Ceramics International ■ (■■■■) ■■■–■■■

Contents lists available at ScienceDirect

Ceramics International

journal homepage: www.elsevier.com/locate/ceramint

Surface resistivity regulation of zirconia ceramics for anti-static purposes by novel solution infiltration method

Xianfeng Yang [a], Xiaole Yang [a], Wei Liu [b,*], Jianghong Yang [a], Xiewen Xu [a]

[a] College of Materials Science and Engineering, Changsha University of Science & Technology, Changsha, 410114 PR China
[b] School of Electromechanical Engineering, Guangdong University of Technology, Guangzhou, 510006 PR China

ARTICLE INFO

Article history:
Received 13 August 2016
Received in revised form
27 August 2016
Accepted 30 August 2016

Keywords:
C. Hardness
C. Impedance
D. ZrO_2
Infiltration

ABSTRACT

An innovative processing method for regulating the surface resistivity of zirconia ceramic has been developed based on solution infiltration in porous preform body. In this study, effects of infiltration solution concentration on the surface resistivity and Vickers hardness were investigated. It was shown that the surface resistivity decreased with the increase of infiltration concentration, while the surface hardness was not significantly influenced. For a typical sample infiltrated in 3 mol/L $Fe(NO_3)_3 \cdot 9H_2O$ solution for 2 h, the surface resistivity is reduced to $10^4\Omega/\square$, while the Vickers hardness was kept at 13 GPa. The content of element Fe decreased gradually from the surface to the core of the samples. The status of element Fe was verified as FeO and Fe_2O_3. About 49.4% of the element Fe was in the status of FeO, which was the conductive composition for anti-static purpose.

1. Introduction

Zirconium dioxide is one of the most studied structural ceramic materials for its excellent mechanical properties. Since the first paper “ceramic steel ?” published on the journal of Nature [1], numerous studies were focused on tetragonal polycrystalline zirconia doped with 3 mol% yttria (3Y-TZP), including the fracture toughness enhancement mechanism [2], preparation of zirconia toughening composites [3], ultrafine powder preparation [4] and advanced molding techniques [5]. However, few studies were related with the electronic properties of 3Y-TZP because it is known as an insulator at normal temperature.

If the resistivity of the 3Y-TZP could be reduced to the range of static dissipation or static conduction, the advantages of structural ceramic material's mechanical properties and the ability to dissipate static surface charges will be combined. Zirconia ceramics with anti-static properties will be a very promising candidate especially for the harsh environments, such as petrochemical processing, outer space for aerospace or high frequency friction.

According to U.S. statistics, annual loss due to electrostatic hazards caused up to more than 10 billion dollars just in the electronics industry. During IC manufacturing in semiconductor industry, anti-static materials and precaution must be taken because static voltage higher than 100 V will result in the damage of bonding point or oxidation film [6]. At present anti-static materials applied in engineering include polymer-based composite and antistatic glaze. Polymer-based anti-static materials or coatings are prepared by adding conductive materials like carbon nanotubes, graphite, carbon black or conductive fiber. Unfortunately, polymer-based composites exhibit poor durability and poor resistance to high temperature [7]. In anti-static glaze for traditional household or construction ceramic, semi-conductive or conductive oxide powder, conductive fibers are added [8]. This kind of anti-static ceramic have the main disadvantages of higher porosity and lower mechanical properties.

Though ZrO_2 ceramics showed excellent high temperature and ware resistance , it was difficult to keep the balance between electrical and mechanical properties in the previous studies. Susuma showed that 40 wt% ZnO can reduce the surface resistivity of Zirconia to $10^4\,\Omega/\square$, however the fracture strength was only 60 MPa [9]. In this study, surface resistivity of 3Y-TZP ceramics was regulated via solution infiltration for anti-static. Composition and microstructure evolve during processing and anti-static mechanism was discussed.

