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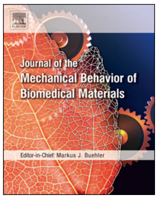

Effect of grinding on subsurface modifications of pre-sintered zirconia under different cooling and lubrication conditions

P. Suya Prem Anand, N. Arunachalam*, L. Vijayaraghavan

Department of Mechanical Engineering, Indian Institute of Technology Madras, Chennai, India

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A B S T R A C T

Pre-sintered zirconia is preferred as a restoration material in dental applications due to its excellent strength and fracture toughness. When abrasive processes were used to obtain the required shape of (Y-TZP) yttria-stabilized tetragonal pre-sintered zirconia, it resulted in material strength degradation in the presence of coolant. Therefore, experiments were carried out on pre-sintered zirconia with diamond grinding wheel to evaluate the performance of cooling conditions such as dry, wet and minimum quantity lubrication (MQL). The effects of different environments on the grinding performance were studied based on the temperature distribution, phase transformation, flexural strength, microhardness and edge chipping damage. The Raman spectroscopy and X-ray diffraction analysis were used to estimate the quantity of monoclinic phase in pre-sintered zirconia. The temperature rise of the workpiece material during the grinding experiment was not higher and insufficient to cause the thermal stresses. The microstructural changes induced by grinding under different cooling strategies were associated with the quantitative assessment of monoclinic phase. The flexural strength of ground components was improved in the dry condition compared to the other process due to the absence of the defective layer and the occurrence of Y^{3+} ions segregation. After grinding, there was a slight decrease in the hardness value by (1–8 HV), which was due to the formation of microcracks in the subsurface layer of the ground surface. In addition, to ensure the presence of microcracks, the edge chipping depth was measured. The damage depth obtained from the wet condition showed a higher value of 30 μm compared to the dry and MQL conditions.

1. Introduction

Zirconia has been used as an artificial material in the dental field for the manufacture of fixed partial dentures and dental crown. Zirconia based ceramics are routinely used in the engineering structural applications such as fabrication of gas sensors, cutting tools, refractories and biomedical implants. The yttria stabilized zirconia (3 mol%) in sintered and pre-sintered forms are separately used for different purposes in biomedical applications due to their biocompatible nature and fracture toughness. The grindability of pre-sintered zirconia is easier compared to the sintered blocks, due to the low hardness and high material removal rate of pre-sintered zirconia. The use of pre-sintered blocks finds more application in fixed dentures, where the abrasive process is used to finish these ceramic blocks (Denry and kelly, 2008). The stress generated by the surface treatments like grinding or sandblasting tends to trigger the phase transformation from tetragonal to monoclinic due to the metastability of tetragonal zirconia (Devill et al., 2006). When pure zirconia is mixed with stabilizing oxides like yttrium oxide, calcium oxide, magnesium oxide or cerium oxide, it retains the tetragonal phase at room temperature and controls the phase transformation (Garvie and Nicholson, 1972; Heuer et al., 1986). The phase transformation from tetragonal to monoclinic is similar to martensitic transformation in steel, where a large shear strain and a volumetric strain occurs during this transformation. The tetragonal phase is usually found at high temperature, but in some cases, it can be retained at low temperature. If the grain size of zirconia is less than 0.5 μm, then the retention of the tetragonal phase occurs in the material. This controlled phase transformation arrests the crack propagation and enhances the toughness of the material (David Green, 1998; Tanaka et al., 1995).

Many researchers have discussed the importance and effects of grinding on sintered zirconia (Pereira et al., 2015, 2016a, 2016b). After grinding, the microstructure and flexural strength of sintered zirconia are studied, where edge chipping damage, amount of monoclinic phase and transformed zone depth are calculated. The quantity of monoclinic phase obtained from the X-ray diffraction analysis was found to be lower in the dry condition (Kosmac et al., 1999; Pereira et al., 2015, 2016a, 2016b). The grinding of sintered zirconia under the wet cooling condition increases the percentage of monoclinic phase. The phase

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* Corresponding author.
E-mail addresses: suyaprime@yahoo.co.in (P. Suya Prem Anand), chalam@iitm.ac.in (N. Arunachalam), lvijay@iitm.ac.in (L. Vijayaraghavan).

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transformation is activated in the presence of hydrothermal loading, which deteriorates the material strength of zirconia. This causes microcracks in the transformation zone and weakens the interatomic bonding strength, which leads to subsurface damages and affects the strength of the material (Schröde et al., 2014; Pereira et al., 2016a, 2016b). When the depth of cut increases, there is a slight reduction in the strength of the material. The observed subsurface damages are viewed through the cross-section, which shows the presence of cracks in zirconia toughened alumina but no cracks are formed in the yttria-stabilized tetragonal zirconia (Xu Hockin et al., 1997). The surface and subsurface cracks are examined, where the surface cracks are obvious and can be checked effectively, but the subsurface cracks are hard to distinguish. This also relates the cracks to various parameters like depth of cut, grit size, wheel speed and feed rate (Maksoud et al., 1997). After the grinding process, the changes in the mechanical properties and the microstructure of the ground ceramic components are studied, where the materials are separated as a result of the microcracks developed below the pileup formation (Munoz-Tabares et al., 2011).

