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Elastic Properties of Polycrystalline Cubic Boron Nitride and Diamond by Dynamic Resonance Measurements

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The elastic properties of ceramic materials are important to their performance in severe abrasion and wear applications and also provide a useful, quantitative measure of quality. We have measured the Young’s modulus E, shear modulus G, and Poisson’s ratio ν of several commercial polycrystalline cubic boron nitride and diamond products, in the form of free-standing disks, using the dynamic resonance method. The latter method is accurate and fast enough for routine quality control. Measurements on polycrystalline cBN yielded values of E, G, and ν in the ranges of 630-770 GPa, 270-340 GPa, and 0.14-0.18, respectively, depending on the volume fraction of superabrasive, binder phase and microstructure. As a point of comparison, the orientation-averaged values of E, G, and ν for pure, equiaxed, polycrystalline cBN are calculated as 909 GPa, 405 GPa, and 0.12, respectively. Measured values of E, G, and ν for sintered diamond lay in the ranges of 915-990 GPa, 415-450 GPa, and 0.10-0.11, respectively. The modulus results are compared to selected additional material properties.

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I. INTRODUCTION

Sintered, polycrystalline diamond (PCD) and cubic boron nitride (PCBN) have many outstanding properties, combining hardness and abrasion resistance with excellent toughness [1]. These products have been available commercially for many years and are useful in a wide variety of machining, drilling, and wire die applications. As with any ceramic, however, consistent performance depends on the reliable control of material properties.

We report the first application of the dynamic resonance method to the evaluation of the elastic constants of PCBN and PCD in the form of free-standing disks. This method requires inexpensive equipment and is very fast, yet can accurately determine both the Young’s modulus and shear modulus. The measured elastic constants are compared to theoretical values for pure, polycrystalline, equiaxed cBN and diamond as derived from measurements on single crystals, and are correlated with some additional material properties.

II. THEORETICAL BACKGROUND

The dynamic resonance method is a well-established technique for the evaluation of elastic constants [2-4]. In the original approach, vibrations in the specimen were driven by an external, variable-frequency oscillator and the frequency response was measured. However, the impulse technique, where oscillations are induced in the part by tapping and the transient “ringing” signal is detected by a microphone and digitally analyzed, is much simpler experimentally and yields the same information. The application of the technique to rectangular bars is well known and has been established as an ASTM standard [4]. The method has been successfully applied to free-standing [5] and thin-film diamond [6] grown by chemical vapor deposition, and has also been used to measure relative values of the Young’s modulus of PCBN as a function of temperature [7].

The elastic properties of free-standing disks may also be extracted from the torsional and flexural mode frequencies, which is not widely known. Accurate equations relating the resonant frequencies of free-standing disks to their elastic constants were first derived by Martincek [8], and a more refined set of tables was calculated by Glandus [9,10]. These equations are believed to be accurate to approximately 1% or better for a wide range of specimen geometries and elastic constants. The mode with the lowest frequency, the fundamental torsional vibration, has bisecting nodal lines and is illustrated in Fig. 1(a). The flexural vibration is a biaxial drumhead mode with a nodal circle occurring at 68.1% of the disk diameter [8-10], as illustrated in Fig. 1(b). The extension of the impulse dynamic resonance method to disks has recently been proposed as an annex to ASTM Standard C1259 [11].

