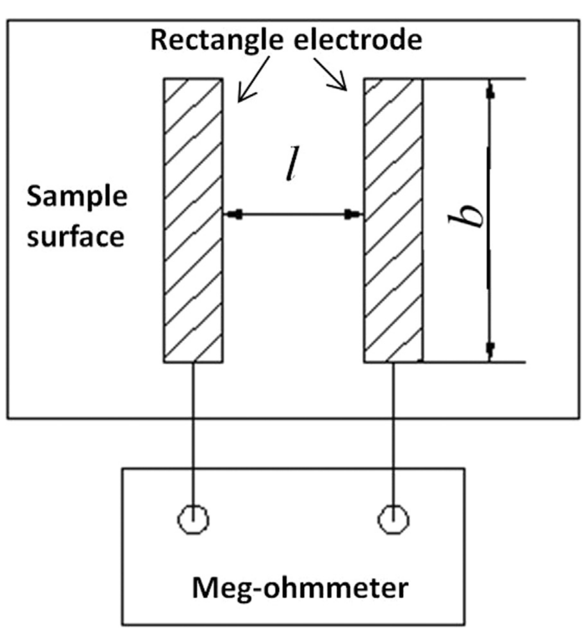

Fig. 1. Device for measuring the surface resistivity.

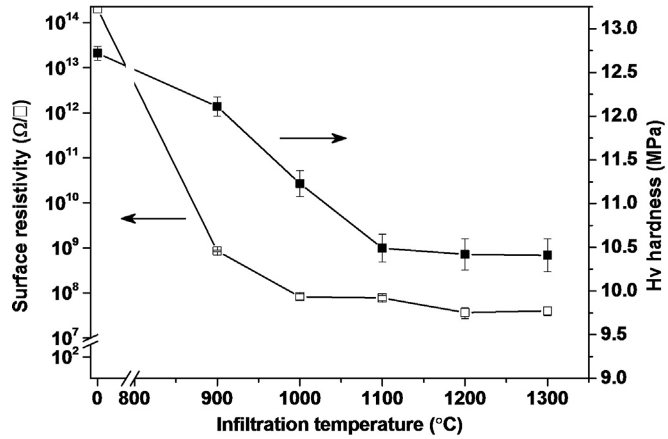

Fig. 2. Influences of infiltration temperature on the surface resistivity (open squares) and Vickers hardness (solid squares) of the 3Y-TZP ceramics.

3. Results and discussion

3.1. Influence of infiltration temperature on the surface resistivity and hardness

Fig. 2 depicts the influences of infiltration temperature on the surface resistivity and hardness of the 3Y-TZP ceramic. The figure shows that the surface resistivity of the traditional 3Y-TZP was higher than 10^{14} Ω/□. The surface resistivity decreased drastically to 10^8 Ω/□ at 900 °C. The surface resistivity of the sample heated to 1000 °C decreased further, reaching 10^7 Ω/□. Infiltration temperatures above 1000 °C did not induce further significant decreases in the surface resistivity.

Hardness comprehensively reflects the overall mechanical properties of materials such as elasticity, plasticity, strength, and toughness. Generally, higher hardness corresponds to higher strength in ZrO_2 ceramics [9,10]. Therefore, hardness was selected as the parameter to investigate the ceramic's mechanical properties. The influences of infiltration temperature and infiltration time on the hardness of the 3Y-TZP ceramic are also shown in Fig. 2. The hardness of ZrO_2 was 12.7 GPa before infiltration. After infiltration, the hardness decreased to 12.1 GPa at 900 °C and to 10.1 GPa at 1100 °C. However, the hardness further decreased only slightly at higher temperatures, which indicates that even after processing, this ceramic has mechanical properties superior to the values reported for Fe-infiltrated zirconia thus far [4].

3.2. Influence of infiltration time on the surface resistivity and hardness

Fig. 3 shows that both the surface resistivity and hardness of zirconia ceramic presented an overall declining trend with the extension of infiltration time. The variation of the surface resistivity was tight when the infiltration time was more than 4 h. This was because the iron element distributed on the surface of 3Y-TZP ceramic reached the saturation concentration by this time, so infiltrating the ceramic for longer times yielded little change. Hence, the surface resistivity did not decrease significantly when the infiltration time was longer than 4 h.

The rate of change of the hardness achieved the highest value between infiltration times of 2 h and 4 h. The hardness of the sample infiltrated for 4 h was 11.2 GPa, but evidently, it changed slowly after 4 h. On the whole, the hardness values of zirconia after infiltrations for various times were all lower, further decreases were small after times longer than 4 h, and the hardness values were all above 10 GPa. Taken