Non-Conductive Welding Pins Based on Zirconia Ceramics

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Abstract

High strength, high toughness ceramics based on zirconia have been developed for use as nonconductive welding pins. Three classes of ceramics have been manufactured each with different combinations of mechanical properties. One class of ceramics has a high fracture toughness of ~15.7 $MPa.m^{1/2}$ and a flexural strength of 780 MPa. The second class has fracture toughness of 6.6 $MPa.m^{1/2}$ and a flexural strength of ~1155 MPa. The third class has a moderately high fracture toughness of ~12 $MPa.m^{1/2}$ and an intermediate ratio of tetragonal and monoclinic phase) were found to be the key factors in determining the mechanical properties.

Introduction

Phase transformation from tetragonal to monoclinic ZrO_2 provides a powerful means in strengthening and toughening ZrO_2 based ceramics [1, 2]. Many dopant cations including Ca^{2+}, Mg^{2+}, Y^{3+}, Ce^{4+} etc. have been introduced into the structure of ZrO_2 at different levels forming a series of fully or partially stabilized ZrO_2 ceramics (PSZ). Examples are Ca-PSZ, Mg-PSZ, Mg/Ca-PSZ, Y-PSZ [3-6] and single phase t-ZrO_2 polycrystals (TZP) such as Y-TZP and Ce-TZP [7, 8]. In recent years, more emphases have been focused on Y doped and Ce doped ZrO_2 due to potentials they offer in obtaining high strength, high toughness, or improved thermal stability for various applications. Usually, doping with Y_2O_3 imparts high strength whereas CeO_2 addition offers high fracture toughness. For instance, hot-isostatic pressed yttria-doped zirconia polycrystals (Y-TZP) exhibit flexural strength as high as 1500 MPa with approximately 6 mol% of $YO_{1.5}$ while the highest fracture toughness of 12 $MPam^{1/2}$ is achieved with ~4mol% $YO_{1.5}$ addition [9,10]. Normally, in this type of ceramics, the increase in toughness is followed by a decrease in strength and vice versa. For example, fully stabilized zirconia with 12mol% CeO_2 leads to a fracture toughness of 40 $MPam^{1/2}$ whereas the flexural strength drops to 525 MPa [11,12]. This toughness is comparable to or even better than that of aluminum alloys showing a great potential for structural applications provided that its strength is increased to a levels closed to 1000MPa. This paper presents the characterization results of three classes of Ce-PSZ ceramics having different combination of strength, toughness and hardness.

Experimental

Three classes of Ce-PSZ ceramic samples designated as FMM-1, FMM-2 and FMM-3 were supplied by Functional Materials Manufacturing Inc. (FMM) and were produced by pressureless sintering technique. The samples were cut, ground and polished into rectangular bars with a dimension 3mm x 4mm x 40mm for four-point bend strength test measurements and with dimensions 3mm x 4mm x 6mm for hardness and toughness tests. The flexural strength measurements were done on an Instron Testing Machine 8505 with a loading rate of 0.018mm/min. The outer and inner spans of the jig used for flexural strength measurements were 23.8mm and 13.0mm, respectively. The strength is calculated according to the equation:

$$\sigma = \frac{3Pa}{bh^2} \qquad (1)$$

where σ is the strength, P is the fracture load, **a** is the distance between the outer span and inner span, **b** is the specimen width, and **h** is the specimen thickness.

Fracture toughness and hardness tests were done using a Mituya Hardness Tester (AVK-210) with a load of 30kg and 10kg, respectively. The load was held for 10 seconds. The fracture toughness was calculated using the following equations [13]:

$$\left(\frac{K_{IC}\Phi}{H_v a^{1/2}}\right)\left(\frac{H_v}{E\Phi}\right)^{2/5} = 0.035\left(\frac{l}{a}\right)^{-1/2} \qquad \text{for } 0.25 \le l/a \le 2.5 \tag{2}$$

$$\left(\frac{K_{IC}\Phi}{H_v a^{1/2}}\right)\left(\frac{H_v}{E\Phi}\right)^{2/5} = 0.129\left(\frac{c}{a}\right)^{-3/2} \qquad \text{for } c/a \ge 2.5 \tag{3}$$

