

Surface and subsurface characterisation of bioceramic workpieces machined by micro-grinding

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Abstract

In the past few decades, ceramics have been used in biomedical engineering applications due to their biocompatibility, low plaque affinity and particular mechanical properties like high flexural strength. Micro-grinding of bioceramics can cause a poor surface integrity including residual stresses, microcracks and insufficient surface topography due to the inherent hardness and brittleness of such materials. This work aims to determine the influence of various process parameters on material properties, viz. roughness, bending strength and residual stresses. Zirconia (ZrO₂) and zirconia toughened alumina (ZTA) materials are selected as typical bioceramics under test. In this regard, ceramic specimens are ground with ultrasonic vibration assistance concerning different diamond grain sizes (d_g) and a variation of the machining parameters feed speed (v_f) and depth of cut (a_e). Roughness is measured by white light interferometry, flexural strength is evaluated by 4-point bending tests and residual stresses are characterised through X-Ray Diffraction (XRD). Based on these results, it is possible to establish a relationship between the micro-grinding process and material behaviour allowing to understand the mechanisms influencing ceramic materials due to the machining process. Moreover, the improvement of the workpiece integrity through the optimisation of the process parameters is enabled.

Micro-grinding, bioceramic, material integrity.

1. Introduction

In recent years, the scientific research contributed to the improvement of bioceramics mechanical strength. Additionally, despite the brittle nature of ceramic materials, developments in ductile-mode grinding technologies and machine development have catalysed investigations in all-ceramic biomaterials and their applications [1-3]. Ultrasonic vibration assisted grinding (UVAG) is an efficient machining process which improves the machinability of brittle materials by changing the kinematics of the process [1]. In this research, the UVAG response in two types of bioceramic materials was characterised by measuring the surface roughness and subsurface damage (SSD).

2. Experimental procedure

2.1. Experimental setup

As illustrated in Fig. 1, the process kinematics of UVAG applied consists of spindle ultrasonic vibration, spindle rotation, and horizontal feed motion of the grinding tool, in this case diamond hollow drills of 1 mm diameter. Consequently, the micro-grinding process comprises a combination of the cutting actions of the entire diamond grains along the ceramic workpiece leading to a resultant force highlighted in Fig. 1.

According to the kinematics, five workpieces of each material were ground in both x-direction using two grinding steps which are summarised in Table 1. Essentially, a grinding tool with an average diamond grain size of 107 μm was used in the first grinding step. Subsequently, the second tool with an average diamond grain size of 54 μm was applied for finishing the same surface side. After both procedures along the length direction, the workpiece was turned 180° to perform the same grinding

conditions due to the requirements for the bending tests where both faces should be ground. The process was performed under cooling with a water-based lubricant and the ultrasonic generator was excited at a frequency of 30 kHz in all conditions. The strategy applied aimed to achieve high removal rates as well as to reduce the possible effects of subsurface damage (SSD).

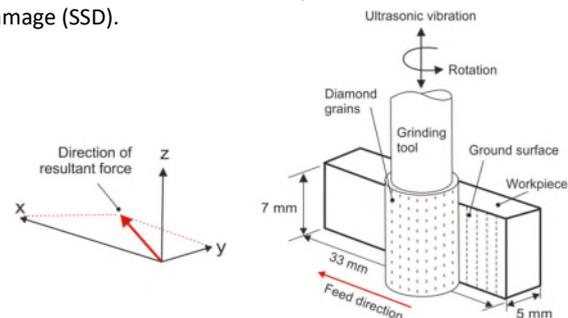


Figure 1. Illustration of the ultrasonic vibration assisted grinding (UVAG) performed in this work.

Table 1 Grinding conditions applied

Grinding step	Grinding tool	Feed speed V_f (mm/min)	Rotation (RPM)	Depth of cut (a_e)
1 st	D107	60	4500	45
2 nd	D54	33	6000	15

2.2. Material properties

The ceramic workpieces (dims. 5.0 x 7.0 x 33.0 mm³) under investigation are fully sintered, partially machined, and commercially available tetragonal polycrystalline zirconia (TPZ), also known as zirconium dioxide (ZrO₂) or zirconia, and zirconia toughened alumina (ZTA). These materials are zirconium-based ceramics and have intrinsic toughening mechanisms but differ in their mechanical properties [3], as shown in Table 2.

