

Development and Characterization of Al₂O₃/ 3Y-TZP for Cutting Tools

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Abstract: Science and technology are advancing at a rapid pace and the ways in which they interact with economy, society and environment are becoming increasingly complex. Within this context, the ceramic materials become solution for some applications. Therefore the alumina ceramics are promising materials advanced. The alumina based ceramics are wear-resistant, corrosion-resistant and lightweight materials, and are superior to many materials with regard to stability in high-temperature environments. Another available class of ceramic is based on zirconia which exhibits very high strength and good toughness but shows relatively poor hardness. The strength and toughness of an alumina ceramic may be enhanced by incorporating tetragonal zirconia particles in alumina matrix. In Al₂O₃ matrix the t-ZrO₂ grains undergo the known tetragonal to monoclinic phase transformation due mainly by stress induced, and microcracks form around existing m-ZrO₂ grains (microcrack formation). The objective of this work is to develop materials with mechanical properties to apply as cutting tools. For this, ceramic cutting inserts produced from Al₂O₃/3Y-ZrO₂ nano (AlZ) and 3Y-ZrO₂ nano/Al₂O₃ (ZTA) systems have been investigated with the objective to analyse the effect of 3Y-ZrO₂ nano addition on mechanical properties. The two compositions studied were developed by mixture route and compacted by cold uniaxial pressing and sintered at 1600°C. The results showed an appropriate balance among the Vickers hardness, fracture toughness and densities (>98%) which enable the employment of these ceramic as cutting tool inserts.

Keywords: Al₂O₃/3Y-TZP, cutting tools, advanced ceramic, microstructure, mechanical properties

1. Introduction

The ceramics materials have been widely researched for structural applications because of the superior physical and mechanical properties when compared to metals at elevated temperature [1-2]. These materials constitute important properties in high temperature application. Special condition is applied to ceramics cutting tools which constitute the major important part in machining process. Machining is the most widespread metal shaping process in mechanical manufacturing industry. All over the world, machining operations such as turning, milling, boring, drilling and shaping consume large amount of money annually [3]. Out of these machining processes, turning still remains the most important operation used to shape metals because in turning, the conditions of operation are most varied. Increasing productivity and reducing manufacturing cost have always been keys to successful business [4]. The ceramics cutting tools can promote dry machining with consequent reduced infrastructure, lower costs and a cleaner environment, compared to today's practice of wet machining.

The Al₂O₃/ZrO₂ and ZrO₂/Al₂O₃ composites have excellent strength, toughness and heat-shock property, and it is used to prepare mashed materials for fine forming, cutting tools and bearing balls and so on. Due to their unique properties (high toughness, high-mechanical strength, corrosion resistance, fracture resistance, etc.), zirconia and alumina based ceramics have attracted special attention in the last two decades [5-8]. Zirconia, unlike many other ceramics, undergoes a martensitic phase transformation with the temperature according to: monoclinic (m) up to ~1240 °C, tetragonal (t) to 2380 °C, and beyond this cubic (c) [9-10], [11-13]. The most destructive is the transformation of tetragonal to monoclinic phase resulting in approximately 3–5% volume change. On the other hand, Al₂O₃ has very high

hardness and good strength but relatively low toughness in comparison to zirconia, which exhibits very high strength and good toughness but shows relatively poor hardness. The strength and toughness of alumina may be enhanced by dispersion of zirconia nanometric inclusions, mainly due to the tetragonal–monoclinic transformation [14-17]. Thus, toughness of Al₂O₃-ZrO₂ composites is improved by both stress-induced phase transformation toughening and microcrack toughening of dispersed zirconia. The main objective of this work was to study the mechanical properties of two composite materials which are prepared based on the ZrO₂ reinforced with alumina particles, ATZ, and alumina reinforced with zirconia particles, ZTA.

2. Experimental procedure

As starting powders, Al₂O₃ (99,5 % α), OP-1000, Almatiss, Alcoa and nano t-ZrO₂ stabilized with 3mol% Y₂O₃, containing 10 % of residual monoclinic phase, Shandong Zhongshun Sci. & Tech. Devel. Co. Ltd., were used. Powder batches of Al₂O₃ and 3% mol yttria-stabilized zirconia, as well as homogeneous mixtures of different percentages this materials were carefully mixed in an agate mortar and to avoid any variations that can occur due to relative humidity changes. Two compositions containing 18,5 and 30% (weight) ZrO₂ the matrix of Al₂O₃, Were compacted and sintered at 1600 °C.

