Micromilling of sintered ZrO$_2$ ceramic via cBN and diamond coated tools

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Abstract

This paper presents an experimental investigation on micromilling of fully sintered Zirconia ceramics by means of advanced cutting tools, including cBN and diamond coated mills. Zirconium oxide is selected because of its high hardness, resistance to wear, heat and chemicals. Two-flute end mills of nominal diameter 1 mm and negative rake angle were used. The effect of the coating and tool material, along with the cutting parameters, were investigated by means of a one-factor variation experimental design; and the responses of interest, including tool wear and sample surface quality, were monitored over the machining time. The worn tools and the machined surfaces were also analyzed on a SEM to investigate the type of material removal mechanisms (MRM). The diamond coated tools provide the best performance in terms of life time, however delamination of the diamond coating followed by wear of the underlying hardmetal is identified as the prevailing tool wear mechanism. A ductile type of material removal mechanism could be observed, inducing an achievable surface roughness below 60 nm. On the contrary, cBN tools exhibited fast breakage. A combined ductile-brittle material removal mechanism occurs, resulting in a surface roughness around 300 nm.

1. Introduction

New technical challenges often demand the use of ultralight components of long durability, wear endurance, chemical inertness and highly thermal resistance. On the top of it, high quality in low quantity and at competitive cost is a must. Technical ceramics, such as nitrides, carbides and oxides can be an answer to those needs. Among them, zirconium oxide (ZrO$_2$) is an excellent thermal insulator and ionic conductor; it is also bio-compatible and it provides an high strength-to-weight ratio. Main applications include thermal insulating coatings, oxygen sensors, dental and orthopaedical implants, and precision components, such as pump impellers, micro-fluidic devices and micro-molds.

However, the machining of ZrO$_2$ (and of ceramics in general) is still a difficult task. Traditional machining of ceramics is usually hindered because of their extreme hardness and wear resistance. Instead, ceramic components are today produced by near-net-shaping techniques, followed by post processing techniques, such as grinding and polishing. Nevertheless, this production chain is time consuming, and expensive. Besides, it finds technical limitations, especially as far it concerns the manufacturing of complex shaped components, and microfeatures. Mechanical cutting can also induce defects and cracks on the machined surfaces, inducing the risk of premature failure due to catastrophic propagation of the defect during service.

Efficient machining of ceramics is receiving increasing attention and new methods have been investigated in the recent years. As an example, electrical discharge machining (EDM) of ceramics has been widely studied by Ferraris et al. to supply the demand of complex shaped and highly performing ceramic components [1, 2, 3]. The approach is applied to fully sintered ceramic blanks of simple form, it is limited to electrically conductive composites. Another technique, studied by Bowman et al., is laser-assisted machining of zirconia ceramics, where by locally heating ceramic materials a higher removal rate and excellent surface quality is achieved [4].
The recent appearance onto the market of advanced cutting tools, such as cBN, PCD, and diamond (coated) cutters have also opened new possibilities. In [5], Ferraris et al. presents a first study on high speed turning of sintered ZrO$_2$ bars via PCD tools. The effect of the cutting parameters on the tool wear and achievable surface quality is studied; $R_a$ below 0.6 μm could be easily obtained. Moreover, Bian et al. [6, 7] used ultra-miniature diamond coated tools to mill fully sintered ZrO$_2$. Wear of the tool was described by three stages: 1) early coating delamination; 2) extended coating delamination with slight wear of the exposed cutting edge; and 3) severe wear of the tool blank. The entire process evolves in the order of minutes; a surface roughness of 20 nm could be achieved. Furthermore, Fledrich et al. [8] did research on milling of ZrO$_2$ with cBN tools. In their results, a higher cutting speed results in higher surface roughness. In all these tests macro tools were used. Recent works on the use of cBN tools in difficult cutting also include high speed milling of tungsten carbide and titanium alloy (TA15) [9, 10, 11].

However, the research status on hard cutting of hard and brittle materials by advanced tools is still on an early stage, especially as far it concerns micro scale applications. Based on the analysis conducted, the main challenges can be summarized as follows: a) availability of ultra-miniature tools with appropriate cutting edge geometry to ensure proper compression/tensile stress distribution in the zone of chip formation; b) tool stiffness and sufficient wear resistance; c) identification of suitable cutting parameters to ensure ductile material removal and damage-free machined surfaces [12].

