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On the Properties of Polycrystalline Diamonds

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***Abstract* - Polycrystalline diamond materials have a variety of applications, mainly as cutting tools for machining non-ferrous metals and non-metallic materials. A significant application of PCD is in the oil and gas industry for rock drilling operations. Other important areas, such as mining, have yet to reach their full potential. The unique combination of hardness, toughness and strength makes components containing PCD an excellent solution for a variety of rock-drilling operations. Furthermore, the extraordinary hardness of diamond, it's very high thermal conductivity and low friction coefficient make it an ideal tool material for the machining of wood and wood-based composites. Therefore, there are a number of reasons for the superior performance of PCD and diamond-coated carbides over tools made of cemented carbides, such as larger abrasion resistance, improved ability of machining to smaller tolerances and lowered acoustic emissions. In order to accurately predict the behavior of a PCD cutting tool under typical operating conditions encountered during drilling operations, knowledge about the range of mechanical and fracture properties is crucial. There has been very little research conducted in the area of fracture of super hard materials. Lack of available mechanical and fracture properties in addition to a poor understanding of the fracture process results in relatively high and unnecessary financial losses associated to use of these materials. Therefore, establishing some sort of standard for ranking PCD materials is essential to the development of new improved materials.**

Keywords: Polycrystalline diamond, Brittle fracture, Fracture mechanics, Young's modulus, Flexural strength, Fracture toughness.

I. INTRODUCTION

Diamond can be a single continuous crystal or may consist of many smaller crystals (polycrystal). Single-crystal diamond, which is large and transparent, is generally used for gemstones. Polycrystalline aggregates of diamonds are rarely found in nature. They consist of a number of small grains and are produced in technological processes, in contrast to natural diamond which is made in a geological process. There are several methods used to manufacture the polycrystalline diamond (PCD). The original method makes use of high pressure and high temperature (HPHT) and it is still being widely utilized due to its relatively low costs [1, 2]. Fine-grained aggregate is synthesized by sintering diamond crystallites into a coherent structure at a high temperature and pressure in the presence of a metal acting as a binder, which is usually cobalt. During sintering, grain boundaries of randomly-oriented diamond grains dissolve and join closely. Molten cobalt has a role of a solvent/catalyst and also fills in the gaps amongst the grains. Solvents effectively reduce the pressure and temperature necessary for synthesis to more easily achievable levels of about 6 GPa and 1,500 °C, and enhance the rates at which graphite to diamond transformation occurs. The other role of cobalt in the sintering process is to dissolve any graphite formed in the early stages of the synthesis cycle, and it also rounds of sharp corners of the grains and the necks between bonded diamonds and therefore reduces their effects as stress raisers. Such created material is not suitable for gems and it is used in industry as a main constituent of mining and cutting tools. PCD is often described by the average grain size of its crystals. Grain sizes could range from nanometers to hundreds of microns, usually referred to as "nanocrystalline" and "microcrystalline" diamond, respectively [3].

The properties of polycrystalline diamond depend on the details of the manufacturing processes, and they are generally superior to those of natural diamond: high hardness and strength, abrasion resistance and thermal conductivity. PCD is also considerably tougher than single crystal diamond because of the random orientation of grains in its structure which forces any propagating crack to take a strolling zigzagging path, consuming more strain energy in such a way. These properties are best utilized in cutting tools for machining a wide variety of abrasive materials, as well as in wear part applications, where they greatly contribute to improved life of the tool or wear part and offer additional technological advantages, such as process reliability and low frictional behavior. The unique combination of hardness, toughness and strength makes components containing PCD an excellent solution for a variety of rock-drilling operations. Furthermore, the extraordinary hardness of diamond, its very high thermal conductivity and low friction coefficient make it an ideal tool material for the machining of wood and wood-based composites. Therefore, there are a number of reasons for the superior performance of PCD and diamond-coated carbides over tools made of cemented carbides, such as larger abrasion

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resistance, improved ability of machining to smaller tolerances and lowered acoustic emissions.