The compaction experiment was conducted by using steel die square of 16 x 16 x 6 mm. About 4.5g of the powder were poured into the die cavity and uniaxial pressed at a pressure of 80MPa (pressure for crack-free samples). Sintering was carried out in air, at 1600 °C. The sintering cycle consisted in heating up to 1100 °C with a heating rate of 10 °C/min, up to 1400 °C with 5 °C/min and then up to 1600 °C with 3 °C/min. The sintering time at 1600 °C was 180 min. The

cooling rate to 1400 °C was 5 °C/min and to 1100 °C was 3 °C/min, below that the furnace was switched off. Relative density of the sample was measured by the Archimedes method. The samples were ground and polished by means of 0.25µm diamond paste and subsequently polished. Samples were characterized by Scanning Electron Microscopy (SEM: JEOL JSM - 531, Japan) using an accelerating voltage of 2–20kV after the carbon coating to avoid charging during exposure to the electron beam. Identification of phases was carried out by X-ray diffraction technique (XRD), using a diffractometer model PW1380/80 Philips, equipped with Cu K α monochromatic radiation and θ –2 θ geometry. The data was collected on the 2 θ range from 10° to 90° at intervals of 0.05° 2 θ using a step-counting time of 10s. Vickers hardness measurements were made by using a microhardness Tester Wilson Tukon, model 1798. Indentations were made on polished surfaces with a load of 2 kg held for 10 s. Approximately 10–20 indents were made for each composition and the average hardness was determined. The corresponding indentations sizes and crack lengths were determined using an optical microscope. The fracture toughness (K_{IC}) for a ratio $c/a > 2.5$ (present study), where c is the crack length and a is the half diagonal length of the indentation impression, is calculated using the following equation [18]:

$$K_{IC} = \frac{0.0752.P}{\frac{3}{c^2}} \quad (1)$$

Where: K_{IC} is the fracture toughness, P the load and c the crack length.

3. Results and discussion

Composition of phases of the “as-received” 3Y-TZP nano powder was investigated by means of XRD and the respective pattern is illustrated in Figure. 1a and 1b. It is observed that the main phase is tetragonal (t) as well as few amount of monoclinic (m) phase corresponding to reflections $2\theta=28^\circ, 31^\circ$ and commercial alumina (α -Al $_2$ O $_3$) purity 99.50 %; mean particle diameter 0.5 µm.

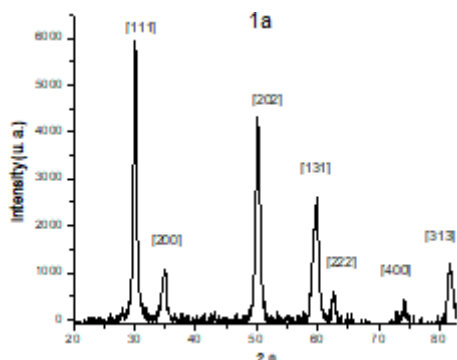


Figure 1 (a): XRD pattern of powder: 3Y-TZP nano

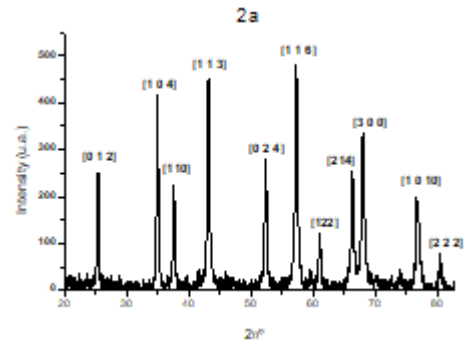


Figure 1(b): XRD pattern of powder Al $_2$ O $_3$

3.1 Sintering

After the two samples were sintered at 1600°C, the relationships of the linear shrinkage, weight loss and relative density were shown in Table 1. The sintered materials presented relative densities higher than 98% of the theoretical density, independently of the majority phase. This reduction of the porosity contributes in obtaining high resistant, reliable ceramic for cutting tools applications. Linear shrinkage, as well as weight loss, during sintering of the samples are due to volatily organics materials. No considerable variations were observed as function of kind materials.

Table 1: Shown the results of the sintered samples, at 1600°C: ATZ and ZTA.

| Samples | Linear shrinkage (%) | Relative density (%) | Weight loss (%) |
|---------|----------------------|----------------------|-----------------|
| ATZ | 22.4 ± 0.7 | 14.5 mm (0.58 in) | 2.6 ± 0,18 |
| ZTA | 21.3 ± 0.8 | 13 mm (0.51 in) | 1.9 ± 0,15 |

