Fracture mechanisms of polycrystalline advanced ceramics

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Polycrystalline advanced ceramics are synthetic products produced by sintering together selected ceramics grains in a metal matrix serving as a binder. In order to be able to propose their optimisation, achieving high performance cutting and leading to reduced operating costs and improved working environment, relevant fracture mechanisms involved in their failure need to be determined. In this work, experimental results of plane strain fracture toughness obtained earlier on single-edge-V-notched-beam specimens were supported with microscopy analysis. These findings establish a clear connection between the fracture toughness results and the fracture mechanisms visible on and beneath the fracture surfaces, revealing adiabatic conditions that occur at the crack tip during fracture.

Keywords: Brittle fracture; Fracture mechanics; Impact fracture; Scanning electron microscopy; Toughness testing

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1. Introduction

Polycrystalline advanced ceramics are a synthetic products produced by sintering together selected ceramic grains in a metal matrix. Random orientation of the grains enables high hardness and abrasion resistance in all directions. Cutting tools made of polycrystalline advanced ceramics achieve impressive results in machining a wide range of non-ferrous metals and non-metallic materials. The accurate and efficient determination of the fracture toughness and the associated fracture mechanisms of these materials are of fundamental importance to advanced ceramics material manufacturers and end users alike. The ability to predict the behaviour of such materials can lead to better designs, increased performance, reduced costs and enhanced safety.

Relative qualities and advantages of five different methods of fracture toughness testing were investigated by Morrell [1]: the chevron-notched beam, indentation fracture, the indentation strength in four-point bending, the single-edge precracked beam in four-point bending (SEPB-B) using popped in bridging cracks, and the single edge notch beam - saw cut (SENB-S) methods. A chevron notch is commonly used to obtain controlled crack growth where the initial cracks run into a field of decreasing stress intensity. However, as controlled crack growth is often unfeasible in hard material, this feature cannot be exploited in this case. Also, as the introduction of a chevron notch is more complicated than a simple V notch and requires more time, the method was initially outruled for this research. On the other hand, SENB method required very little specialised testing apparatus and specialist skills, which makes it relatively cheap and applicable in most standard fracture laboratories. Also according to [1], amongst all of these alternatives, the SENB method was found to provide the most reproducible results and therefore was chosen for investigation.

The findings presented in this paper lead to a greater understanding of the fracture behaviour of polycrystalline advanced ceramics, informing the development of improved future grades of material.

2. Materials and methods

Two grades of polycrystalline advanced ceramics have been used for investigation [2] containing different mean WC grain sizes and amounts of cobalt employed as a binder in the structure. These grades of material will be referred to as grade A and grade B and contain average WC grains of 6 and 30 μm, respectively. As the grade A material has more grain boundaries, a higher percentage of cobalt binder is expected. Single edge V notched beam (SEVNB) specimens were cut from bulk circular disks of 50 mm diameter and two different thicknesses using a laser. The test specimens had the following average dimensions:

- 'thick' samples: (*b*) 4.73 mm x (*h*) 6.22 mm x (*l*) 28.5 mm
- 'thin' samples: (*b*) 2.98 mm x (*h*) 6.22 mm x (*l*) 28.5 mm.

The specimens were positioned on a three-point bend (TPB) fixture with a span (*S*) of 24.8 mm and two supporting rollers with a radius of 3 mm. The load was applied by a stainless steel striker fitted with a loading roller with the same radius of 3 mm. All rollers were made from Inconel alloy 600 (Ni72/Cr16/Fe8) which retains its stiffness at elevated temperatures. A total of 228 specimens were tested in the TPB configuration. Tests were carried out at a range of loading rates: 1 mm/min and 100 mm/min on a standard screw driven testing machine [3], and 0.3 m/s, 1 m/s and 5 m/s on an instrumented drop-weight tower. These dynamic rates are the conditions occurring in real cutting processes [4, 5]. Choice of loading rates between quasistatic and the highest dynamic rate was taken as a uniform distribution across five decades of the loading rate in logarithmic scale. Three temperature levels were employed: 25, 300 and 600°C [6], with the highest temperature appearing in real operating conditions [7]. At least three and no more than 5 repeats at each combination of rate and temperature were conducted. The plane strain fracture toughness is determined by the load at initiation method according to ASTM E1820-01 fracture standard, using single-edge vee notch beam (SEVNB) method (CEN 14425-5:2004), and presented in Fig. 1 [8]. Unfortunately, it was unfeasible to obtain results across the full range of testing conditions for both types of specimens due to their limited supply. However, the number of available specimens and obtained results were very consistent and quite sufficient to quantify the effects of different parameters on the fracture toughness and draw important conclusions.