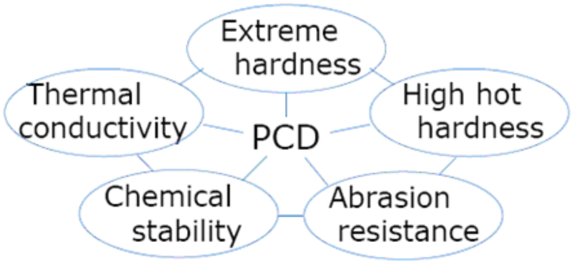

Figure 1: Properties of polycrystalline diamond

Electronic applications of synthetic diamond are also being developed, including high-power switches at power stations, high-frequency field-effect transistors and light-emitting diodes. Synthetic diamond detectors of ultraviolet (UV) light or high-energy particles are used at high-energy research facilities and are available commercially. Because of its unique combination of thermal and chemical stability, low thermal expansion and high optical transparency in a wide spectral range, synthetic diamond is becoming the most popular material for optical windows in high-power CO2 lasers [4] and gyrotrons [5, 6].

Diamond has an extremely stable covalent lattice which is responsible for many of its extreme physical characteristics, and which makes it suitable for various industrial and technological applications [7]. It is most often deemed as being “perfectly brittle” and, therefore, it is an excellent material to compare with analytical approaches of fracture mechanics.

The most significant use of PCD is in the oil and gas industry for drilling, where the cutting tool has to resist fracture (toughness),plastic deformation (hot hardness) and wear (abrasive, adhesive, tribochemical wear).Cutting tool premature failure, caused by tool fracture and chipping, is a frequent problem in the drilling industry. Considering the expenses of one day's drilling, changing the drill bit in the middle of the drilling process triggers excessive costs and therefore the most important demand on the cutting tool is to resist fracture.

The PCD can only be used for the machining of non-ferrous materials, whereas for machining hard ferrous materials such as hardened steels and grey cast iron PCBN is used. The reason for this is that ferrous materials quickly create high heat at the cutting point while machining at certain speeds. As the PCD is not thermally stable at temperature more than 700 °C, this action could easily cause cutting tools revert back to graphite. Another reason is that carbon is known to have an affinity towards iron at higher temperatures, so iron can also serve as the catalyst instead of cobalt. For these reasons, PCD materials cannot be conveniently used for ferrous applications.

The most common bit sizes used for rock drilling range from 6 1/8" to 12 1/4" in diameter, where the average rates are about 400 min-1 for bigger bits and about 975 min-1 for smaller bits [8]. In wood and metal matrix composites (MMC) machining, rates up to 30,000 min-1 are used with cutters as small as 4 mm in diameter [9, 10]. These give the loading rates of 5-6 m/s.

As the cobalt expands more than diamond when heated, above 700 °C this expansion breaks cobalt and diamond bonds, so the temperature of the PDC cutters is always tried to be maintained below 700 °C in operating conditions to avoid thermal breakdown. However, the cutting-tip temperature sometimes exceeds this critical limit [11, 12].

II. YOUNG’S MODULUS

The room temperature mechanical properties of PCD, i.e. tensile strength, transverse rupture strength, compressive strength, impact strength, fracture toughness and elastic constants, have been the topic of investigation of many researchers. Clearly, 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. [13] made remarks on Young's modulus of synthetic diamond at high temperatures. The Young's modulus of this material grown by microwave assisted chemical vapor deposition (CVD) was determined by dynamic three-point bending measurement between room temperature and 750 °C. The room temperature Young's modulus was approximately one-half of the theoretical value of 1,143 GPa. The lower Young's modulus was ascribed to the voids and micro cracks and consequently a smaller effective sample cross-section. Also, the density of the polycrystalline samples of 3.387 g/cm3 was smaller compared with single crystal diamond. Up to temperatures of 600 °C the Young's modulus remains approximately constant. At higher temperatures the start of diamond etching in air leads to a considerable decrease of the Young's modulus. The measured Young's modulus was in this case basically determined by the remaining undamaged sample cross-section.

