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Title	Thermal shock resistance of polycrystalline cubic boron nitride
Authors(s)	Carolan, Declan, Ivankovic, Alojz, Murphy, Neal
Publication date	2012-08
Publication information	Carolan, Declan, Alojz Ivankovic, and Neal Murphy. "Thermal Shock Resistance of Polycrystalline Cubic Boron Nitride" 32, no. 10 (August, 2012).
Publisher	Elsevier
Item record/more information	http://hdl.handle.net/10197/4613
Publisher's statement	This is the author's version of a work that was accepted for publication in Journal of the European Ceramic Society. Changes resulting from the publishing process, such as peer review, editing, corrections, structural formatting, and other quality control mechanisms may not be reflected in this document. Changes may have been made to this work since it was submitted for publication. A definitive version was subsequently published in Journal of the European Ceramic Society (32, 10, (2012)) DOI: http://dx.doi.org/10.1016/j.jeurceramsoc.2012.03.013
Publisher's version (DOI)	10.1016/j.jeurceramsoc.2012.03.013

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Thermal Shock Resistance of Polycrystalline Cubic Boron Nitride

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Abstract

The effect of thermal shock on the flexural strength has been investigated experimentally. It was found that the variation in flexural strength with quench temperature was influenced by the CBN grain size. Polycrystalline material containing small CBN grains showed a discontinuous drop in measured flexural strength above a material dependent critical quench temperature difference, ΔT_c . The sharp decrease in measured strength is accompanied by unstable crack propagation. Material containing a significantly larger CBN grain size, exhibited a gradual decrease in strength above the critical quench conditions. The experimental observations agreed with an established theory developed for thermal shock of alumina. The theoretically calculated critical temperatures agree well with the observed experimental data for each material when a flaw size equal to the CBN grain size is employed.

Keywords: Cubic Boron Nitride, fracture, thermal shock

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1. Introduction

Polycrystalline Cubic Boron Nitride (PCBN) is a super hard material used in the machining of hardened steels, aerospace grade alloys and other abrasive materials [1, 2, 3]. In these applications the tools are subjected to high operating temperatures, abrasion and impact loading. This can lead to the brittle fracture of the tool. Accurate determination of the fracture characteristics of PCBN under a wide range of loading rates and temperatures is therefore essential in order to evaluate the performance of the tool under these highly demanding operating conditions. The current work examines the thermal shock performance of two grades of PCBN. Thermal shock occurs when a material is exposed to temperature extremes in a short period of time. Under these conditions, the material is not in thermal equilibrium, and internal stresses may be sufficient to cause fracture of the material. The ability to withstand thermal shock is a function of several variables, including the thermal conductivity, k , the coefficient of thermal expansion, α and the specific heat capacity, c . Winkelman and Schott [4] proposed an empirical parameter they called the coefficient of thermal endurance which gives a qualitative estimate of the ability of a material to withstand thermal shock.

$$E_{th} = \frac{F}{\alpha E} \sqrt{\frac{k}{\rho c}} \quad (1)$$

where E_{th} is the coefficient of thermal endurance, not to be confused with the Young's modulus E , which appears on the right hand side of Equation 1 and F is the material tensile strength. Selected coefficients of thermal endurance are given in Table 1 [6]. It is significant to note the large difference in thermal endurance between cubic boron nitride and a variety of typical PCBN binder

phases. This is an indicator that fracture of polycrystalline cubic boron nitride may be a thermally controlled phenomenon. A theoretical analysis of

Material	E_{th}
Cubic Boron Nitride	648
Diamond (Type IIA)	30.29
Aluminium Nitride	2.325
Silicon Carbide	1.4
Alumina (99%)	0.640
Alumina (96%)	0.234

Table 1: Thermal endurance factor for selected materials.

crack propagation in brittle ceramics under the influence of thermal stresses was conducted by Hasselman and others [5, 7]. They found that for a material with small cracks, which propagate kinetically on initiation, the crack length and material strength are expected to change with the severity of quench, shown schematically in Figure 1. Specimens with initial crack lengths longer than some critical value were found to propagate stably. Further to this, Gupta [8] found for alumina of varying grain sizes the extent of unstable crack propagation was inversely proportional to the grain size of the material. More recently several researchers have applied the analysis to composite ceramic materials [9, 10, 11, 12] as well as functionally graded ceramics [13]. A significant result from the work by Hasselman and others [5] is the difference between resistance to crack initiation and resistance to crack propagation.