In this work, micromilling experiments on fully sintered ZrO$_2$ ceramic blanks by means of cBN and hard coated tools are performed. By cBN tools, the main focus is on the influence of cutting parameters on tool wear and achievable surface quality, while the performance of 3 different coatings in terms of tool wear and achievable surface quality for a given combination of cutting conditions is investigated by hard coated tools.

2. Experimental set-up

2.1. Workpiece material and equipment

The workpiece material used in this work is a tetragonal polycrystalline zirconium oxide (ZrO$_2$) partially stabilized with yttria after final sintering from Hi-Tech Ceramics. Table 1 lists the chemical composition and mechanical properties of the material. According to ISO 13356:1997 the workpiece has an open porosity of 0% and a grain size measured by microstructure analysis as approximately 1 μm. The original surface roughness was 9 nm $R_a$.

The experiments were conducted on a Kern MMP 2522 micromilling centre at KU Leuven. The machine has a positional and repetition accuracy within 1 μm. The maximum spindle rotational speed is 40000 rpm.

2.2. Cutting tools

The cutting tools used were 2-flute end mills with nominal diameter of 1 mm and a small negative rake angle. In particular, two tool types were used: cBN tools and WC tools with an hard coating.

The cBN tools, commercially available, consist of >97% cBN grain with a grain size of 0.5 μm and a titanium carbide binder. The material hardness of the tool is 1630 HV. The coated tools are customized tungsten carbide micromills with a hard coating on demand. Specifically, three types of coatings are investigated: micrograin diamond coating (DM), nanograin diamond coating (DN) and a thin hard carbon layer (HC).

Table 2 lists the properties of these coatings. The coating technology of HC allows the application of very thin layers. This aspect is very much relevant with respect to the preservation of the cutting edge geometry. Two types of diamond coatings with a different crystal size are analysed. Coating thicknesses are chosen according to the usual standards of the supplier, implying a difference in resulting tool geometry.

The area roughness $S_a$ of the rake face of the tools is measured in house using a Veeco Wyko NT1100. For the cBN tools, a $S_a$ of 125 nm is measured, the DM, DN and HC coatings have respectively a roughness of 279 nm, 212 nm and 148 nm.

<table>
<thead>
<tr>
<th>Composition % (According ISO 13356:1997)</th>
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<tbody>
<tr>
<td>ZrO$_2$</td>
</tr>
<tr>
<td>Y$_2$O$_3$</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
</tr>
<tr>
<td>SiO$_2$</td>
</tr>
<tr>
<td>&lt; 96</td>
</tr>
<tr>
<td>&gt; 4</td>
</tr>
<tr>
<td>&lt; 1</td>
</tr>
<tr>
<td>&lt; 0.02</td>
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<table>
<thead>
<tr>
<th>Mechanical properties after sintering (*estimated value)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density $\rho$ [g/cm$^3$]</td>
</tr>
<tr>
<td>E-modulus $E$ [GPa]</td>
</tr>
<tr>
<td>Fracture toughness $K_I$ [MPa.m$^{0.5}$]</td>
</tr>
<tr>
<td>Vickers hardness $HV10$ [kg/mm$^2$]</td>
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<tr>
<td>~ 6.05</td>
</tr>
<tr>
<td>~ 210</td>
</tr>
<tr>
<td>~ 10*</td>
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<tr>
<td>~ 1200</td>
</tr>
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</table>

Fig. 1. SEM images of a new cBN tool.

Fig. 2. SEM images of a new coated tool (a) Coated end mill; (b) DM; (c) DN; (d) HC.
significant delamination of the coating was observed.

Table 4 lists the geometry parameters of the cutting tools measured in a plane orthogonal to the axis of the tool. The geometry of the tools must be suitable to machine hard materials and withstand high forces. The cBN tools have a cutting edge radius \( r_n \) in order of 3 \( \mu m \), which is sharper with respect to the coated tools. But the uncut chip thickness is expected to be lower for all tool types. Thus, it is highly likely the tool will operate in condition of a large negative rake angle. In figure 1 and 2 SEM pictures of the new tools are shown.

2.3. Investigation methodology

Sets of grooves 11 mm long were milled in order to investigate the achievable surface quality and tool wear as a function of the cutting length \( l_c \), as shown in figure 3. The volume of removed material \( V \) is proportional to \( l_c \). Table 4 lists the number of layers of each groove. Tool wear and surface quality were inspected after machining a groove. cBN tests were stopped as soon as tool breakage occurred. Experiments with coated tools were performed until significant delamination of the coating was observed.