2. Experimental procedures

The spray dried zirconia powder (average particle size (d_{50}) of 0.16 μm and BET specific area of 8.3 m^2/g , grade YSZ-F-DM-3.0, Farmeiya Advanced Materials Co., Ltd, China) was cold isostatic pressed to disk-shaped green body (diameter 15 mm, thickness 8 mm). Then the green body was pre-sintered at 900 °C for 2 h to get the perform body for infiltration. The porosity of the obtained

* Corresponding author.
E-mail address: 317127238@qq.com (W. Liu).

http://dx.doi.org/10.1016/j.ceramint.2016.08.187
0272-8842/

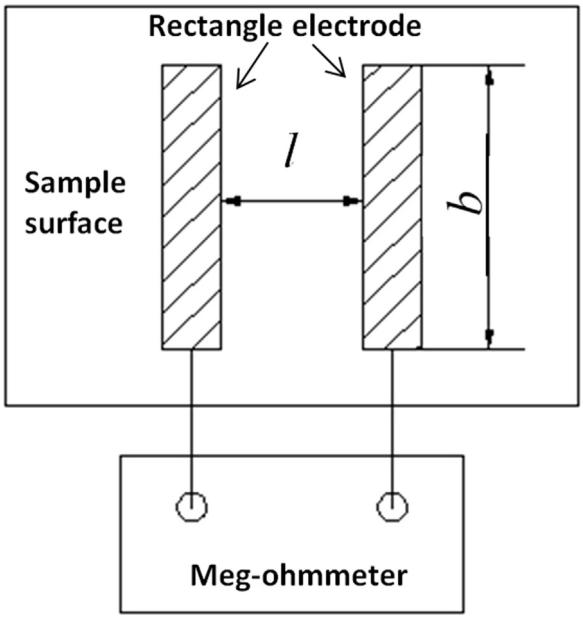

Fig. 1. Device for surface resistivity measurement.

perform was 45% and density 3.5 g/cm^3. The obtained preform was infiltrated in the $Fe(NO_3)_3 \cdot 9H_2O$ solution with the concentration 1 mol/L, 2 mol/L and 3 mol/L at 25 °C for 2 h. Then the infiltrated brown body was dried in the drying oven at 45 °C for 48 h.

Then the dried infiltrated body was embedded in the carbon powder in the Al_2O_3 crucible. Then the crucible was put into the furnace for sintering with heating rate at 3 °C/min and holding time at 1500 °C for 2 h.

Surface resistivity was measured on the devices shown in Fig. 1 and calculated by Eq. (1).

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

ρ_s, surface resistivity; R_s, surface resistance, value on the ohmmeter; b, length of the rectangle electrode; l, distance of the rectangle electrode.

Hv hardness was measured through indentation method (401MVD, Wolpert, America) at pressure of 98 N. Microstructure was characterized by scanning electronic microscope (Quanta FEG 250, FEI company, America). Xray photoelectron spectroscopy measurements (XPS, K-Alpha 1063, Thermo Scientific, England) were carried out to analyze the status of the infiltrated elements.

3. Results and discussion

3.1. Influence of solution concentration on the surface resistivity and Vickers hardness

Fig. 2 demonstrates the influences of solution concentration on the surface resistivity of zirconia ceramic. It is known that the surface resistivity of zirconia ceramic without infiltration was higher than 10^{14} Ω/□ and is the typical insulator, which is also in accordance with the intrinsic value of 3Y-TZP in previous literatures [10]. When the perform was infiltrated by the 1 mol/L solution for 2 h, the surface resistivity decreased rapidly to the magnitude of 10^7 Ω/□. A more concentrated solution at 2.0 mol/L significantly reduced the surface resistivity to ~10^6 Ω/□. A further increase in concentration to 3.0 mol/L leads to an even lower surface resistivity of 10^4 Ω/□. The results indicate that the conductivity of 3Y-TZP was significantly enhanced after infiltration-