where K_{IC} is the mode I critical stress intensity factor ($MPam^{1/2}$), H_v is the Vickers hardness (N), E is the Young's modulus ($2.0x10^{11}$ for zirconia), c is the radius of the surface crack, **a** is the half-diagonal length of the Vickers indent, P is the load of indenter, **l** is the parameter defined as **c-a,** and Φ is defined as ratio of hardness H_v to uniaxial yield stress σ, normally taken to stay constant (2.7 – 3) and is taken to be 3 for zirconia. Phase identification was performed using Miniflex x-ray machine on as-sintered surface and fracture surface after bending test. The volume fraction of monoclinic phase, V_m, was calculated using the following equation [14]:

$$V_m = 1.311X_m/(1+0.311X_m) \tag{4}$$

$$X_m = [I_{(\underline{1}11)\,m} + I_{(111)m}]/\,[I_{(111)t} + I_{(\underline{1}11)m} + I_{(111)m}] \tag{5}$$

where X_m is the integrated intensity ratio and the subscripts m and t represent the intensity of $K_{\alpha 1}$ for monoclinic and tetragonal phases, respectively, after the peak separation and fitting. Microstructure analysis was carried out using a JOEL-2800 scanning electron microscope on polished and fracture surfaces after bending test.

Results and Discussion

The results of flexural strength, fracture toughness and hardness of Ce-PSZ materials are presented in Table 1.

Table 1 Mechanical properties of Ce doped Zirconia ceramics

Samples	Mechanical properties		
	Strength (MPa)	Fracture toughness ($MPam^{1/2}$)	Hardness (GPa)
FMM-1	1155 ± 122	6.6 ± 0.1	10.7 ± 0.1
FMM-2	781 ± 14	15.7 ± 0.9	8.2 ± 0.1
FMM-3	840 ± 8	12.0 ± 1.0	8.7 ± 0.2

As can be seen from Table 1, these materials show different characteristics in properties and thus offer the opportunities for different applications as functional and structural parts. For example, the FMM-1 ceramic shows a very high strength, an intermediate hardness, and relatively low fracture toughness. The FMM-2 ceramic gives relatively low strength and hardness but high toughness. Finally, the FMM-3 ceramic exhibits intermediate strength and fracture toughness. The combination of mechanical properties observed in these ceramics, especially the combination of high strength and high fracture toughness (e.g for FMM-3) indicate that these materials are highly reliable and have potentials for use in a new high performance engineering applications.

In order to correlate the mechanical properties of these ceramics qualitative analysis on the as-sintered and the fracture surfaces of Ce-PSZ were performed using scanning electron microscopy in combination with Image Tool 3.0. The SEM micrographs are presented in Figure 1. The micrographs show that all materials have fine grain microstructures with uniformly distributed relatively small pores of less than 1μm in diameter. The grain size Image Tools 3.0 show that The FMM-1 ceramics have the finest average grain size of ~0.5μm, whereas both FMM-2 and FMM-3 have somewhat larger grain size with an average value of approximately 1.0μm. It is worth noting that the FMM-1 not only has smaller grains but also shows a more homogeneous microstructure than the other two investigated. It is believed that these two factors make contributions to the high strength observed in FMM-1 ceramics although other factors such as processing

