

The Mechanical Properties of Polycrystalline Diamond as a Function of Strain Rate and Temperature

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Abstract

Polycrystalline diamond (PCD) materials are used in various applications, mainly as cutting tools for machining non-ferrous metals and non-metallic materials and for rock drilling operations. A better knowledge of their mechanical properties is of fundamental importance to PCD manufacturers and end users. In order to understand and predict the behaviour and structural integrity of the tools containing PCD, it is first necessary to study the behaviour of the material as a function of loading rate and temperature.

In this paper, material behaviour is determined under testing conditions which correspond more closely to those in actual drilling, which is a significant improvement over investigations to date. Young's modulus determined by four-point bending and a split-Hopkinson pressure bar apparatus was relatively constant with the rate, while a consistent decrease was observed with increase of temperature. The

flexural strength was found to increase with the temperature, while decreasing with an increase in rate.

Keywords: PCD; Mechanical properties; Cutting tools

1. Introduction

Diamond exists in essentially 4 forms: natural diamond, synthetic diamond (grown by the High Temperature High Pressure route), chemically vapour deposited diamond (CVDD) and polycrystalline diamond (PCD). As noted in the review by Field [1] and separately by Field and Pickles [2], all the various forms of diamond have experimental challenges in measuring the mechanical properties.

It is probably true to say that PCD materials are the least well-understood. Applications of PCD include machining and drilling where the temperatures at the workface are very high (red heat). In order to understand its fracture characteristics and to be able to extend the capabilities and potential application areas of polycrystalline diamond (PCD), there is a need to determine the mechanical properties of the material under typical in-service conditions. A limited knowledge of material properties under static loading rates and room temperature conditions is of little value when analysing the transient behaviour of the cutting tool under typical operating conditions encountered during the drilling of an oil well. Some mechanical properties of chemical vapour deposition (CVD) and natural diamond, as well as polycrystalline diamond compact (PDC), have been investigated at room temperature, i.e. tensile

strength, transverse rupture strength, compressive strength, impact strength, fracture toughness and elastic constants as described below. The quasi-static Young's modulus of two grades of PCD has been determined at room temperature in [3]. Clearly, however, the availability of fracture and mechanical properties of PCD at low rates and low temperatures is not sufficient to satisfy industrial and academic needs, due to their irrelevance to in-service drilling conditions. Only a limited number of physical properties of diamond are known at high temperatures. This includes the Young's modulus which is an important parameter for mechanical applications in general. Werner et al. [4] made remarks on the high temperature Young's modulus of synthetic diamond grown by microwave assisted CVD and tested in three-point bending. Young's modulus testing of PCD/WC-Co specimens was carried out by Belnap and Griffo [5] by tapping the simply supported samples and measuring their resonant frequency, according to the relevant testing standard [6].

Tensile and fatigue strength of free-standing CVD diamond was measured in three point bending by Davies et al. [7, 8, 9]. Field et al. [1, 2] worked on determining strength, fracture and friction properties of natural and synthetic diamond. Widely scattered data and even discrepancies in the measurement of the strength of free standing diamond film were frequently reported [10, 11]. Scatter occurs due to the fact that a variety of techniques were used to characterize the mechanical properties, and results from one particular technique are not always comparable to another.

The material properties of PCD include high hardness and strength, combined with moderate toughness. Early measurements of the mechanical properties were made by Gigl [12] and Roberts [13]. A comprehensive study has been made by Lammer [14].

He has shown that PCD materials behave in a manner similar to that of most engineering ceramics, but have the distinct advantage of a higher fracture toughness. He also summarised the properties of PCD materials in terms of the type of PCD, regardless of the grain size and whether they are supported or free-standing. As with other ceramics, the transverse rupture strengths increase as the grain size decreases. When the data are plotted against $d^{(-1/2)}$, where d is the grain size, there are two distinct regimes of behaviour. Various mechanisms are suggested [14]: those based on dislocations are thought to be inappropriate for PCD and the favoured explanation is the one from Rice [15]. He argued that flaws of length c , introduced during machining and surface preparation, are generally independent of the grain size d . Thus for large grains $c/d < 1$ and the appropriate fracture toughness is that of the single crystals. On the other hand, for small grains $c/d > 1$ and the polycrystalline toughness is involved. Based on the measured fracture toughness values for the PCD materials and assuming a simple flaw geometry, the critical crack lengths are estimated [14]. The change from one regime to the other should take place when $c \approx d$ and this agreed well with the data.