Two series of experiments were carried out. With the cBN tools, the influence of the feed per tooth \( f_z \) over a range of 3 levels is investigated. With the coated tools, the performance of the different coatings is studied.

According to the hypothesis of ductile machining, the cutting parameters (depth of cut \( a_p \) and feed per tooth \( f_z \) were selected to be small, so that the maximum un-cut chip thickness \( (h_{max}) \) could be smaller than the critical depth of cut \( (d_{crit}) \). \( d_{crit} \), indicating brittle or ductile material removal, is a material dependent value.

For ceramics, \( d_{crit} \) was empirically defined by Bifano et al. [12] in plunging grinding as in equation 1:

\[
d_{crit} = 0.15 \left( \frac{E}{H} \right) \left( \frac{K_{ic}}{H} \right)^2
\]

Where \( E \) is the Young’s modulus, \( H \) the hardness and \( K_{ic} \) the fracture toughness of the workpiece. For the material investigated in this work, the critical depth of cut has been calculated as 1.82 \( \mu m \).

Under the condition of \( \sqrt{2r_n a_p - a_p^2} \leq f_z \), the maximum uncut chip thickness, \( h_{max} \) can be calculated as a function of the cutting parameters and the tool corner radius, \( r_n \), as follows:

\[
h_{max} = r_n - \sqrt{r_n^2 + f_z^2 - 2f_z \sqrt{2r_n a_p - a_p^2}}
\]

When \( h_{max} \) is smaller than the cutting edge radius \( r_n \), the effective rake angle is likely to be negative during machining, which promotes the condition of large compressive stresses into the region of chip formation, and thus induces plastic deformation.

Experimental conditions were determined, based on one-factor variation methodology. Table 5 and 6 lists the cutting
parameters employed for the cBN and coated tools respectively. Each experiment was repeated three times. For each combination of parameters, a new tool was used. Emulsion was selected as cutting liquid for coated tools, while pure oil was used for cBN to avoid a chemical reaction in the presence of water. Looking at the $h_{\text{max}}$ and $d_{\text{crit}}$-value, brittle machining is expected, except for the experimental condition CBN-a [cBN, $f_z = 2 \mu m$]. One cBN tool experiment was conducted at dry conditions to allow collecting chips.

The surface roughness ($R_a$) of the machined samples was measured along the feed direction at 5 places by means of a contact type Taly surf 120L surface profiler. A ZEISS SteREO Discovery.V20 microscope and a scanning electron microscope FEI XL40 SEM FEG were used to observe the tool wear and surface topography. All samples were ultrasonically cleaned before performing analysis.

3. Experimental results

3.1. CBN tools

In the next paragraph, the experimental results for cBN tools are discussed.

3.1.1. Tool wear

Figure 4 shows the maximal cumulative cutting length of the cBN tools as a function of the feed per tooth $f_z$. A maximal cumulative cutting length of 341 mm was reached. No clear relationship between $f_z$ and $l_c$ can be recognized. Figure 5 shows a SEM picture of the worn tool tips along with EDAX measurements which indicate the presence of ZrO$_2$ on the tool surface. Friction causes rounding of the cutting edge and tool tip. Traces of ZrO$_2$ on the cutting faces indicate the occurrence of an adhesive tool wear mechanism.

3.1.2. Surface quality

Figure 6 shows the surface roughness of the machined surface as a function of the cumulative cutting length $l_c$ and the feed per tooth $f_z$. The roughness rises with $l_c$. $R_a$ below 150 nm is possible during the first 250 mm. In addition, after the first groove, no significant difference in roughnesses is measured for different $f_z$-values. With increasing $R_a$, the strongest growth in $R_a$ was observed. Thus, when tool wear rises, the biggest increase of $R_a$ is observed with the lowest $f_z$, this is in contradiction to the theory which predicts a decrease of $R_a$. Looking at the ratio cutting edge radius $r_e$ to uncut chip thickness $h_{\text{max}}$, this can be explained. For lower $f_z$-values, the ratio rises, which makes engagement of the tool harder, a combination of elastic and plastic effects will occur.
The surface topography for all experiments is observed by means of a SEM microscope, in order to obtain a deeper knowledge of the surface quality and material removal mechanism. Figure 7a shows a typical ZrO2-surface after milling 33 mm. A combination of feedmarks and brittle damage is seen on the surface. Combined brittle-ductile material removal occurred.