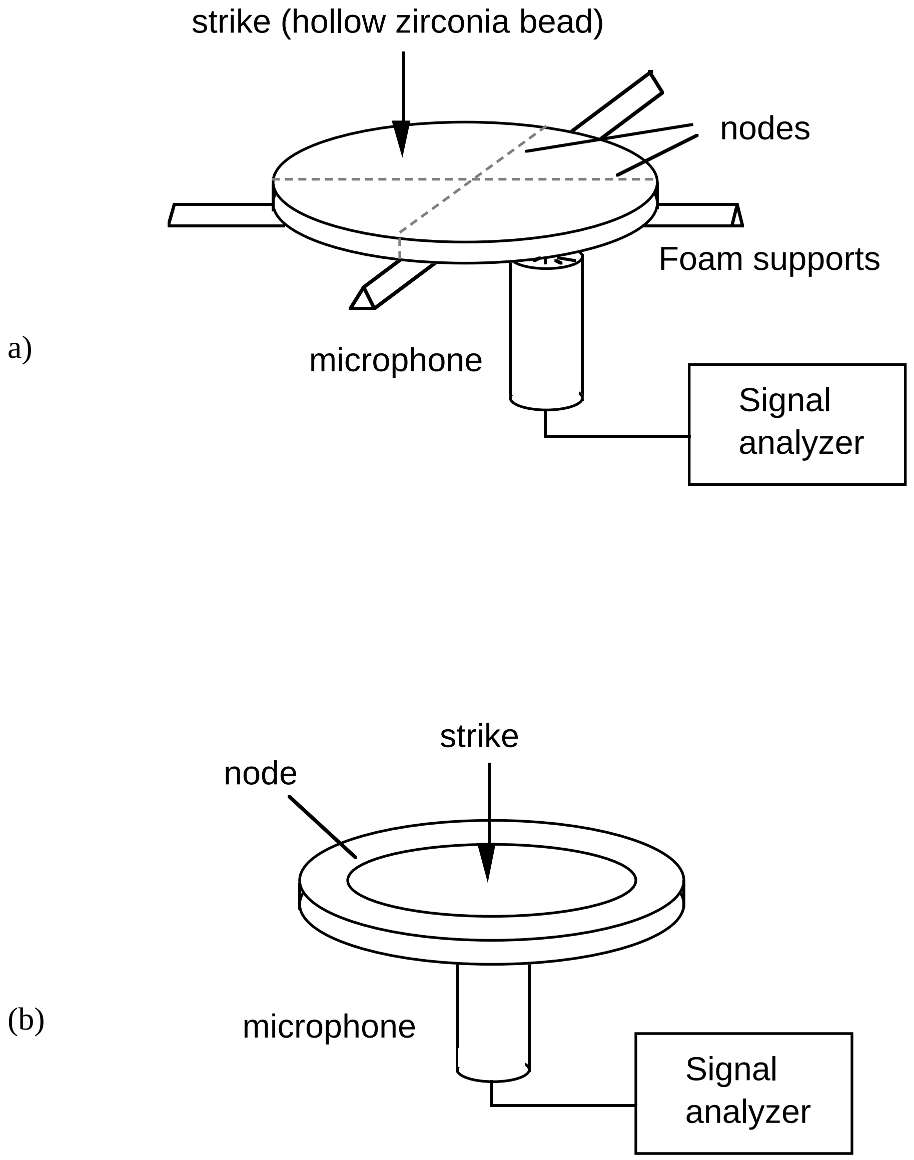

Figure 1. Schematic illustration of ringing measurements on the (a) torsional mode or (b) flexural mode of a disk. The flexural vibration is a biaxial drumhead mode with a nodal circle at 0.681 of the diameter of the part.

The equations relating the Young's modulus and shear modulus to the torsional and flexural frequencies, f_T and f_F, respectively, are summarized below. In these equations d is the diameter of the disk, t is the thickness, and ρ is the density. The Poisson's ratio is derived directly from the ratio of the flexural and torsional frequencies and t / d by interpolation from tabulated values [8-10]. For relatively thin disks ($t / d \lesssim 0.05$) [5],

$$\nu \quad = \quad -3.3382 + 3.834(f_F / f_T) - (f_F / f_T)^2 \tag{1}$$

Once the value of ν is evaluated, the Young's modulus is calculated from

$$E \quad = \quad \tfrac{3}{2}\pi^2\rho(1-\nu^2)(d^2 / t)^2[(f_F / K_F)^2 + (f_T / K_T)^2] \tag{2}$$

The numerical factors K_F and K_T depend weakly on ν and the t / d ratio and are calculated by interpolation from tabulated values [9,10]. The value of the shear modulus then follows from

$$G \quad = \quad E / [2(1+\nu)]. \tag{3}$$

Finally, the bulk modulus [12] is calculated as

$$B \quad = \quad EG/(9G - 3E). \tag{4}$$

III. EXPERIMENTAL DETAILS

Free-standing GE Superabrasives Borazon® (BZN) 6000 and 8100 PCBN specimens were prepared by grinding off the tungsten carbide substrate to which the polycrystalline cBN is bonded, and then lapping to obtain disks of the desired thickness. BZN 7000 is fabricated as free-standing disks, but these specimens required thinning (by grinding) so that the vibrational frequencies would be within the sensitivity range of the microphone. Disk specimens of GE Superabrasives Compax® 1500 and 1600 PCD were prepared by similarly grinding off the tungsten carbide substrate and lapping to the desired thickness.