This paper contains the results of a series of mechanical property tests performed on PCD specimens. Two grades of PCD have been investigated. These materials differ with respect to the mean diamond grain size and the amount of cobalt employed as a binder in the PCD structure: G6 consists of 6 micron grains with 23 wt % Co and G30 consists of 30 micron grains with 7 wt % Co. As the G6 material has more grain boundaries, a larger amount of cobalt is expected. Rectangular cross section specimens were employed for the quasi-static flexural tests and the tests were performed using a standard screw-driven Hounsfield tensile testing machine. The

second set of specimens were disc shaped and used for high rate compression tests on a split-Hopkinson pressure bar apparatus (SHPB).

Experiments were carried out in air at a range of loading rates and temperatures. The average modulus and strength of a material are often obtained from a larger number of samples, but cost effectiveness was a primary concern in this case, and therefore between 3 and 5 specimens were used at each combination of loading rate and temperature. Loading rates were varied from quasi-static values of 1 mm/min up to dynamic impacts of 38 m/s. The temperature was varied from room temperature up to 680°C in the quasi-static tests using a custom made high-temperature testing chamber [16]. In the case of dynamic tests, a small cylindrical heating unit was made of stainless steel and attached to the SHPB setup in order to heat the specimen up to 300°C.

2. Experimental procedure

2.1. Quasi-static Young's modulus tests

Young's modulus quasi-static tests were performed in four-point bending under normal atmospheric conditions at room temperature, 300°C, 600°C and 680°C, according to the standard BS EN 843-2:2006. Specimens were of the following average dimensions: (*h*) 4.76 mm x (*b*) 6.25 mm x (*l*) 28.5 mm. The width and thickness of each individual test piece was measured at several places and average values recorded. The test piece was supported on two bearing edges perpendicular to its length. The outer support bearing edges took the form of parallel rollers of 3 mm

diameter with a span of 25 mm. The inner span between the loading rollers in four-point bending was 10 mm. The loading rollers had the same diameters as the support rollers.

The set of samples was instrumented by strain gauges (Radionics type 632-146 at room temperature with gauge dimensions of 1.6 mm x 2.0 mm, Micro-Measurements type WK-06-062AP-350 at 300°C with gauge dimensions 1.57 mm x 1.57 mm and Micro-Measurements type ZC-NC-G1262-120 at 600°C and 680°C with gauge dimensions 1.57 mm x 1.93 mm) bonded in the middle of the tensile surface. The reason the strain gauges were used is that the stiffness of the test pieces was an order of magnitude higher than the stiffness of the other parts of the machine. This results in measured Young's modulus being 17 times smaller than the actual value if the displacement of the crosshead is used in the calculation. After wiring, each specimen was connected to a 2310B Signal Conditioning Amplifier, capable of achieving a maximum excitation of 15V DC and a maximum gain of 11000, and a 100MHz Handyscope HS3 for capturing the strain signal. Each test piece was inserted into the test jig and centralized. A steadily increasing force was applied to the test specimen at a constant cross-head displacement rate of 1 mm/min and 100 mm/min until the specified maximum load was obtained which avoided the occurrence of fracture. When the maximum selected force was achieved, the direction of the machine was reversed and the load was reduced to zero. This cycle was repeated a few more times to the same peak load until repeatable results were obtained. The first cycle may show a different response to subsequent cycles as the test piece beds down into the test jig and the machine movement stabilizes. The use of both loading and unloading cycles is required in order to take machine hysteresis into account and to test strain gauge

adhesion. The load and time were recorded automatically by the machine and the strain on the tensile surface was measured using the strain gauges continuously over the whole loading range.