Resistance to crack initiation, R' , and crack propagation, R'' , is given by:-

$$R' = \frac{GE}{\sigma_t^2(1-\nu)} = \frac{K^2}{\sigma_t^2(1-\nu)} \quad (2)$$

$$R'' = \sqrt{\frac{G}{\alpha^2 E}} = \frac{K}{\alpha E} \quad (3)$$

where G is the fracture energy, K is the stress intensity factor, E is the Young's modulus of the uncracked specimen and σ_t is the tensile strength of the material. It can be clearly seen that resistance to crack initiation is proportional to K^2 and inversely proportional to σ_t^2 whereas resistance to crack propagation is linearly proportional to K . Therefore a material will be more resistant to crack initiation if it has a larger fracture process zone size since most estimates of process zone size indicate a proportionality to $(K/\sigma)^2$ [14, 15, 16], whereas resistance to crack propagation is purely a function of toughness. The intrinsic strength of the material does not affect the materials ability to resist crack propagation. Hasselman developed an analytical expression to calculate the critical temperature difference at which the flexural strength begins to decrease. For initially short cracks this is given as [5, 17]:-

$$\Delta T_c = \left[\frac{\pi\gamma(1-2\nu)^2}{2E\alpha^2(1-\nu^2)^2} \right]^{1/2} c^{-1/2} \quad (4)$$

where γ is the energy required to create a new surface ($\gamma = G/2$), α is the linear expansion coefficient, E is the Young's modulus of an uncracked specimen, ν is the Poisson's ratio and D is the grain size.

2. Materials and Methods

Rectangular specimens of length, 28.5 mm, width, 6.25 mm and thickness 4.76 mm were provided by Element 6 for testing. PCBN material is generally

sintered under high temperature (1200° C - 1500° C), high pressure (4 GPa - 7 GPa) conditions. Two grades of PCBN material were provided. The first grade, denoted PCBN A has a grain size of 22 μm , with 90% vol. CBN grains. The second grade, denoted PCBN B has a grain size of 1 μm with a lower volume percent of CBN, approximately 60%. The binder phase for PCBN A is best described as aluminium based ceramic, while for PCBN B the binder is a typical titanium carbo-nitride binder. Typical micrographs for the two materials under investigation are given in Figure 2. Thermal constants for the constituent materials of both PCBN A and PCBN B are shown in Table 2 [18]. In total 84 samples were tested in three point bending, 46 for PCBN

	PCBN A	PCBN B
G_{PCBN} [J/m ²]	70	11
D [μm]	22	1
E_{CBN} [GPa]	800	800
ν_{CBN}	0.14	0.14
α_{CBN} [$10^{-6}/\text{K}$]	1.2	1.2
E_{binder} [GPa]	315	250
ν_{binder}	≈ 0.2	≈ 0.2
α_{binder} [$10^{-6}/\text{K}$]	4.8	9.4

Table 2: Thermal properties of PCBN grades and binder constituents [18].

A and 46 for PCBN B. For each grade, 15 were samples were tested in the *as-received* condition and a full Weibull analysis of the strength was carried out [19]. A full description of the Weibull's method is given in [20]. The important outputs from the analysis are the characteristic strength, σ_0 and

the Weibull modulus, m , a measure of the degree of scatter of the data. The flexural strength of each sample was calculated via Equation 5 in accordance with BS-EN 843:1 [21] where σ_f is the flexural strength, P_{in} is the breaking load, b is the width, h is the height and s is the span of the supporting rollers. The samples were loaded to fracture using a constant crosshead displacement rate of 1 mm/min.

$$\sigma_f = \frac{3P_{in}s}{2bh^2} \quad (5)$$

The remaining samples were then subjected to a variety of heat treatments, water quench with quench temperature difference, ΔT , varying between 220° C and 1080° C, oil quench, 220° C < ΔT < 1080° C and water quench with subsequent anneal. The oil used was a light grade oil. Approximate heat transfer coefficients for unagitated water, oil and air quenching are give in Table 3. For each heating cycle, samples were heated at a rate of 40° C/min

Quench Media	h [W/m ² K]
Water	5000
Oil	2000
Air	200

Table 3: Approximate heat transfer coefficients for various media.

up to the required holding temperature and then held at that temperature for a period of one hour. The annealing temperature in all cases was the same as the quench temperature and the anneal hold time was 2 hours. After annealing the samples were allowed to cool slowly in the closed furnace. They were subsequently fractured in three point bend using the method previously described.