3.2 Phase analysis

The X-ray diffraction patterns of the samples sintered at 1600 °C, for two different compositions, are shown in Figure. 2a and 2b. The sintered samples presented only α -Al $_2$ O $_3$ and tetragonal-ZrO $_2$ as crystalline phases, independently of the composition. The absence of monoclinic m-ZrO $_2$ (present in the 3Y-ZrO $_2$ nano starting powder) shows that all the ZrO $_2$ phase transformed into the tetragonal phase, improving the mechanical properties of the composite material. In the Figure 2b (ZTA), are is observed a reflection in $2\theta = 30^\circ, 50^\circ, 60^\circ$ and 74.67° corresponding to the tetragonal phase. This results to suggesting a full stabilization of the tetragonal phase. Full stabilization of tetragonal phase has been reported by Lange [19] in ZrO $_2$ +3 mol% Y $_2$ O $_3$ which is in agreement with results this present work. However, the present results confirm that ceramics composites sintered above 1600 °C can exhibit a dual microstructure, formed by α -Al $_2$ O $_3$ and tetragonal grains. Considering that the monoclinic \rightarrow tetragonal phase transformation occurs during the final sintering stage, it is important to highlight that the intensity of peaks from tetragonal phase present in the starting powder is approximately the same (See Figure 1a). In this context, it is confirmed that during all sintering process additional monoclinic \rightarrow tetragonal phase transformation was developed.

In the Figure 2a, the XRD patterns of ATZ ceramics containing 18.5 wt % ZrO_2 are almost the same (Figure. 1a and 1b). The ZrO_2 particles are mainly of tetragonal phase embedded in $\alpha-Al_2O_3$. The main peaks of t- ZrO_2 are sharp and high. However, t- ZrO_2 retention becomes much easier to trigger the transformation to monoclinic. As a result, its contribution to transformation toughening predominates. However, fracture toughness value reduces abruptly when zirconia grain size is over a critical size [20] and the ease of transformation inhibited above the critical size. The coexistence of tetragonal (t) and $\alpha-Al_2O_3$ (α) phase can be confirmed by means of Scanning Electron Microscopy (SEM).

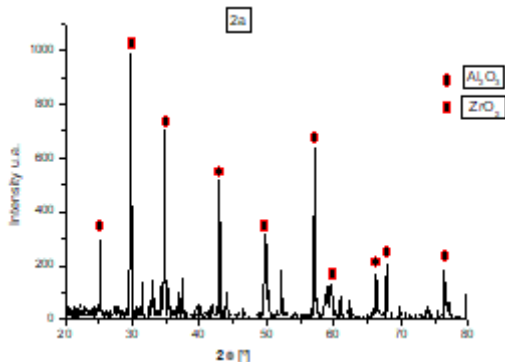


Figure 2a: XRD patterns of ATZ ceramics containing ZrO_2 - Al_2O_3 (18.5 and 81.5 wt %)

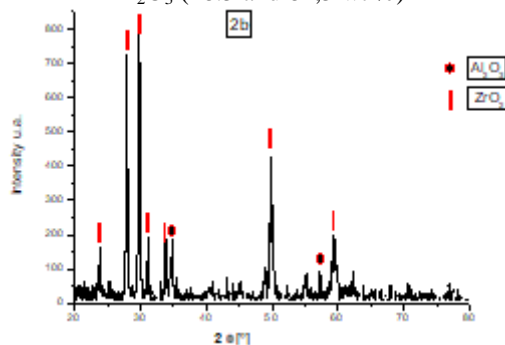


Figure 2b: XRD patterns of ceramics containing Al_2O_3 - 3Y- ZrO_2 (70 and 30 wt%).

3.3 Microstructure

The sintered microstructures consisted of zirconia (bright) and alumina (black) grains (Figure. 3a and 3b). The sintering process condition of the two powder systems was the same. The ZrO_2 has an action of transformation toughening on Al_2O_3 matrix [21]. There is a little solid solubility between Al_2O_3 and 3Y-TZP nano. The Al^{3+} (0.57 Å), Zr^{4+} (0.87 Å) and Y^{3+} (1.015 Å) form interpenetration boundaries which can hinder Al_2O_3 growth. In addition, little 3Y-TZP nano has an action of pinning Al_2O_3 boundary because of the smaller yttria particle size compared to the Al_2O_3 powder used. In the Figure 3a and 3b, shows images of fracture surface of the Al_2O_3 /18.5 vol.% 3Y-TZP nano and 3Y-TZP nano /30 vol.% Al_2O_3 composites sintered at 1600 °C for 3h. As shown in Figure. 3b, Al_2O_3 and ZrO_2 grain distribute homogeneously, and the grain size is obviously smaller than in Figure. 2a, which indicates that grain growth suppression action of ZTA is stronger than ATZ. In view of the fact that t- ZrO_2 is predominantly retained in the investigated materials, transformation toughening is considered to be the

predominant toughening mechanism in the ZTA composite. While explaining the transformation toughening contribution to the total toughness of the 30 vol.% Al_2O_3 composites, the presence of residual stress coupled with the influence of different microstructural variables, such as the t- ZrO_2 grain size, the Al_2O_3 content and the Al_2O_3 distribution on the transformability of the t- ZrO_2 should be considered.