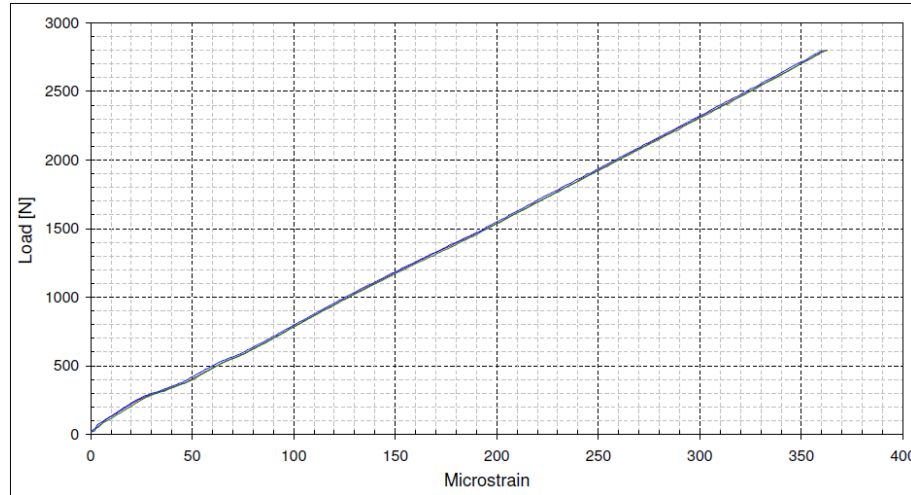


Fig. 1. Force vs. microstrain for PCD G30 sample loaded in four point bending

Based on the load-time history recorded by the machine and the corresponding strain gauge readings, the load as a function of microstrain was obtained, as shown in Fig. 1.

The load increases linearly with strain from which a reasonable calculation of Young's modulus can be made according to the following equation:

$$E = \frac{3d_1}{bh^2} \frac{\Delta F}{\Delta \epsilon}, \quad (1)$$

where d_1 is the spacing between the inner and outer roller, and $\Delta \epsilon$ is the strain change over the defined load range ΔF .

2.2. Dynamic Young's modulus tests

The SHPB apparatus used for these tests consists of two long cylindrical bars of 10 mm diameter, 1000 mm long and made of 316L stainless steel. A gas gun capable of achieving projectile velocities in excess of 100 m/s fires the stainless steel striker. The barrel length is 750 mm with a 10 mm diameter bore. PCD specimens tested were cut into disc shapes with a 9 mm diameter and 3 mm thickness.

SHPB testing of superhard PCD materials was found to be very difficult. Problems encountered during testing necessitated modifications of conventional Kolsky analysis [17]. It was necessary to instrument the specimen with a minute strain gauge and measure the strain directly rather than using the conventional method of integrating the reflected strain in the incident bar. The reason for this is the very high impedance of the PCD material. Generally, the harder the material being tested, the lower the level of the reflection is obtained. This effectively means that the reflected wave in the steel bar is very small and becomes lost in the noise, and significant errors resulting from the strain integration are unavoidable. For this reason, the specimens were instrumented by Micro-Measurements type EA-031EC-120 minute strain gauges with gauge dimensions 0.79 mm x 0.81 mm, as shown in Fig. 2.