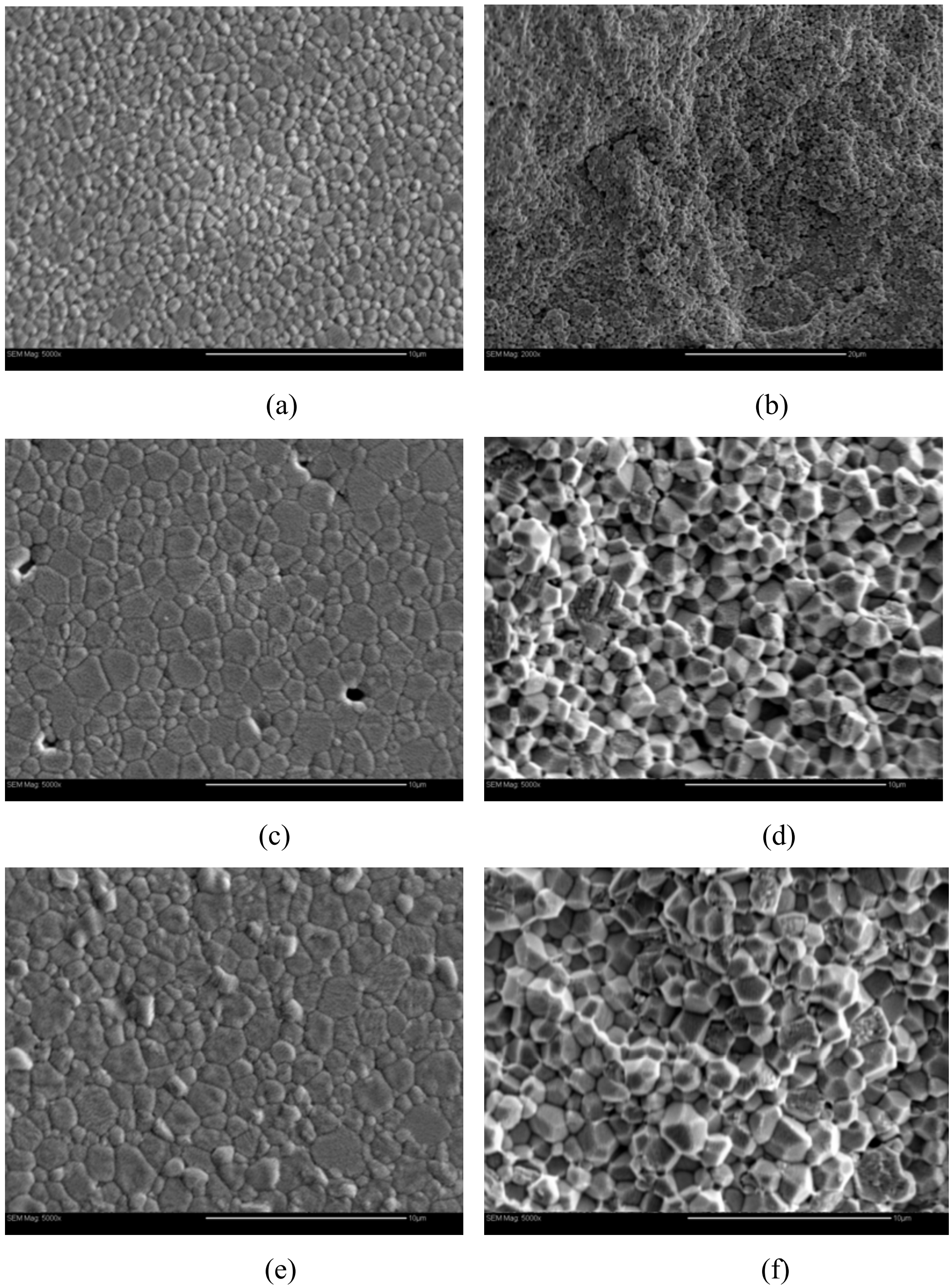

Figure 1 scanning electron micrographs of Ce-PSZ ceramics.

(a) polished surface (5000x) and (b) fracture surface (2000x) for FMM-1

(c) polished surface (5000x) and (d) fracture surface (5000x) for FMM-2

(e) polished surface (5000x) and (f) fracture surface (5000x) for FMM-3 parameters, compositions and phase composition could make a difference in strength values.

It is well known that the major toughening mechanism in partially or fully stabilized ZrO_2 ceramics is the stress-induced transformation from tetragonal to monoclinic phase. To identify the mechanism and the level of toughening, qualitative analysis of the dependence of toughness on the amount of tetragonal ZrO_2 was measured on the as-sintered surface and on fracture surface by x-ray technique. The results show that all Ce-PSZ ceramics investigated demonstrated a similar transformation behavior except that the amount of the transformation from tetragonal to monoclinic phase is different. Figure 2 gives typical x-ray diffraction patterns showing the phase change on the as-sintered and fracture surfaces of the FMM-2. It can be seen from Figure 2 that the as-received samples contain mostly tetragonal phase with some small amount of monoclinic phase. However, after fracture the amount of monoclinic-ZrO_2 increased markedly and on the expense of the tetragonal phase.