Table 2 Description of the material properties [4]

Material	Density (g/cm ³)	Fracture toughness K _{IC} (MPa m ^{1/2})	Young's modulus (GPa)	Hardness (GPa)
ZrO ₂	6.03	4.8	200	11
ZTA	4.10	8	380	16

2.3. Workpiece characterisation

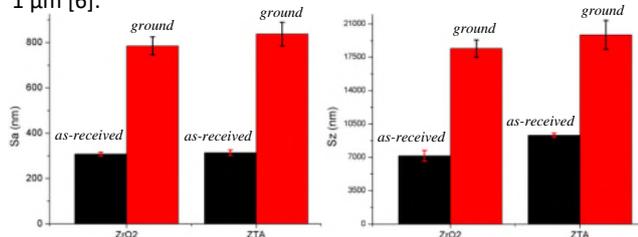
The four bioceramic batches (as-received and ground) of ZrO₂ and ZTA workpieces were analysed by 3D white light interferometry (Gaussian filter with a specified cut off λ_c of 0.08 mm and a magnification of 20X).

In order to evaluate the flexural strength of the workpieces, a 4-point bending test under a load of 0.1 mm/min was applied on 5 workpieces of each batch following the ASTM C1161 - 02c (Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature). Herein, Weibull distribution was used to describe the strength of the tested samples. In this matter, the flexural strength and the Weibull modulus were determined with the maximum likelihood estimation [5].

Finally, residual stresses were measured through X-Ray Diffraction (XRD) along the longitudinal stress axis (Cu K α source, 30 kV and 40 mA radiation).

3. Results and Discussion

Fig. 2 shows the surface roughness values Sa (arithmetical mean height) and Sz (maximum height) of the as-received and ground ZrO₂ and ZTA specimens. For both materials, the surface roughness increased considerably due to the machining conditions. However, such roughness results are optimum values for specific biomedical uses, like dental implants suggested to exhibit a surface roughness Sa between 0.5 to 1 μ m [6].

**Figure 2.** Surface roughness of the workpieces.

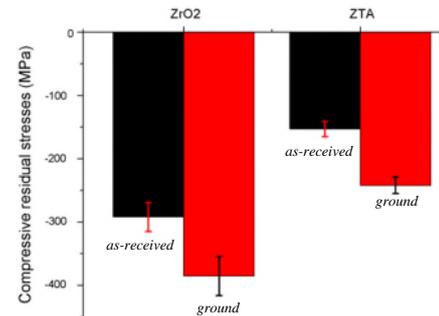
The bending tests performed indicated an improvement of the flexural strength of the ground specimens as well as a slight increase of the Weibull modulus – see Table 3. Consequently, the increase of the Weibull modulus shows a better reliability of the flexural tests and a narrow probability curve of the strength distributions [3, 5]. The increased strength of the workpieces due to grinding is associated to the toughening mechanisms in the ground surface of zirconium-based ceramics due to the martensitic (tetragonal-monoclinic) phase transformation, accompanied by 4.5% volume change [3].

Table 3 Results of the 4-point bending test

Material	Condition	Flexural strength (MPa)	Weibull modulus
ZrO ₂	As-received	846.45	11.18
ZrO ₂	Ground	930.12	12.40
ZTA	As-received	304.08	8.47
ZTA	Ground	382.70	10.64

According to XRD analysis, an increase in the compressive residual stresses of the ground samples was observed (Fig. 3). This phenomenon is due to the toughening mechanism which also involves a phase transformation. In general, compressive

values are likewise wished in the specimen surface for further biomedical application. E.g. the compressive residual stress tends to increase fatigue strength and fatigue life of bioceramic materials [6].

**Figure 3.** Residual stresses of the as-received and ground ZrO₂ and ZTA workpieces measured through XRD technique.

4. Conclusion and Outlook

Through the experiments, an increase of the flexural strength and compressive residual stresses of ground ZrO₂ and ZTA workpieces was observed in comparison to the as-received specimens. Although the surface roughness of the ground bioceramics got worse, the finished parts are suitable for biomedical uses yet. These results are helpful to understand the bioceramic materials response under micro-grinding conditions as well as to set further machining investigations.

In other to investigate the microstructure of the four bioceramic batches presented in this work, it is still necessary to quantify the amounts of tetragonal and monoclinic zirconia phases through XRD analysis and correlate them with the compressive stresses values to investigate the phase transformation phenomenon. The next steps of the research include the test of different grinding tools ranging the diamond grain sizes as well as apply diverse grinding conditions with and without ultrasonic assistance. Lastly, in regard the 4-point bending experiments, it is desired to increase the number of the tested workpieces for statistical purposes.

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