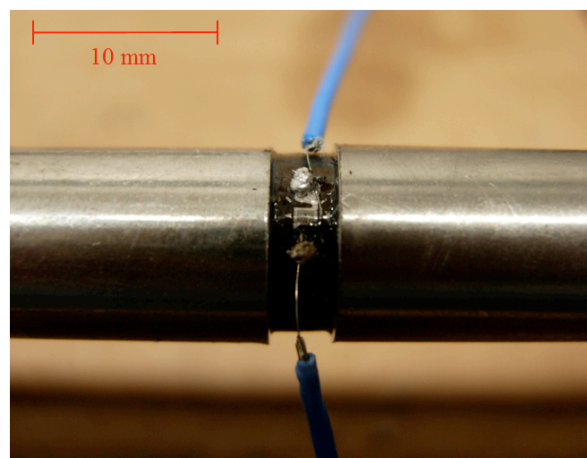


Fig. 2. Specimen instrumented with minute strain gauge

The measurement of the stress wave was performed by Radionics type 632-146 strain gauges attached to the transmitter bar and wired in a full bridge configuration, thus compensating for bending and temperature. Gauges were attached as close to the bar/specimen interface as possible, thus improving measurement sensitivity over a shorter period of time. The stress value in the test specimen $\sigma_s(t)$ can be found by a 1D wave analysis using the transmitted wave:

$$\sigma_s(t) = E \frac{A_b}{A_s} \varepsilon_t(t), \quad (2)$$

where E is the Young's modulus of the bar material, A_s is the instantaneous cross-sectional area of the specimen, A_b is the cross-sectional area of the bars and $\varepsilon_t(t)$ is the transmitted strain.

The temperature is taken as the initial equilibrium temperature of the specimen and therefore poses no experimental problems. Heat generated during the process is neglected as the PCD samples are extremely stiff and their deformation is negligible.

The high temperature tests were performed in the same way as those at room temperature, with the addition that the cylindrical heating unit was placed symmetrically around the specimen itself and a short portion of the pressure bars adjacent to the specimen, as shown in Fig. 3. The heating unit has a hole on top for easy insertion and removal of the test pieces. Two cartridge heaters with power rating of 200W each are connected in series and inserted into holes drilled and reamed on one side of the heating unit. The specimens were instrumented with Micro-Measurements WK-031EC-350 pattern minute strain gauges, intended for use up to

300°C, with gauge dimensions 0.79 mm x 0.81 mm. Another strain gauge was placed on the transmitter bar area held at room temperature.



Fig. 3. Cylindrical heating unit attached to SHPB

Elevated temperature tests with the SHPB method pose a problem if temperature gradients exist along the length of the bar, because the wave propagation speed is a function of the temperature-dependent Young's modulus of the bar. Therefore, it is necessary to correct the test data for the temperature gradients in the pressure bars, which are roughly exponentially decreasing with the distance from the test specimen. These gradients have two effects on the strain signal: a continuous change of amplitude and a continuous change of wave velocity. An amplitude correction factor is derived [18, 19] and employed in the following form:

$$\frac{\varepsilon_0}{\varepsilon_T} = (1 + C_a)^{3/4} \quad (3)$$

and

$$C_a = \frac{a_2}{a_1}(T - T_0), \quad (4)$$

where T_0 is the ambient temperature, T is the temperature of the specimen, a_1 and a_2 are constants that satisfy a relation for a linearly temperature dependent Young's modulus $E = a_1 + a_2(T - T_0)$. The ratio $\varepsilon_0/\varepsilon_T$ is the ratio of the strain recorded at the gauge station at ambient temperature T_0 to the actual strain at the specimen at temperature T . The correction factor for the pulse duration can be derived through the velocity of the pulse given by the strain amplitude multiplied by the elastic wave speed:

$$\frac{v_0 / c_0}{v_T / c_T} = (1 + C_a)^{3/4}, \quad (5)$$

from which the following relation results:

$$\frac{t_T}{t_0} = \sqrt{\frac{E_0}{E_T}} (1 + C_a)^{3/4}. \quad (6)$$

Testing at temperatures above 300°C is not feasible at present, due to the absence of the appropriate miniature strain gauge pattern on the market, and also due to the problems of sintering thicker disk shaped specimens to allow the use of larger strain gauges.

The Hopkinson bar was also used by Feng and Field [20] to obtain the strength of diamond particles at different strain rates. They found that the ends of the WC bars in the Hopkinson bar apparatus were damaged. To avoid having to polish the ends of the

bars after every shot, thin WC slices of 2 mm thickness were glued to the bars. However, this problem has not been encountered in the present work.