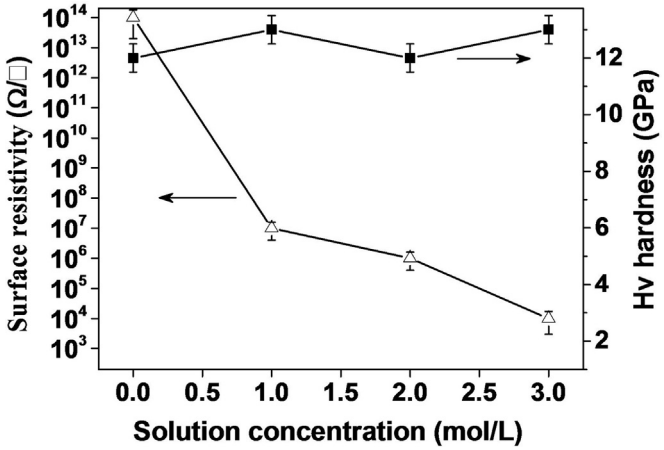

Fig. 2. Influence of solution concentration on surface resistivity and relative infiltration content.

doping treatment, making it more suitable for anti-static use. Meanwhile, the Vickers hardness of the 3Y-TZP was not been influenced remarkably and kept at 12–13 GPa.

Surface resistivity reduction was from the existence of element Fe by infiltration. During infiltration, solution containing Fe^{3+} was sucked into the pre-sintered part gradually by the capillary force till all the pores was saturated. The relative infiltration was characterized and shown in Fig. 3. It was obviously that more Fe element was sucked into the pre-sintered part with higher concentration solution for the preform with certain pores volume and diameter distribution. Cross section of the infiltrated body after different infiltration time was also shown in Fig. 3. The infiltrated and uninfiltrated part was marked obviously by the brown lines. The color of the infiltration reached area was the same as the $Fe(NO_3)_3$ solution and uninfiltrated zone was still white. It is shown that the infiltration front could approach the center of the brown body just in 40 s.

The microstructure and composition evolution was shown in Fig. 4. Fig. 4(a) demonstrates that the preform was porous and pores were formed between the ceramic particles. After sintering, the pores were eliminated and the ceramic parts became dense (Fig. 4(b)). During infiltration and drying, element Fe containing solution probably migrated along the interconnected pore network driven by the capillary force. Areas with different distance to the surface were selected, i.e. at surface, middle or core in the sintered parts (Fig. 4(c)) for EDS analysis. The results of EDS were

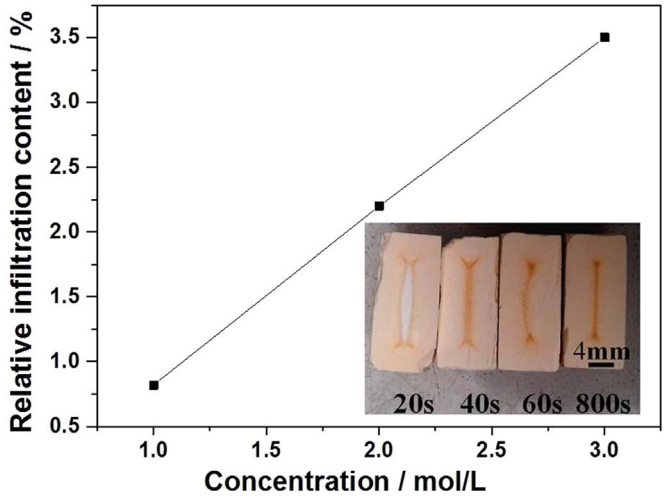

Fig. 3. Relative infiltration content and cross section photograph of the infiltrated body.

Please cite this article as: X. Yang, et al., Surface resistivity regulation of zirconia ceramics for anti-static purposes by novel solution infiltration method, Ceramics International (2016), http://dx.doi.org/10.1016/j.ceramint.2016.08.187