Dynamic resonance experiments were performed in separate laboratories at CRD and at GE Superabrasives. For measurement of the torsional mode frequency, the disks were supported on thin foam strips or tensioned cotton threads crossing at a 90° angle, defining the nodal lines, and were excited by dropping a hollow zirconia bead weighing approximately 25 mg from a height of 5-8 cm near the nodal circle of the flexural mode, equidistant between the supports (cf. Fig. 1(a)). This procedure maximizes the amplitude of the torsional mode relative to the flexural mode. The ringing sound was detected with a microphone/preamplifier located under the opposite side of the specimen. In the experiments at CRD, the resonant frequency was measured using a digital oscilloscope with fast-Fourier-transform capability, whereas at Superabrasives the frequency was evaluated using a Grindosonic instrument [13], which performs the signal analysis automatically and reports the resonant frequency on a digital readout. To detect the flexural mode with minimal interference from the torsional mode, the disks were supported on 3 or 4 small foam buttons under the nodal circle, a zirconia bead was dropped onto the center of the disk (node of the torsional mode), and the microphone was placed under the center of the specimen (cf. Fig. 1(b)). Placement of the specimen on the fixture, dropping the beads, and acquisition of several ringing measurements required less than one minute per sample. For the parts analyzed here K_F and K_T in Eq. (2) are approximately equal to 8.5 and 5.7, respectively.

Additional material property measurements are reported here for completeness and comparison with the elastic constants of the PCBN samples. The Vickers hardness was measured [14] using an 8 kg load. The fracture toughness was evaluated from the average length of cracks produced by a Vickers indenter at an 8 kg load (Palmqvist crack model [15]). The transverse rupture strength was evaluated by a 4-point bend test [16] using a down-scaled fixture with upper and lower spans of 6.76 mm and 13.51 mm, respectively. The bend test specimens were carefully-prepared rectangular bars measuring 0.64 mm thick and 1.27 mm wide. Transverse rupture strength values are often obtained from a 3-point bend test [17,18], which is less useful for quantitative material characterization purposes but yields systematically larger values due to the smaller specimen volume placed in tension relative to the 4-point technique.

IV. RESULTS

A total of 28 BZN and 4 PCD specimens measuring 18-33 mm in diameter and 0.5-1.5 mm thick were tested, yielding flexural and torsional frequencies between 6 and 100 kHz. The Poisson’s ratio, Young’s modulus, shear modulus, and bulk modulus were then calculated for each specimen using Eqs. (1)-(4), and the BCBN and PCD results are summarized in Tables 1 and 2, respectively. Finally, hardness, fracture toughness, and fracture strength measurements were performed on a different set of representative PCBN specimens, and the results are also included in Table 1. The standard deviations of the Young’s modulus and shear modulus of BZN 8100, BZN 7000, BZN 6000, and PCD 1500 were 2%, 1%, 3%, and 2.5%, respectively.

Several groups [19-21] have reported the effective Young’s modulus, shear modulus, and Poisson’s ratio for pure, equiaxed, polycrystalline diamond, as calculated from the single-crystal elastic constants. The results ($\overline{E}$ = 1143 GPa, $\overline{\nu}$ = 0.069) are included in Table 1 for comparison with the properties of sintered PCD.

We have calculated the effective Young’s modulus, shear modulus, and Poisson’s ratio for pure, equiaxed, polycrystalline cBN by the Hershey-Kröner-Eshelby orientation-averaging method [20,22], using the single-crystal elastic constant data recently reported by Grimsditch et al. [23]. The orientation-averaged Young’s modulus and Poisson’s ratio may be written in terms of B (a rotationally-invariant scalar) and $\overline{G}$ as

$$\overline{E} = 9B\overline{G} / (\overline{G} + 3B) \tag{5}$$

$$\overline{\nu} = (3B/2 - \overline{G}) / (\overline{G} + 3B) \tag{6}$$

where B is calculated directly from the single-crystal elastic stiffness constants as $(c_{11} + 2c_{12})/3$ [12]. The orientation-averaged shear modulus $\overline{G}$ is determined by solving the Hershey-Kröner-Eshelby equation [20,22]

$$8\overline{G}^3 + (9B+4C')\overline{G}^2 - 3c_{44}(B+4C')\overline{G} - 6Bc_{44}C' = 0 \tag{7}$$

Table 1. Summary of results on PCBN including means and standard deviations. Tabulated are: volume % superabrasive (%), diameter (*d*), thickness (*t*), mass (*m*), torsional frequency (f_T), flexural frequency (f_F), Young's modulus (*E*), shear modulus (*G*), bulk modulus (*B*), and Poisson's ratio (ν). Also included are typical values of the Vickers hardness (*H*), fracture toughness (K_{IC}), and transverse rupture strength (σ) of BZN compacts.