2.3. Flexural strength tests

Flexural strength quasi-static tests were performed in three-point bending at 1 and 100 mm/min and two temperature levels, 25°C and 300°C, according to the standard BS EN 843-1:2006. The test setup was the same as for the quasi-static *E* tests. The force was applied at a constant cross-head displacement rate until the specimen fractured. The only measured parameter required is the peak force supported by the test piece at the instant of failure, which is recorded automatically by the machine. The nominal fracture strength of each test piece can be calculated from the following bending equation:

$$\sigma_f = \frac{3Fl}{2bh^2}. \quad (7)$$

3. Results

Overall Young's modulus results, at both quasi-static and dynamic rates, are presented in Figs. 4 and 5 for the G6 and G30 material, respectively.

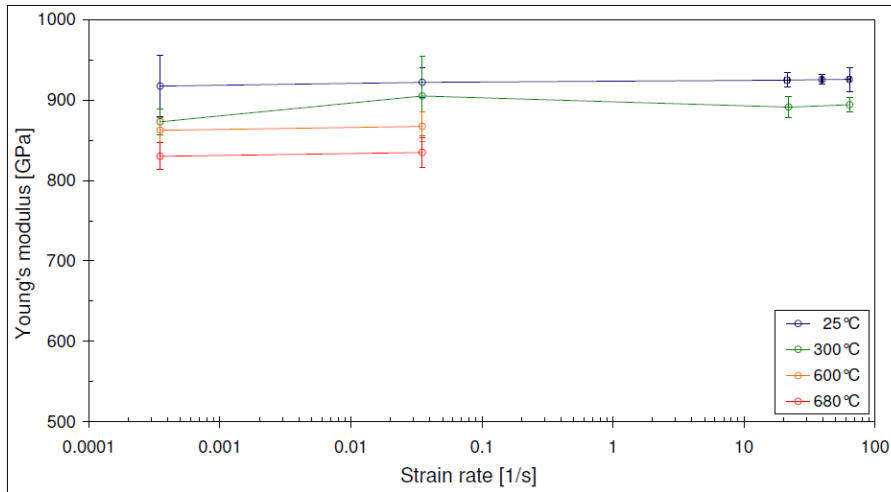


Fig. 4. Young's modulus as a function of strain rate for PCD G6 grade.

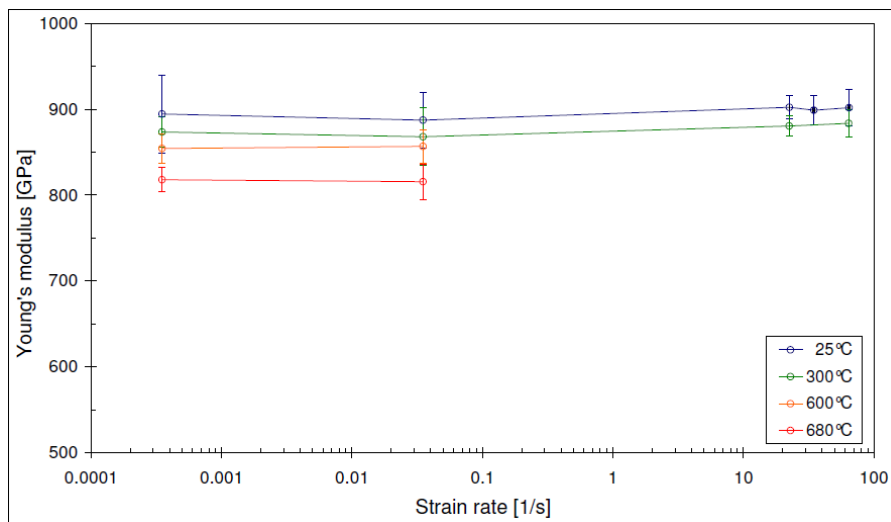


Fig. 5. Young's modulus as a function of strain rate for PCD G30 grade.

Fig. 6 shows the change in Young's modulus at 1 mm/min of both PCD grades as a function of temperature up to 680°C.