Test results in Fig. 1 revealed the apparent fracture toughness K_{Ib} of grade A material to remain generally constant at quasi-static rates, and then to decrease with a further increase in the loading rate above 100 mm/min. It can also be seen from the graph that the material was relatively independent of temperature changes up to 300°C at lower rates. At 600°C, it is evident that the grade A material exhibits improved fracture toughness at quasistatic rates where cobalt in the mixture becomes more ductile, while the opposite is true at higher rates (at least at 0.3 and 1 m/s) with a significant difference between room temperature and the elevated temperatures (300°C and 600°C). This can not be claimed with certainty for 5 m/s results due to much higher discrepancy and overlap of the standard deviations. Fig. 1 also shows that the apparent fracture toughness of the grade B material remains relatively constant at quasi-static and intermediate rates up to 0.3 m/s, and then sharply drops at higher rates. Weak dependence on temperature was observed at quasi-static rates where K_{Ib} slightly increases with an increase in temperature, while the temperature effect is considerable at higher rates and sharply decreasing K_{Ib} values.

3. Result discussions

In order to gain a better insight into the fracture process, fractured specimens were sectioned and polished. The sectioning and polishing were performed as shown in Fig. 2.

Edges on the polished surfaces of these specimens were then examined using field emission gun scanning electron microscope (FEG-SEM). The location of the examined region was up to 150 μm below the notch tip, hence the region of crack initiation. The micrographs obtained for grade A specimens with magnification of 15000 x are presented in Fig. 3, while Fig. 4 shows micrographs taken for grade B specimens with 3000 x magnification, all fractured at room temperature. The direction of crack propagation is from top to bottom of the images in each case, where the fracture surface is shown on the right of image in Fig. 3 and on the left of image in Fig. 4.

Using energy dispersive X-ray analysis (EDX), it was found that the light greyish areas represent cobalt, while dark grey corresponds to WC grains. Fig. 3(a) shows the polished surface of the grade A specimen fractured at 1 mm/min with a corresponding apparent fracture toughness of 11.1 MPa m$^{1/2}$. It can be seen that the cobalt is completely undamaged, the edge is fairly straight and the fracture toughness is relatively high. Fig. 3(b) points at the notch root with a clearly visible grain (marked in the image) and deteriorated binder around it (marked by ellipses), corresponding to the specimen fractured at 5 m/s with an apparent fracture toughness of 6.0 MPa m$^{1/2}$. A long crack is also visible at the fracture surface, sweeping all the way from the top to the bottom of the image. Comprehensive deterioration of cobalt is noticeable – this region is debonded, providing a path of low fracture resistance and resulting in intergranular fracture.

Fig. 4(a) shows the polished surface of the specimen fractured at 1 mm/min with a corresponding apparent fracture toughness of 12.4 MPa m$^{1/2}$. This time again the binder is completely undamaged. Adhesion between the

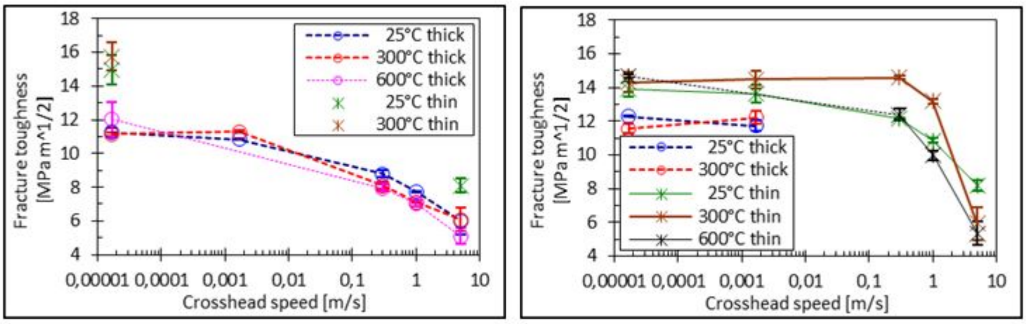

Fig. 1. Apparent fracture toughness of PCD grade A (left) and grade B (right) as function of crosshead speed and temperature.