Product	%	#	d	t	m	f_T	f_F	E	G	B	ν	H	K_{IC}	σ
			mm	mm	g	kHz	kHz	GPa	GPa	GPa		GPa	MPa m$^{1/2}$	MPa
BZN 8100	60	1	25.15	0.638	1.446	12.89	19.51	657	280	336	0.174			
		2	25.15	0.640	1.452	12.94	19.60	657	279	338	0.176			
		3	25.15	0.648	1.422	13.15	19.95	643	273	333	0.178			
		4	34.08	0.682	2.729	7.58	11.48	631	268	326	0.177			
		5	34.08	1.276	4.954	14.42	21.85	651	277	334	0.175			
mean								**648**	**275**	**333**	**0.176**	**30**	**7.8**	**846**
±								**11**	**5**	**5**	**0.002**			
BZN 7000	80	1	18.31	1.519	1.351	67.83	99.80	690	301	325	0.146			
		2	18.31	0.935	0.829	43.05	64.07	712	309	343	0.152			
		3	18.31	1.191	1.057	54.65	80.87	715	311	341	0.150			
		4	18.29	1.247	1.106	57.04	84.37	711	309	337	0.149			
		5	18.26	0.861	0.759	39.74	59.24	703	304	341	0.156			
		6	18.29	0.960	0.850	44.02	64.94	699	306	326	0.142			
		7	18.29	1.295	1.149	59.32	87.59	713	311	338	0.148			
		8	18.29	1.044	0.924	48.10	71.27	712	310	338	0.149			
		9	18.31	1.135	1.006	51.65	75.90	696	306	321	0.139			
		10	18.31	1.328	1.174	60.64	89.50	709	309	336	0.148			
		11	18.31	0.926	0.819	42.85	63.25	714	313	332	0.142			
		12	18.29	1.232	1.090	56.51	83.32	711	310	334	0.145			
		13	18.29	1.321	1.171	60.63	89.53	718	312	341	0.149			
		14	18.31	1.168	1.037	53.64	79.41	716	312	340	0.149			
		15	18.31	0.982	0.872	45.26	67.12	714	311	339	0.149			
		16	18.29	0.897	0.796	41.42	61.12	709	311	330	0.142			
mean								**709**	**309**	**335**	**0.147**	**34**	**7.7**	**512**
±								**8**	**3**	**6**	**0.004**			

Table 1. **C**ontinued.

Product	%	#	d	t	m	f_T	f_F	E	G	B	ν	H	K_{IC}	σ
			mm	mm	g	kHz	kHz	GPa	GPa	GPa		GPa	MPa m$^{1/2}$	MPa
BZN 6000	90	1	23.51	0.432	0.769	11.46	17.26	769	329	386	0.168			
		2	25.43	0.635	1.361	13.80	20.65	727	313	356	0.160			
		3	25.45	0.648	1.376	14.30	21.50	748	321	373	0.166			
		4	34.00	0.510	1.923	6.21	9.36	719	307	364	0.171			
		5	34.08	0.522	1.967	6.32	9.46	705	304	345	0.160			
		6	34.08	0.778	2.825	9.98	14.80	754	328	358	0.149			
		7	34.08	0.818	2.935	10.29	15.12	739	326	336	0.133			
Mean								**737**	**318**	**360**	**0.158**	**37**	**10.8**	**747**
±								**22**	**10**	**17**	**0.013**			
Theoretical	100							**909**	**405**	**400**	**0.121**			

Table 2. Summary of results on PCD including means and standard deviations. Tabulated are: volume % superabrasive (%), diameter (d), thickness (t), mass (m), torsional frequency (f_T), flexural frequency (f_F), Young's modulus (E), shear modulus (G), bulk modulus (B), and Poisson's ratio (ν).