3. Results and Discussion

Flexural strength results for both grades of PCBN investigated are presented in Figure 3. The Weibull modulus, m , was lower for PCBN B, $m = 7.4$, than for PCBN A, $m = 15.7$, indicating a greater scatter in strength data for the smaller grained material. Figures 4 and 5 present the results of the various heat treatments, oil quench, water quench and water quench with subsequent annealing from the quench temperature on both PCBN A and PCBN B. PCBN A exhibits stable crack propagation for water annealing at all levels of quench as explained in Figure 1. The maximum quench temperature difference, 1080°C , resulted in a drop in strength to 87.6 MPa , 20% of the *as-received* value. Oil quenching had no effect on the measured flexural strength of PCBN A. An oil quench on PCBN B has the effect of increasing the average flexural strength. The oil quench, which is a less severe quench than a water quench, may have had the effect of relieving the existing residual stress field, caused by the sintering and machining of the sample. A severe water quench causes a large discontinuous drop in flexural strength for a quench temperature difference of between 240°C and 375°C . This type of behaviour can be termed unstable crack growth as explained previously in Figure 1. Below 240°C no drop in flexural strength was recorded. The flexural strength recorded for a quench temperature difference of 780°C and above was very low, ($\approx 25\text{ MPa}$). It was noted that the failure was very low energy and the fracture initiated from the centre of the sample. This is clearly illustrated in Figure 6. The failed material also exhibited long unopened cracks extending from the initiation site to the edge of the sample as shown in Figure 7. A significant surface colour change was also noted

on quenching at these temperatures, from dark grey to light blue and upon microscopic investigation, significant voids close to the surface were noted as shown in Figure 8. It is thought that this colour changes is a result of the oxidation of titanium based binder near the surface. This has not been investigated further in this work. It was found that simply heating either material up to a temperature following by slow cooling in the furnace showed no change in the measured value of flexural strength for PCBN B. Therefore furnace heating alone does little to anneal the residual stresses and there exists some intermediate rate of cooling which appears to be beneficial in terms of the measured flexural strength at least for PCBN A. Using the thermal properties for the binders in both cases from Table ?? in conjunction with Equation 4, we find that for an initial crack length, c , equal to the average CBN grain size, D , of the material, $\Delta T_c = 326^\circ \text{C}$ for PCBN A and 319°C for PCBN B. This prediction agrees with the experimental data shown in Figures 4 and 5. If, on the other hand, the critical temperature is calculated using the thermal properties of CBN grains, $\Delta T_c = 1070^\circ \text{C}$ for PCBN A and 1990°C for PCBN B. The available data on the thermal properties of the composite materials, i.e. binder and CBN together, are obtained under steady-state experimental conditions and do not take account of any transient behaviour which may occur for short time intervals. It is important to note that the thermal properties presented in Table 2 are room temperature values. However work by Carolan [22] has shown that the bulk thermoelastic properties of both PCBN A and PCBN B are not significantly affected by a global increase in temperature up to 800°C . Thus, the use of room temperature properties in the thermal shock calculations is justified.

4. Conclusion

The thermal shock resistance of two grades of PCBN material is determined experimentally by measuring the flexural strength of the material after being subjected to a quench treatment. It was found that the thermal shock resistance of the material was governed by the thermal properties of the binder phase and not by the thermal properties of either the composite materials or the CBN grains. However the size of the CBN grains plays an important role in determining the type and extent of crack propagation. It was shown that the crack can propagate either kinetically or quasi-statically depending on the initial crack length. Excellent agreement was found with an existing theory of thermal shock developed for monophase materials if the initial crack length was assumed to be a flaw of one CBN grain in length. Microscopic examination of fracture surfaces revealed that the locus of failure for severely quenched material was located in the centre of the specimen where tensile residual stresses are known to occur as a result of the rapid cooling. It is concluded that the theory developed by Hasselman sufficiently describes the thermal shock characteristics of PCBN.

5. Acknowledgements

The authors are grateful to Dr. Alun Carr and Dr. Kenneth Stanton for helpful discussions. This research was made possible by the financial support of *Element 6 Ltd, The Irish Research Council for Science, Engineering and Technology* and *Enterprise Ireland*.

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Figure 1: Crack propagation and strength for unstable (a) and stable (b) crack propagation conditions [5].

(a) PCBN A

(b) PCBN B

Figure 2: Scanning electron micrographs of PCBN A and PCBN B showing CBN particles (dark phase).

(a) PCBN A

(b) PCBN B

Figure 3: Weibull plot of strength data for (a) PCBN A ($n = 15$, $m = 15.7$, $\sigma_0 = 438$ MPa) and (b) PCBN B ($n = 15$, $m = 7.4$, $\sigma_0 = 670$ MPa).

Figure 4: Flexural strength of PCBN A quenched in water and subsequent annealing from quench temperature as a function of quench temperature difference $[\Delta T]$.

Figure 5: Flexural strength of PCBN B quenched in oil and water as a function of quench temperature difference $[\Delta T]$.

Figure 6: SEM Micrographs showing main features of quench failure of PCBN
B.

Figure 7: Knitted SEM micrograph of long unopened crack extending from failure initiation region to edge of sample.

Figure 8: Fracture surface of PCBN B flexural strength sample, quenched from 1100° C. Microscopic voids are clearly evident close to the surface which are not present in the unquenched sample.