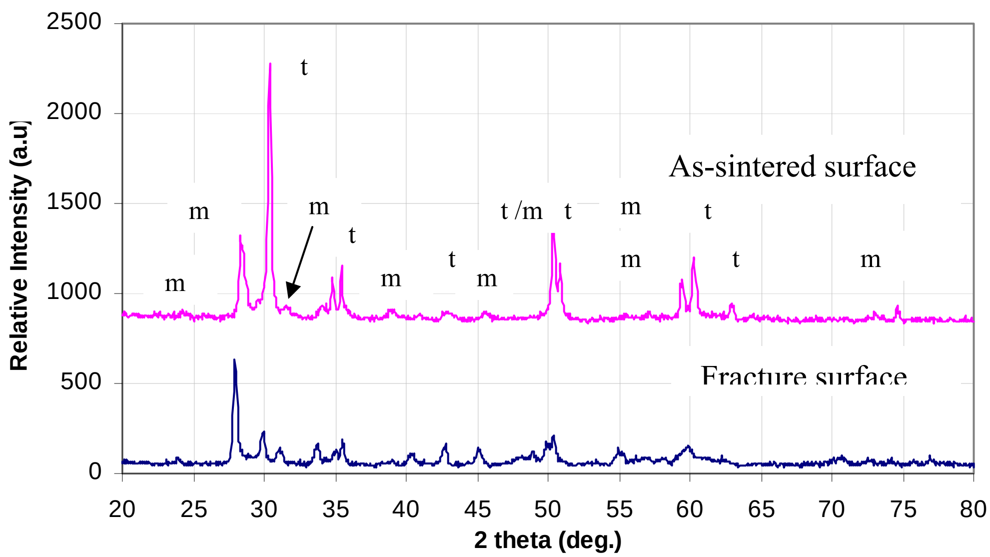

Figure 2 X-ray diffraction patterns of FMM-2 Ce-PSZ ceramics. t and m stand for tetragonal and monoclinic ZrO_2 phases respectively

The relative amounts of the t- and m-ZrO2 phases as determined by x-ray qualitative analysis are given in Table 2.

Table 2 Relative content of t- and m-ZrO_2 phases in as-sineterd sample and after fracture

ZrO2 materials	Volume % of t- and m-ZrO2 phase (%)				Transformability of t-ZrO_2
	As-sintered surface		Fracture surface		Volume % of transformed t-ZrO2
	t	m	t	m	
FMM-1	86.4	13.6	71.6	28.4	17
FMM-2	71.1	28.9	41.9	58.1	41
FMM-3	72.2	27.8	42.2	57.8	41

Examination of Table 2 shows that the volume fraction of the tetragonal phase in as-sintered FMM-1 is ~15% higher than the value fraction of the tetragonal phase in FMM-2 and FMM-3, the latter two showed a similar amount of t-ZrO_2 in as-sintered samples. However, the volume fraction of tetragonal phase in the fracture surface in FMM-1 is 30% higher than that in FMM-2 and FMM-3, suggesting a poor transformability of the t-ZrO_2 in FMM-1 compared to that in FMM-2 and FMM-3. It is believed that, apart from the difference in compositions which eventually control the stability of the partially stabilized ZrO_2, the smaller grain size in FMM-1 may play some roles in restraining t- to m-ZrO_2 transformation especially when fine grain size reaches a critical value. The transformability of t-phase in partially stabilized ZrO_2 is a key factor in determining the toughness of this kind of materials because the toughness is highly dependent on the stress induced transformation mechanism. In the present study, the lower toughness observed in FMM-1 materials is believed to be mainly caused by the poor transformability of t-ZrO_2 even though the initial volume of the t-ZrO_2 in the sample is high. This is further evidenced by the fact that the high toughness obtained in FMM-2 and FMM-3, in which much larger volume (~41%) of t-ZrO_2 transformed into m-ZrO_2 during fracture, which is believed to be the cause for significantly larger fracture toughness obtained in these two materials.

The nonconductive welding pins made from the three Ce doped ZrO_2 materials have been put into test and the initial results showed that all the pins demonstrated an excellent performance in practical welding operations. The evaluation of the difference in performance and service live of these materials is under way.

Conclusions

Three classes of Ce doped ZrO_2 ceramics have been characterized on mechanical properties and their potential use as nonconductive welding pins was evaluated. One class of ceramics has high flexural strength (1155 MPa) and lower fracture toughness (7 $MPa.m^{1/2}$). The second class has high fracture toughness (~15.7 $MPa.m^{1/2}$) and a flexural strength of 780 MPa. The third class has moderately high fracture toughness (12 $MPa.m^{1/2}$) and an intermediate strength (840 MPa). Fine-grain sized microstructure and phase transformation are the main strengthening and toughening mechanisms in these materials. Nonconductive welding pins made from the three classes of ceramics were tested in welding operations and showed to have different performances depending on the level of toughness and strength. The composition and microstructure (grain size and the relative ratio of tetragonal to monoclinic phase) were found to be the key factors in determining the mechanical properties and hence the performance in welding operation.

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