Young's modulus testing of PCD/WC-Co specimens was carried out by Belnap and Griffo [14] by tapping the simply

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supported samples and measuring their resonant frequency, according to the relevant testing standard [15]. Ultrasonic tests on PCD samples have also been attempted in the current work, according to [16], but unfortunately have not returned satisfactory results due to miniature specimen sizes.

III. FLEXURAL STRENGTH

Some mechanical properties of CVD and natural diamond, as well as polycrystalline diamond compact (PDC), have been investigated at room temperature. Tensile and fatigue strength of free-standing CVD diamond was measured in three point bending [17, 18, 19]. Field et al. [7, 20] 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 [21, 22]. 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 scatter in the results may be also a result of differences in direction of crack advancement (generally 0-10°). Other possible reasons for discrepancies include uneven distributions of impurities and micro-defects in different regions of the diamond film specimens.

IV. FRACTURE TOUGHNESS

The measurement of fracture toughness (K_{Ic}) of relatively hard brittle materials (ceramics, hardmetals, tools steels, etc.) is somewhat of a challenge, especially for small test-pieces. Lammer [23] has shown that polycrystalline diamonds behave in a manner similar to that of most engineering ceramics, but have the distinct advantage of higher fracture toughness. Relative qualities and advantages of five different methods of fracture toughness testing are investigated by Damani et al. [24]: the chevron-notched beam, the indentation fracture, the indentation strength in four-point bending, the single-edge pre-cracked beam in four-point bending (SEPB-B) using popped in bridging cracks, and the single edge notch bend - saw cut (SENB-S) methods.

A chevron notch has a V-shaped ligament, such that the notch depth varies through the thickness, with the minimum notch depth at the center [25, 26, 27, 28, 29]. It 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 unfeasible in super hard PCD material, this feature cannot be exploited in this case. Also, as the introduction of chevron notch is more complicated and time consuming than simple V notch.

Nano indentation is another method used to measure the fracture toughness of thin films of brittle materials at small volumes. The method was developed by Lawn, Evans and Marshall [30]. The interface toughness is obtained from direct measurements of the applied load, crack length and material properties without imaging the indentation [31]. Numerous fracture toughness investigations made by the indentation technique on nano polycrystalline diamond and CVD diamond film have been published [32, 33, 34]. However, while this technique might be useful to see the relative difference in hardness and toughness between two grades of material, repeatability levels are understandably low. The reason is that indentation techniques suffer from large uncertainties deriving from a number of factors including subjectivity of crack length measurement depending on observation conditions, residual stress field from indentation, variability of indentation crack shape, susceptibility to residual stress in the prepared surface, and results which may be indentation force dependent [35]. Moreover, when indenting the PCD material, the softer indenter will generally fail.

According to [24], amongst all mentioned methods, the SENB method was found to provide the most reproducible results and was chosen for this investigation. The major limiting feature of SENB method, however, is a limiting critical notch root radius, above which the values of K_{Ic} determined are systematically too high. It is shown that if correct values of fracture toughness are to be determined with the SENB method, the notch width must be of the order of the size of the relevant micro structural or machining-induced defects (e.g. large pores and weak grain boundaries). Fischer et al. [36] have recently investigated the influence of the preparation of ceramic SEVNB specimens on fracture toughness results. They determined and quantified the influence of the notch root radius on the fracture toughness value for the high strength ceramic material zirconia (ZrO_2). The notch root radii showed a pronounced effect on the determined fracture toughness values. It was considered extremely important that the notch of a SEVNB sample is properly sharpened to be able to determine the true K_{Ic} value. A similar investigation has been done by Morrell [35]. He focused on conditions which strongly influence the fracture toughness results and outlined a procedure for the straightforward introduction of a sharp crack, which is the most important parameter affecting the accuracy of the results. Since the test piece production costs are significant, most attention is dedicated to the simple beam geometries, and to the methods of introduction of the sharp cracks. In the case of the SEVNB, a sharp notch is machined into the specimen by honing using a razor blade and a diamond paste. The main question is then how closely the sharpened notch represents a sharp crack. This highlights the main restriction of this

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method: the notch root radius needs to be very small. In the case of blunt notches, residual stresses from machining inhibit sharp crack development. Techniques for the production of sharp cracks have been reviewed by Warren and Johannesson [37]. Major advantages and disadvantages were outlined.