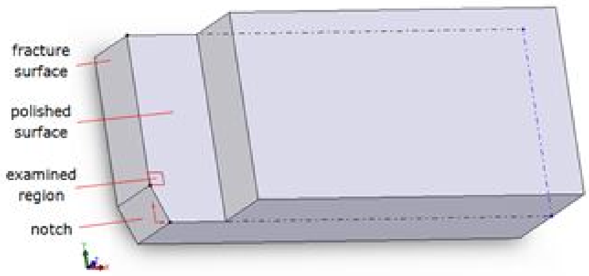

Fig. 2. The method of sectioning and polishing the specimens.

grains is quite strong, resulting in high fracture toughness and crack propagation occurs through the grains rather than around them, creating a fairly flat fracture surface. Fig. 4(b) shows characteristic views of the edges of two specimens fractured at 5 m/s with the apparent fracture toughness of 7.7 MPa $m^{1/2}$. Identically as for the grade A material, comprehensive deterioration of the cobalt is noticeable and the binder region is debonded, facilitating lower energy crack propagation. The dark lines visible in Fig. 4 are introduced by the sectioning process and are commonly called the gouging effect. A comparison between the sectioned surfaces of one grade A specimen fractured at the quasistatic rate and another fractured at the dynamic rate is shown in Fig. 5. Smaller magnification was used in order to get a better view over a greater region. It can be seen that the binder in regions close to the edge is considerably deteriorated during dynamic fracture (b) which extends up to 30 μm beneath the fracture surface, as opposed to the fracture at the quasi-static rate (a), where the binder is left completely undamaged.

4. Conclusions

Polycrystalline advanced ceramics are finding increased application in industry, where brittle fracture can often render the cutting tool unfit for purpose. In applications such as oil well drilling, the replacement of the cutter head is a time-consuming and costly exercise. The characterisation of its fracture properties is therefore of fundamental importance for the classification of existing materials and the development of improved grades in the future. The present paper has presented microscopy studies of the fracture behaviour of two grades of polycrystalline advanced ceramics specimens subjected to a range of loading rates between 1 mm/min and 5 m/s, and a range of temperatures between room temperature and 600°C. These two grades contained different mean WC grain sizes of 6 and 30 μm and, therefore, different amounts of cobalt employed as a binder in the structure. As the finer grade material has more grain boundaries, a higher percentage of cobalt binder is expected, located within smaller cobalt pools. On the other hand, coarser grade has greater cobalt pools due to larger grains. This can clearly be seen comparing micrographs for fine and coarse grade material.

The paper explains fracture mechanisms of polycrystalline advanced ceramics based on microscopy analysis observations of the fractured specimens. This also complies with outcomes of other previously published work within the same research. Experimental results of plane strain fracture toughness obtained earlier on single-edge-V-notched-beam specimens were supported with microscopy analysis. These findings establish a clear connection between the fracture toughness results and the fracture mechanisms visible on and beneath the fracture surfaces, revealing adiabatic conditions that occur at the crack tip during fracture. This behaviour has not been observed earlier in literature for the same type of material. At quasistatic rates the fracture toughness was found to be relatively constant.