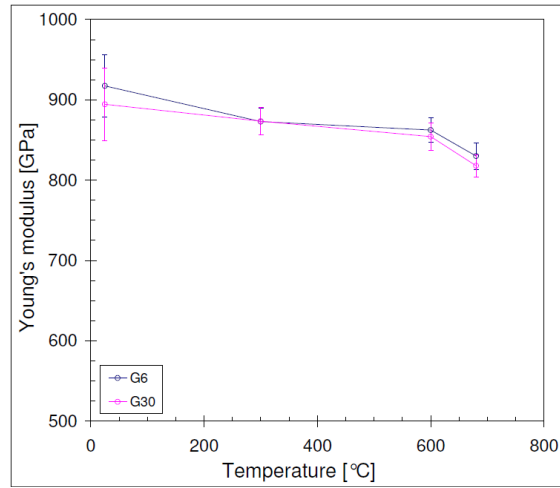


Fig. 6. Quasi-static Young's modulus of G6 and G30 as a function of temperature.

The flexural strength of each material as a function of loading rate and temperature is presented in Fig. 7.

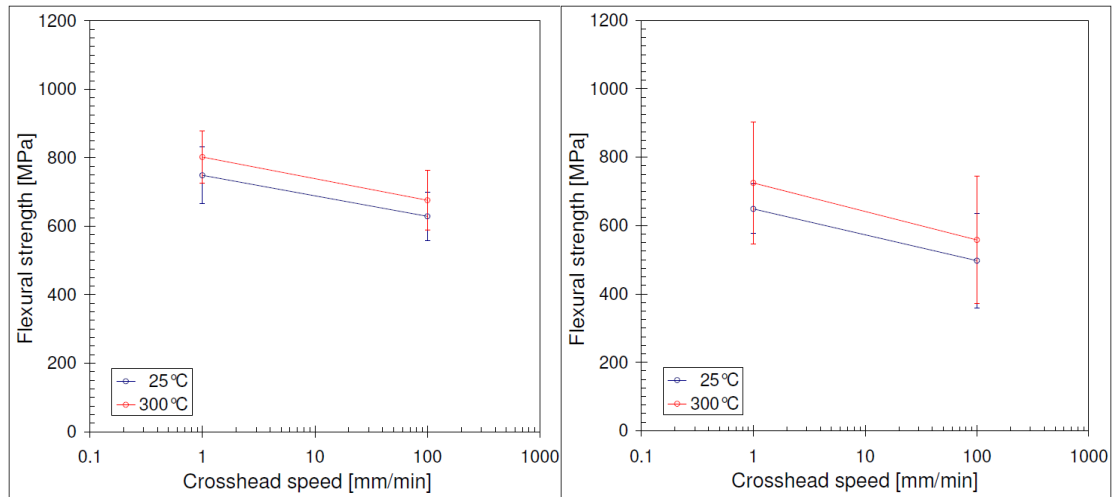


Fig. 7. Flexural strength of G6 (left) and G30 (right) as a function of crosshead speed and temperature

4. Heat treatment of fine grain PCD test specimens

In order to determine if the residual stresses contained in the PCD samples as a result of the manufacturing process have a significant influence on these test results, two forms of heat treatment were carried out on the fine grain material: annealing and quenching. Six G6 three point bend specimens were heated up to 650°C in a programmable oven for 10 minutes. Three of those were cooled down rapidly by quenching in oil, subjecting the material to thermal shock, while the other three were cooled very slowly in air, decreasing the temperature in the oven by 10°C every minute, in an attempt to relieve any residual stresses they might contain. The specimens were then tested in three point bending at 1 mm/min and their flexural strengths were compared to the as-received values, as shown in Fig. 8.