The same conclusions could be made by the chips collected during dry machining. As shown in figure 7b, curled chips and fractured flakes are mixed together.

3.2. Tools with hard coating

The results for coated tools are presented in the paragraph below.

3.2.1. Tool wear

Figure 8 shows the maximal cumulative cutting length as a function of the different coatings. Hard Carbon tools immediately broke after 66 mm, while nanograin diamond coated tools all survive till 1980 mm; micrograin diamond coated tools show intermediate performance. Figure 9 shows some images of the worn tools.

Figure 10 shows in particular the tool wear as a function of \( l_c \) for a nanograin diamond coated tool, the different stages described by Bian et al. [6] are recognized. Early coating delamination is already visible after the first groove (66 mm), while at 660 mm (figures 10c and 10d) extended coating delamination with slight wear of the cutting edge can be noted. Wear of the underlying tool is visible after 1980 mm. The performances with a nanograin diamond coating are the best as compared to the other coatings. With the micrograin diamond coating, the third phase is already visible after 1100 mm.

The micrograin diamond coating has a higher crystal size, furthermore, the surface roughness of these tools is higher. Little peaks in the surface capture the forces during machining and the breaking out of this crystal will follow. In addition, as we look to nanograin diamond coating, the crystals are 100 times smaller, therefore at the outbreak of a crystal, only a part of the coating will be degraded.

Delamination of the coating limits the tool performance; further research to prevent the delamination should be done.

3.2.2. Surface quality

Figure 11 shows the achieved surface quality as a function of the type of coating and cumulative cutting length \( l_c \).

The best performance was achieved by nanograin diamond coated tools, who exhibit a quite constant behavior with a remarkably roughness of around 30 nm and a small variation, which makes the performance predictable. The other coatings show less performant behavior.

Figure 12 shows SEM pictures of the grooves after 66 mm for a diamond and Hard Carbon coating. Diamond coated tools exhibit a similar and smooth surface topography. While the ZrO2-surface after machining with a Hard Carbon coated tool presents many irregularities.

After inspection of the machined surface, the material removal mechanism and surface quality can be determined.

Clear feedmarks are visible in the initial phase, indicating completely ductile cutting. While with the increasing of the cutting length, micro burrs can be detected, which indicate the occurrence of plowing. High cutting loads, caused by large tool wear, causes material to flow to the side when failing to form chips. [13] The surface topography for micrograin diamond coated tools is shown in figure 13.
following conclusions could be drawn:

Tool wear, achievable surface quality and the occurring of sintered ZrO2 ceramics by cBN and hard coated end mills.

4. Conclusion

This paper presents an experimental study on micromilling of sintered ZrO2 ceramics by cBN and hard coated end mills. Tool wear, achievable surface quality and the occurring material removal mechanisms were investigated. The following conclusions could be drawn:

A nanograin diamond coating is preferred. With cutting parameters \( a_p = 4\, \mu m, f = 3\, \mu m \) and \( v_c = 120\, m/min \) a roughness below 60 nm over a distance of 2 m is achieved. Ductile machining is the main cutting mechanism, beside, plowing is also observed. Tool wear progresses along three main stages, as observed in literature. Delamination of the coating limits the tool performance, further research to prevent the delamination should be done.

The Hard Carbon coating demonstrates to be not suitable, tool breakage occurred already after milling 66 mm. The micrograin diamond coating has an intermediate performance, which can be explained by the higher diamond crystal size compared to the nanograin diamond coating.

With eBN tools and cutting parameters \( a_p = 10\, \mu m, f = 2-10\, \mu m \) and \( v_c = 60\, m/min \), a roughness below 150 nm and an maximum cumulative milling length of 341 mm was achieved. Brittle-ductile machining is observed independently from \( h_{max} \). Adhesion of ZrO2 on the tool is the prevailing tool wear mechanism.

The above results show that the hypothesis of ductile machining is only an indicative value. Bifano et al. did not take into account the effects of geometry and phase transformations of ZrO2. The critical chip thickness can therefore only be used as an indicative value.

Application of the process is mainly restricted to finishing operations and micro applications, due to rapid tool wear and high tool costs. Future research to prevent delamination of the coating or to find alternative tools may extend the application domain. Tools with a diamond tool tip possess a high hardness and should withstand the abrasive effect of ZrO2. However, these tools are expensive and only offered by few suppliers.

References