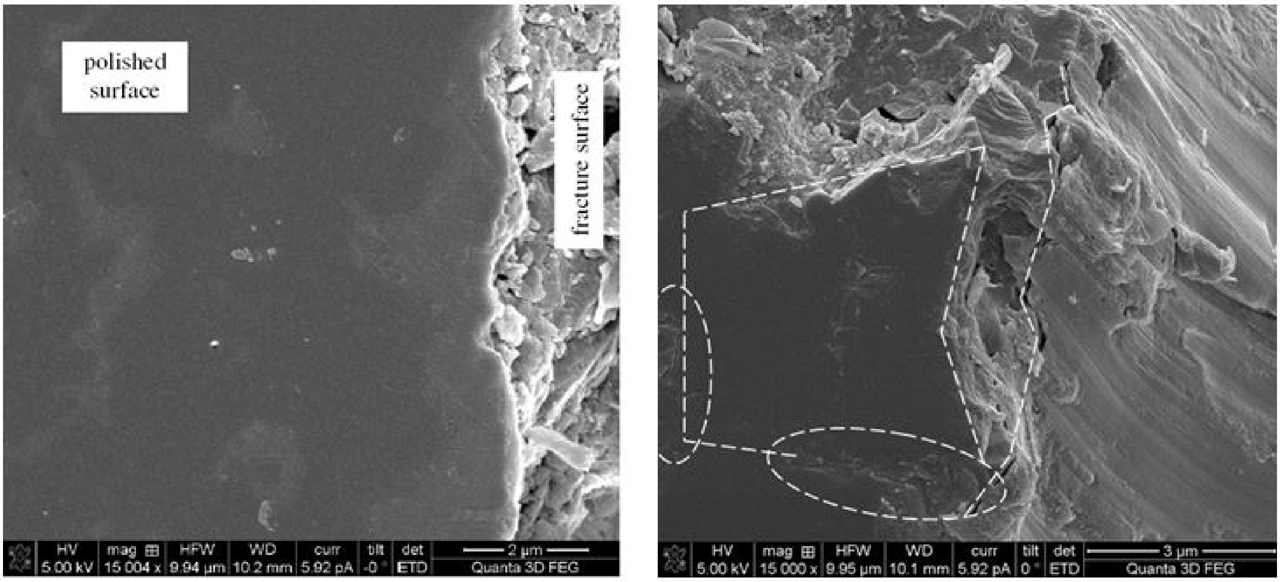

Fig. 3. SEM micrograph showing the polished surface of grade A samples fractured at 1 mm/min (a) and 5 m/s (b).

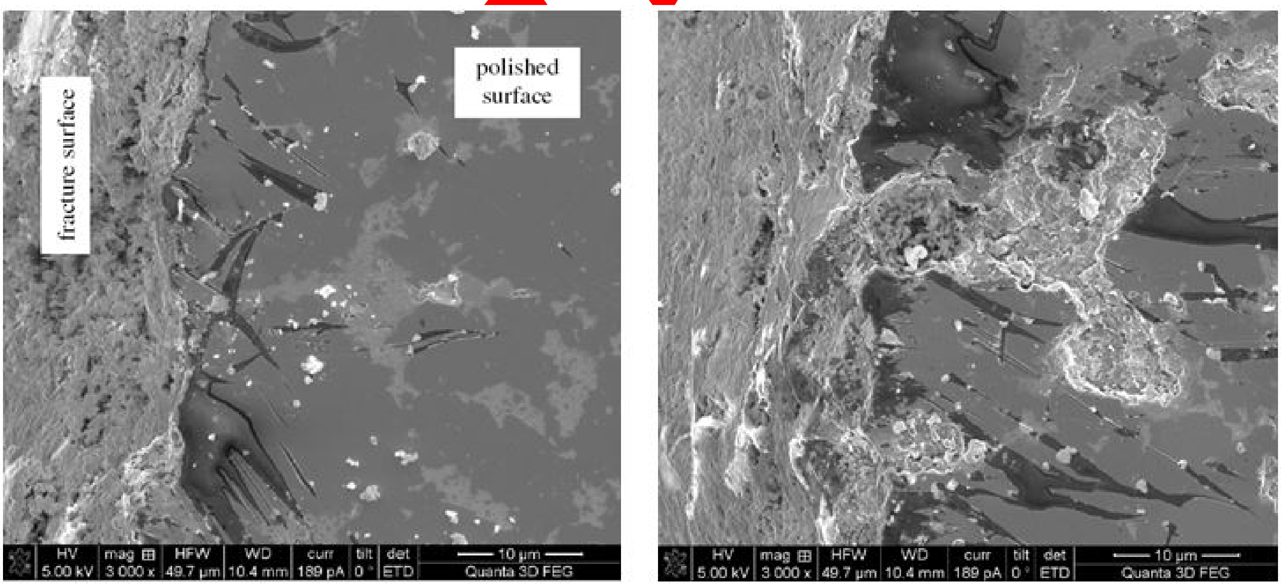

Fig. 4. SEM micrograph showing the polished surface of grade B samples fractured at 1 mm/min (a) and 5 m/s (b).