In the Figure 2a (ATZ), the zirconia inclusions are distributed uniformly within the composites. The ZrO_2 particles of the t-phase, are mainly located at the grain boundaries of alumina, so the microstructure of alumina is thus refined due to the pinning effect exerted by the zirconia particles [22]. Comparing, Figure. 3a and 3b, can be observed the size of alumina grains in the t- ZrO_2 containing composites (ZTA) is smaller than that in the Al_2O_3 /3Y- ZrO_2 composites (ATZ), indicating that the presence of a small amount of Y_2O_3 , the stabilizing agent for ZrO_2 , can further prohibit the grain growth of alumina. Though the ionic charge of yttrium is the same as that of aluminum, the yttrium ion is much larger than the aluminum ion [23]. Large yttrium ions tend to segregate at the grain boundaries of alumina, thus reducing elastic strain energy [24]. Although the solubility of yttrium in alumina is extremely low (<10 ppm), [25] large yttrium ions can block the diffusion of ions along grain boundaries, leading to reduced densification and grain growth rates [26]. Though the yttria content in the composites is low, the amount is high enough to suppress the coarsening of alumina matrix grains.

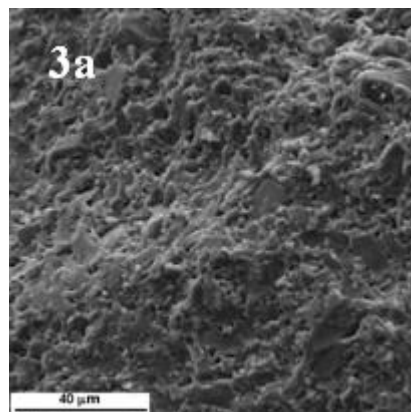


Figure 3a: Micrograph of the ATZ samples sintered

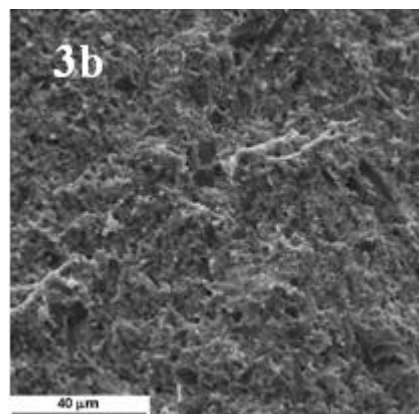


Figure 3a: Micrograph of the ZTA sample sintered.

3.4 Mechanical properties

Considering that the samples of $\text{Al}_2\text{O}_3/18.5\text{vol.}\% \text{ 3Y-ZrO}_2$ (ATZ), at 1600°C and 180min presented the best Vickers hardness, the samples of $3\text{Y-ZrO}_2/30 \text{ vol.}\% \text{ Al}_2\text{O}_3$ (ZTA) had been sintered in the same conditions and presented the best fracture toughness. It is observed that the samples presented relative density up to 98 %, indicating that the temperature of 1600°C for 180min was enough for activation of the sintering mechanisms with almost total elimination of the porosity. In the ATZ sample was observed that the 18.5wt % 3Y-ZrO₂ addition, reaching values of 16.20 GPa. The ZTA sample produces an increasing in the fracture toughness of 6.2 to 7.2 MPa.m^{1/2} due to the presence of t-ZrO₂ (minoritary).

The higher fracture toughness presented by ZrO₂ is result of the martensitic transformations during crack propagation. Beside of martensitic transformation, in t-ZrO₂-Al₂O₃ sample, the presence of the residual stress between two phases, contributed for the increasing of fracture toughness. The reduction in the fracture toughness of the sample ATZ having alumina (majoritary) is likely caused by a decreasing in volume fraction of transformable t-ZrO₂ phase and an increasing in grains size population due to parameters sintering.

4. Conclusions

From evaluation of the microstructure and mechanical properties of the 30 wt % Al_2O_3 (ZTA) sample (more dense samples), it was found that this composite exhibited higher fracture toughness than 18.5 wt % ZrO_2 (ATZ) sample. In fact, the composites with high fracture toughness are suitable in the system alumina toughened zirconia (ZTA) while sample with high hardness and relatively low fracture toughness belong to zirconia toughened alumina (ATZ). The grain growth of alumina and zirconia during sintering has shown an effect on the microstructure and mechanical properties of ZrO_2 reinforced with Al_2O_3 particles (ZTA) and Al_2O_3 reinforced with ZrO_2 particles (ATZ). The Al_2O_3 reinforced with ZrO_2 particles (ATZ), shows a higher Vickers hardness value (16.20 GPa) and fracture toughness ($6.2 \pm 0.45\text{MPa m}^{1/2}$) while that ZrO_2 reinforced with Al_2O_3 particles (ZTA), shows a intermediate Vickers hardness value (15.04 GPa) which makes this ceramic proper for cutting tools applications.

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