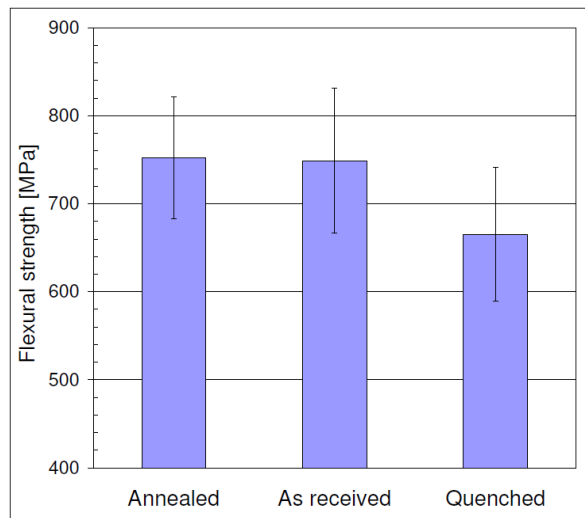


Fig. 8. Effect of heat treatment in PCD G6 specimens tested at 1 mm/min

Although the deviation is again quite high, it can be seen that there is negligible difference between the flexural strength of the annealed specimens and those previously tested, while there is a considerable drop in the flexural strength of the

quenched specimens. This implies a much greater presence of thermally-induced damage compared to the 'as received' samples. This simple experiment and the associated results suggest that the heat treatment effect in the PCD specimens is quite negligible and does not influence the mechanical and fracture properties of the materials being tested.

5. Discussion

There has been very little research conducted in the area of mechanical properties of superhard materials. The sparse data which is currently available in the literature is not sufficient to accurately predict the behaviour of a PCD cutting tool under typical operating conditions encountered during drilling operations. The boundaries have now been pushed to dynamic rates and high temperatures very close to the temperature of diamond graphitization.

As can be seen from Figs. 4 and 5, both material grades exhibit relatively constant Young's modulus behaviour across five decades of strain rate. A consistent decrease of Young's modulus is observed with an increase of temperature up to 600°C, as can be expected due to softening of the binder material, after which a rather sharp drop in Young's modulus occurs. This strong reduction in E values above 600°C is clearly visible in Fig. 6, which is in line with what has been observed before in [4].

The flexural strength experimental results show that an increase in average grain size reduces the flexural strength. This was ascribed to the flaws present on the tensile surface, whose size was found to be proportional to the grain size of the material. An

increase in temperature level results in a corresponding increase in flexural strength by about 7-12% for both PCD grades. On the other hand, the increase in loading rate also caused a decrease in flexural strength of both material grades. The mechanism responsible for this behaviour can be explained considering the structure of PCD. Essentially, PCD materials have a skeleton of diamond grains filled with cobalt, or in other words, hard crystals of diamond with a softer metal. Using SEM and XRD, Pipkin and Wilson [21] have shown that plastic deformation is induced in the particles during sintering and true diamond to diamond bonding occurs. Increasing the temperature, cobalt becomes softer resulting in an overall decrease in modulus and an increase of the flexural strength. On the other hand, an increase in the loading rate causes cobalt to become more brittle [22, 23, 24], which as a consequence has an overall reduction of flexural strength.

Quite high standard deviation was obtained. This is common however when testing the flexural strength of brittle materials as these materials have long been known to exhibit a wider degree of scatter, even under identical testing conditions, when compared to that of ductile materials. This deviation in PCD materials is associated with the effect of small defects on the tensile surface of up to 2 microns in depth, as well as the extremely brittle nature of the material. This is discussed further in the context of the fracture characterization of these materials as reported in [25]

6. Conclusions

The Young's modulus results obtained from the instrumented sample tests were very satisfactory with a noticeably small standard deviation returned of up to 2.3%

maximum. A slight and almost negligible increase in Young's modulus of only few GPa was observed at higher rates, which indicates that PCD does not manifest any considerable increase in stiffness when subjected to increased strain rates. Discrepancies within calculated Young's modulus values can be most likely ascribed to experimental error considering the complexity of the testing system.

Although a very high discrepancy was found in the flexural strength tests, it was still possible to see a clear trend of increasing flexural strength with temperature, as well as its reduction with strain rate. Further tests on annealed samples indicate that the amount of residual stresses in the PCD specimens is quite negligible and does not influence the mechanical and fracture properties of the tested materials.

Acknowledgements

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