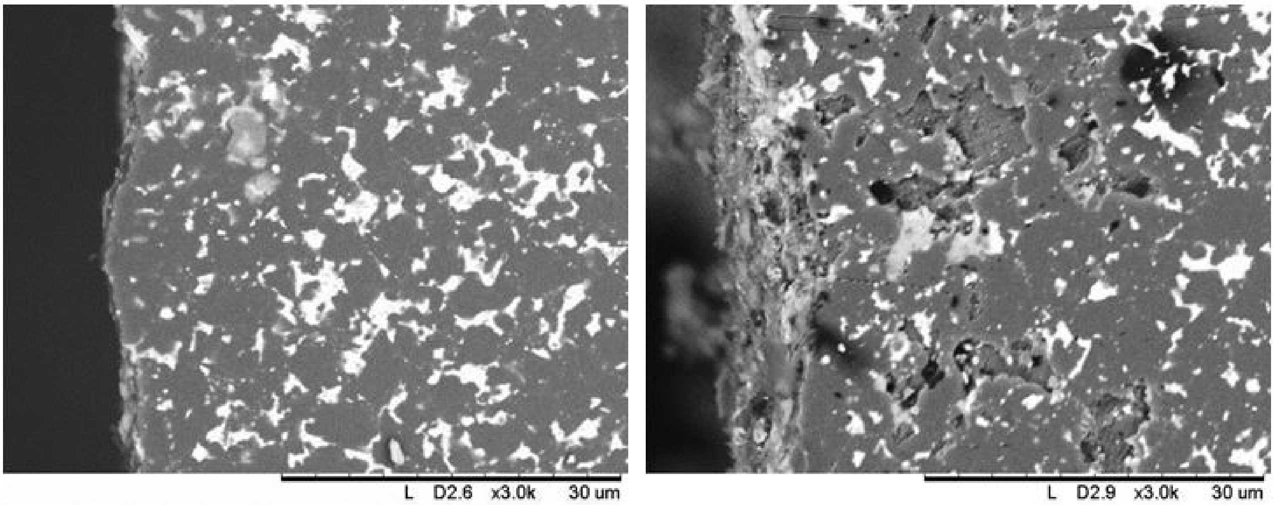

Fig. 5. Scanning electron micrographs showing the polished surface of grade A specimens, fractured at 1 mm/min (a) and 5 m/s (b), at a magnification of 3000 x.

There is enough time for the heat generated at the crack tip to be conducted into the bulk material, so the process is essentially isothermal. At dynamic rates the fracture toughness drops consistently, most likely due to a temperature increase at the crack tip. Because of the extremely small time scales available for fracture initiation and propagation, heat generated in the small region ahead of the crack tip becomes trapped and adiabatic conditions take place, which is suggested by the fact that material satisfies an adiabatic thermal decohesion model as found in [2]. This results in the temperature and temperature gradients in this region becoming high, leading to the deterioration and damage of the cobalt phase due to differences in the coefficients of thermal expansion of ceramics grains and cobalt binder, and a corresponding decrease in fracture toughness. Therefore, it may be concluded that cobalt plays a major role during the fracture of polycrystalline advanced ceramics and it is mainly responsible for the deterioration of its fracture properties under the extreme working conditions encountered during the actual cutting process.

Apart from the theory of adiabatic heating in the vicinity of the crack tip resulting in lower KIc values at high loading rates, it is also known that cobalt, like most metals with a face centred cubic microstructure, is very rate sensitive with the flow stress seen to be an increasing function of strain rate [9–11]. Therefore, the faster it is loaded, the more brittlely it behaves, and the rate dependency in the material is mainly due to the influence of the cobalt binder phase. Considering all these findings, it may be concluded that the cobalt binder phase is responsible for the deterioration in fracture toughness of polycrystalline advanced ceramics sintered compacts at high loading rates.

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