The development of a procedure for fatigue crack growth in a PCD material was outlined by Achilles and Brondsted [38]. Initiation of the fatigue crack is greatly sensitive to the quality of the surface finish and the geometry of the starting notch. The fatigue crack was found to grow in both a continuous and an irregular sporadic crack growth mode. This behavior was explained in terms of the R-curve crack growth behavior. An attempt was also made, by the same authors, to correlate the crack growth rates and the crack morphology. Naturally, for this hard and brittle material, the introduction of a fatigue pre-crack is a nearly impossible task. The load required to grow a fatigue crack is very close to the load which would cause unstable crack growth and most of the samples may be lost. Some attempts were undertaken to obtain controlled pre-cracking in specimens using certain stiff pre-cracking facilities [39], which rely on the concept of ensuring that the energy required for an increment in crack length is greater than the driving energy available in the test-piece itself and in the machine. Although the developed methods can be made to work for the majority of advanced technical ceramics, they do not work adequately for tougher hard metals and tool steels, as well as with PCD.

Veldkamp and Hattu [40] dealt with the influence of several structural parameters, such as porosity, grain boundaries and second-phase inclusions on the fracture toughness of brittle materials. Experimental data from the literature on the influence of, for instance, porosity on fracture toughness have also been used. The main tool used for explaining the phenomena observed is the fracture process zone, which is defined here by an effective size and an effective surface area of the propagating crack per unit volume created during propagation. It was shown that the parameters often have counteracting effects and that it is difficult to vary one particular parameter. Porosity will always have a negative influence on the fracture toughness. Instead of the grain size, the fraction of transgranular fracture on the fracture surface appeared to determine the fracture toughness. Moreover, the toughness of the boundaries and the anisotropy of the grains were found to be important. Tough inclusions can under certain conditions have a positive effect which can be further enhanced by thermal stresses. The same holds for micro cracking in the matrix material but this effect also needs further investigation.

The diametric compression test, also known as a Brazilian test or an indirect tensile test, is commonly used to determine fracture properties of brittle materials [38, 41, 42], in which a pre-cracked disc-shaped specimen is loaded diametrically in compression in order to generate tensile failure in the materials. During the sintering process of the polycrystalline diamond compacts (PDC), WC-Co liquid from the WC disc is infiltrated into a diamond powder providing a liquid phase to facilitate inter-grain diamond bonding. Finer diamond sizes tend to have a higher volume fraction of metallic content. The role of this residual metallic content of the diamond layer in conjunction with that of the average grain size on the diamond layer fracture toughness was investigated by Miess and Rai [43] using a diametric compression test. Larger grain PCD compacts having lower amounts of metallic content were found to have a higher toughness than fine grained materials with higher amounts of residual metallic phase. However, the results of some studies have shown that the properties obtained by diametric compression test are greatly influenced by the magnitude of compression load [44]. Wide variation between specimens can be experienced for brittle materials as cracks easily grew in certain preferred direction and not necessarily in mode I [45]. Another problem with the use of Brazilian disk technique for fracture toughness determination of PCD is that it is much more difficult to machine and nearly impossible to sufficiently sharpen the notch on disk specimens, particularly for fine grained materials.

V. OTHER PROPERTIES

Inter-granular wear, grain cleavage, peeling and spalling of grains have been proposed as possible wear mechanisms of the PCD [46, 47, 48, 49, 50]. Glowka and Stone [51] argued that two distinct failure modes of diamond grains are possible on a microscopic level. After impact with abrasive rock particles, some diamond grains crush and the edge of the grain is slowly removed in small pieces. Others exhibit cleavage fracture across the entire grain. This dual behavior is attributed to the development of deformation twin bands in some individual diamond grains during the HPHT sintering process. At increased operating temperatures (but still below 750 °C), the micro chipping mode of wear increases in intensity [52, 53]. This behavior is caused by a decrease in the hardness of individual diamond grains.