Product	%	#	d	t	m	f_T	f_F	E	G	B	ν
			mm	mm	g	kHz	kHz	GPa	GPa	GPa	
PCD 1600	90		34.77	1.192	4.258	16.74	23.92	**916**	**417**	**380**	**0.098**
PCD 1500	94	1	34.82	1.001	3.669	14.05	20.15	942	427	396	0.104
		2	34.77	1.030	3.779	14.69	21.22	976	438	420	0.113
		3	34.82	0.984	3.670	14.04	20.16	991	448	418	0.105
Mean								**969**	**438**	**411**	**0.107**
±								**25**	**11**	**13**	**0.005**
Theoretical	100							**1143**	**535**	**442**	**0.069**

where $C' = (c_{11} - c_{12})/2$. Inserting the elastic stiffness constants for cBN (c_{11} = 820 GPa; c_{12} = 190 GPa; c_{44} = 480 GPa [19]) into the above equations and numerically solving Eq. (7), we obtain B = 400 GPa, $\overline{G}$ = 405 GPa, $\overline{E}$ = 909 GPa, and $\overline{\nu}$ = 0.121. These values are included in Table 1 for comparison with the properties of sintered cBN.

V. DISCUSSION

The absolute values of the elastic properties and their trends with superabrasive content are consistent with previous observations based on other methods. The Young's modulus and shear modulus of PCBN and PCD increase with the volume fraction of superabrasive, as reported previously for PCD [18]. However, the Poisson's ratio does not display a clear trend, and is probably affected by grain size and binder phase. A more quantitative analysis of the trends in the present results is unwarranted, as the elastic properties also depend on grain size [1,18], which are not the same among the commercial products examined. The Vicker's hardness also increases with superabrasive content, consistent with previous reports [18,24]. The values of the Young's modulus for PCD are similar to measurements obtained by 3-point bend (905 GPa) [17] and from the speed of sound of longitudinal and transverse waves (749-953 GPa) [18]. In the case of PCBN, a measurement obtained using a microindentation technique [25] yielded a value, 795 GPa, that is within the range observed in this study.

The elastic constants for each type of PCBN are in reasonably good agreement with values derived from longitudinal and shear-wave speed-of-sound measurements. Using similarly-prepared free-standing specimens, Gilmore [26] obtained values for $\overline{E}$ and $\overline{\nu}$ of 681 GPa, 0.164; 727 GPa, 0.137; and 801, 0.144 for individual specimens of BZN 8100, BZN 7000, and BZN 6000, respectively.

The Young's modulus of 100%-dense PCBN, derived from speed-of-sound measurements, has been reported as 890 GPa [27], in rather good agreement with our theoretical estimate of 909 GPa. Overall, the theoretical elastic constants for 100% PCBN are not as accurate as those for diamond, due to the larger uncertainties in the single-crystal elastic constants in the former case [23]. The bulk modulus calculated from these constants, 400 ± 20 GPa [23], is somewhat larger than values derived from the pressure dependence of the lattice constant, 369 ± 14 GPa [28] and 385 ± 3 GPa [29]. The uncertainties in the calculated values of $\overline{E}$ and $\overline{G}$ for pure PCBN are therefore estimated to be in the range of 20 - 40 GPa. Nonetheless, the excellent agreement with experiment is encouraging, and it is clear that the experimental modulus values approach the theoretical values as the volume fraction of superabrasive is increased.

In contrast to the correlation of the Young's modulus, shear modulus, and hardness with superabrasive fraction, the fracture toughness and transverse rupture strength clearly are strongly affected by grain size and binder phase. The roles of grain size and binder phase in arresting crack propagation have been documented previously [18,24,30,31]. A more detailed analysis is beyond the scope of this report.

The accuracy of the elastic constants of PCD and PCBN evaluated by the dynamic resonance method is limited mainly by the dimensional measurements, particularly the thickness. The weight and frequencies can easily be measured to 0.1% accuracy or better, and the formulas are believed to be accurate to approximately 1% or better. The inferred modulus values are approximately proportional to t^{-3} ($\rho \propto$ weight/t) and therefore an accurate (and uniform) thickness is particularly important. If the thickness of the disks is not sufficiently uniform, the vibrational mode structure becomes complex, precluding measurement of the elastic properties.

In conclusion, we have demonstrated that the impulse dynamic resonance method as applied to free-standing PCBN and PCD is simple, fast, and yields accurate values for the Young's modulus, shear modulus, and the Poisson's ratio, and is suitable for routine characterization of superabrasive materials. However, the technique works best with free-standing disks, since the interface with the tungsten carbide substrate in compacts fabricated by infiltration is typically not accurately planar, and separation of the carbide and superabrasive properties might be difficult. As-finished specimens of free-standing products such as BZN 7000 could be analyzed without thinning by employing a microphone and preamplifier with a higher frequency range.

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