The microstructural changes which happen to the structural material are estimated based on the density, porosity, hardness and grain size. While the alumina abrasives with different grit size are used to abrade the pre-sintered and sintered zirconia samples, the abraded surface with the large grain size exhibit more microcracks and enhances the quantity of monoclinic phase (Monaco et al., 2013). When the sintered and pre-sintered blocks of zirconia are exposed to humid environment or addition of chemical compounds like Fe_2O_3 to the base material will affect the material properties. The investigation is performed based on the microhardness, phase transformation, microstructural and color changes of different zirconia. The addition of chemical compounds tends to increase the microhardness and the density of pre-sintered blocks, but these changes are not feasible in sintered block (Kaya, 2013).

The indentation test is performed on the pre-sintered Zirconia by varying the loading rate to understand the material behavior. The images obtained from the scanning probe exhibit a plastic deformation at different loading rate. The formation of compaction occurs in the lower loading rate and fills the pores in the higher loading rate. This study provides a deep understanding of material behavior in pre-sintered zirconia when grinding with diamond abrasives (Alao and Yin, 2014). The Sakai-Nowak model is used to estimate the resistance to plastic deformation and the hardness of pre-sintered zirconia. The resistance to plasticity and the absorbed energy are used to determine the quantity of elastic and plastic energy. The results show a higher absorbed energy and plastic deformation at different loading rate. This confirms the quasi-plastic behavior of the material (Alao and Yin, 2016). To study the subsurface damage of different ceramic materials like sintered 3Y-TZP (Yttria-stabilized tetragonal zirconia), glass infiltrated zirconia toughened alumina and alumina after the grinding process. The analysis is performed by the diamond disk of varying grain size to evaluate the strength degradation using edge chipping damage size. The damage depth is estimated based on the bonded interface technique. The yttria stabilized tetragonal zirconia is not affected by the edge chipping damage compared to the other ceramic materials (Canneto et al., 2016).

The recent papers have focused only on the impacts of grinding with sintered zirconia and not on the pre-sintered zirconia, where few discussions were about the material behavior. However, the pre-sintered zirconia is easier to grind compared to the sintered blocks due to the elimination of edge chipping. In the present work, the experiments were conducted on pre-sintered zirconia with different environments including dry, wet and MQL conditions. The effects of grinding on pre-sintered zirconia were evaluated based on the analysis of temperature distribution, phase transformation, flexural strength, microhardness and edge chipping damage.

Fig. 1. Microstructure of pre-sintered zirconia.

2. Experimental procedure

The material used was a 3 mol% yttria added pre-sintered zirconia and it was cylindrical in shape. This material was fabricated by the cold isostatic process at 1100 °C having a dimension of 98 mm diameter and 26 mm thickness. To analyze the microstructure, the sample was sliced into small pieces by using a diamond disk and polished with 1 µm diamond paste. Then the sample was thermally etched by placing it in a muffle furnace at 110 °C for about 5 min. The grain size of pre-sintered zirconia was found between 0.15 and 0.35 µm as shown in Fig. 1. The mechanical properties of the material are given in Table 1. The samples were ground with a surface grinding machine (Chevalier H-B81-8II) by using a resin bonded diamond wheel of 200 mm diameter with fine grits. The experiments were performed under three different cooling conditions such as dry, wet and MQL. The grinding condition used remains constant with the wheel speed of 32 m/s, depth of cut of 20 µm and feed rate of 6 m/min.

The maximum temperature rise of the workpiece surface was measured by using an infrared pyrometer (IFbN-1200, Texense). The response time of the sensor was less than 5 µs and could measure a temperature range of 0–1250 °C. The workpiece was drilled at two locations to position the infrared sensor vertically at a distance of 6 mm from the ground surface. The samples are grouped into different categories as shown in Table 2. The water-soluble oil was used as the cutting fluid with 5% concentration for the wet cooling condition and a pliable nozzle was used to supply the fluid at a pressure of 1 bar and a flow rate of 80 l/h in the wet cooling condition. The synthetic oil with compressed air was used as the minimum quantity lubrication fluid. The flow rate and air pressure were constant as 250 ml/h and 6 bar pressure for the MQL condition. The morphology and the microstructure of the ground surfaces were viewed with the help of SEM (Hitachi S4800) images. The surface roughness of the ground surfaces was measured using the confocal microscope (Olympus: Japan, Tokyo).

2.1. Phase transformation

2.1.1. X-ray diffraction

The phase transformation process takes place in pre-sintered zirconia, due to the impact of grinding, where the quantity of monoclinic phase was estimated by using the X-ray diffraction analysis. The investigation was conducted using an X-ray diffractometer on the different ground surfaces. In this experiment, the radiation CuKα was used and scanning was done in a range of 20–40° at a step interval of 1 s with a step size of 0.05°. The peak intensity ratio of monoclinic phase was calculated using the equation (Garvie and Nicholson, 1972).

$$X_m = I_{m(-111)} + I_{m(111)}/I_{m(-111)} + I_{m(111)} + I_{t(111)} \quad (1)$$