Increase in wear performance with decreasing grain size of PCD is reported by Horton and Horton [54] and Ding et al. [55]. Their test consisted of turning a cylindrical piece of granite and MMC, respectively, on a lathe which is a common test in the oil well drilling industry. However, it has also been found by the same authors, when turning aluminum alloys

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under extremely high wear conditions, that coarse grained PCD was substantially more wear resistant than finer grained.

Bex and Robertson [56], when tested PCD material of various grain sizes, reported an increase in shock resistance with decreasing grain size. For their tests, the test pieces were clamped in a standard milling head and used to fly cut extremely hard, abrasive granite.

Willmott and Field [57] reported experimental measurements of fracture velocities between Rayleigh wave speed c_R and 1.3 c_R from well-calibrated high-speed photographic sequences of shocked diamond. Noticeably, these velocities are greater than the formerly suggested limiting velocities in theoretical and practical studies of single mode I cracks in diamond and other materials. A mechanism to load the fracture surfaces in tension at velocities higher than c_R is described. High crack velocities could be most likely explained by a multiple initiation mechanism. According to this, single cracks are coalescing with secondary cracks in the same cleavage plane in an anisotropic material.

It can be seen from this literature review that published fracture data for sintered PCD compacts is relatively scarce and somewhat limited in nature. Values are mostly confined to properties determined at low loading rates and room temperature. However, these properties are rather inadequate when investigating the fracture of PCD under typical working conditions. On one hand, the actual situation offers great opportunities for testing and improvement of these materials, but on the other hand, it is quite difficult to measure the properties of the hardest and nearly perfectly brittle materials at elevated temperatures and rates of loading. In addition, the costs involved in research of this kind are very high, which is main reason why a systematic study of this kind has not been performed to date.

VI. DISCUSSION

There has been very little research conducted in the area of fracture of superhard materials. The sparse data which is currently available in the literature is not sufficient to accurately predict the behavior of a PCD cutting tool under typical operating conditions encountered during drilling operations. Mechanical and fracture properties at low rates and ambient temperature available to date in the literature are inappropriate for analyzing the behavior of PCD materials in actual working conditions.

The accurate and efficient determination of the fracture toughness and the associated fracture mechanisms of a polycrystalline diamond are of fundamental importance to PCD material manufacturers and end users alike. The ability to predict the behavior of such materials can lead to better designs, improved performance, reduced costs and enhanced safety. Consequently, there has been a growing motivation for the development of theoretical models for predicting the damage and failure of superhard materials. These models require the definition of geometry independent material parameters, which are usually obtained from tests with simpler geometries and applied in the analysis of more complex geometries.

The boundaries have to be pushed to dynamic rates and high temperatures very close to the temperature of diamond graphitization. These new data would certainly improve the way in which PCD materials are ranked and allow better insight to be gained into the fracture process. Based on these contributions, there might be a significant commercial potential through a market demand for products which could be developed, as well as economic and technological impact through improved PCD tool performance in oil and gas industry.

VII. CONCLUSIONS

Polycrystalline diamond materials have a variety of applications, mainly as cutting tools for machining non-ferrous metals and non-metallic materials. A significant application of PCD is in the oil and gas industry for rock drilling operations. Other important areas, such as mining, have yet to reach their full potential. These cutters/tools are subjected to high operating temperatures, impact loads and abrasive wear during these operations, which may lead to their sudden failure and high associated costs.

Due to the lack of available mechanical and fracture properties in addition to a poor understanding of the fracture process at high strain rates and high temperatures, current standards in the industry for ranking superhard materials might be well called a "sledge hammer" approach. Therefore, establishing some sort of standard for ranking PCD materials is essential to the development of new improved materials. Moreover, the ranking of a material at low rates and low temperatures may not necessarily be the same at high rates and high temperatures. For this reason, a thorough study of the mechanical and fracture characteristics of PCD needs to be undertaken. Once our understanding of the fracture process is improved, the structure and composition of these materials can be engineered with a view to optimizing the properties required for specific applications.

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