ELASTIC PROPERTIES OF CVD DIAMOND VIA DYNAMIC RESONANCE MEASUREMENTS

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ABSTRACT

Control of the mechanical properties of CVD diamond is essential to achieve optimal performance in various applications. While several methods have been applied to the measurement of the Young's modulus of thick-film CVD diamond, in general these methods are not suitable for diamond characterization on a production scale. In addition, many of these methods cannot determine the shear modulus (or Poisson's ratio), which is necessary for a complete description of the elastic properties. We have developed a simple dynamic resonance method for determining both the Young's and shear modulus of free-standing CVD diamond in the shape of rectangular plates or round disks. The specimen is supported along nodal lines of flexural or torsional modes. Oscillations are induced by impact from a falling ceramic bead, are sensed by a microphone, and the resonant frequencies are determined by a signal analyzer. The Young's and shear modulus are calculated from the frequencies of the fundamental flexural and torsional modes, respectively, using quasi-analytic formulas. CVD diamond grown by several methods routinely achieves Young's and shear modulus values above 1000 GPa and 500 GPa, respectively, in good accord with theoretical values for pure polycrystalline diamond.

INTRODUCTION

The extreme properties of diamond, together with the capability for growth of thick, high-quality polycrystalline diamond films by chemical vapor deposition (CVD) developed over the past decade-and-a-half, has motivated the development of a number of CVD diamond products. Many of these products, including heat sinks, optical windows, machine tool inserts, and wire dies, involve thick, free-standing polycrystalline diamond plates rather than a thin film of diamond on a substrate. Achievement of optimal performance in commercial products, however, obviously hinges on the material properties of the diamond and their reproducibility. In the case of heat sinks for microelectronic thermal management applications, for example, it was necessary to develop suitable methods for routine determination of the thermal conductivity.[1]

The mechanical properties of CVD diamond, including the elastic constants and fracture strength, are critical for many other applications. For determination of the elastic modulus of CVD diamond, the bulge test method is the most widely applied technique.[2-5] In this technique the displacement of the center of a disk is measured as function of the differential pressure applied across it. The bulge test method has the advantages of requiring only inexpensive equipment and permitting fracture strength measurements in the same apparatus. However, it is arguably too tedious for routine use in production. Only the biaxial modulus can determined by this method, requiring an assumed value for the Poisson's ratio. In addition, the boundary conditions of the disk (fixed versus free) must be determined, either directly or by empirical correction,[4-6] which complicates extraction of the modulus from the pressure-displacement data.

Several other methods have been applied to the determination of elastic constants for CVD diamond, but arguably are also unsuitable for routine measurements. Nanoindentation measurements can determine both the Young's modulus and hardness,[7-9] but require expensive, specialized equipment and are rather time-consuming. Speed-of-sound measurements[10,11] are significantly simpler to apply and also have the capability for determining both the Young's and shear modulus, but the cost of the equipment and time required to characterize each specimen are still unattractive.

In this paper we report the first application of another technique, dynamic resonance, to the determination of the elastic constants of free-standing CVD diamond. This method requires only inexpensive equipment and is very fast, yet can accurately determine both the Young's and shear modulus.

EXPERIMENTAL: IMPULSE DYNAMIC RESONANCE METHOD

The dynamic resonance method is a well-established technique for the determination of elastic constants.[12,13] As developed originally the technique involved driving the specimen with a variable-frequency external oscillator and measuring the response as a function of frequency. With the ready availability of digital signal processing techniques, an impulse technique, where oscillations are induced in the part by tapping and the transient "ringing" signal is detected by a microphone, digitized, and Fourier-analyzed, is much simpler experimentally and yields the same information. Although only approximate equations relating the measured resonant frequencies to the elastic constants are available, the accuracy of these expressions has been demonstrated to be approximately 1% or better for a wide range of specimen geometries and elastic properties, more than adequate for our purposes.

In the case of rectangular bars or plates, the Young's modulus may be determined from the frequency of the one-dimensional flexural mode.[12,13] This mode has nodes at positions $0.224l$ and $0.776l$, where l is the sample length, and can be excited by supporting the specimen along the nodes and tapping in the middle, as illustrated in Fig. 1(a). The shear modulus may be determined from the frequency of the torsional mode. This vibration has nodal lines bisecting the face in each direction, as illustrated in *Fig. 1(b)*, and can be excited by supporting the specimen along the intersecting nodes and tapping near one corner. In the case of round disks, the torsional mode is equivalent to that shown in Fig. 1(b), although the position of the nodes and antinodes is

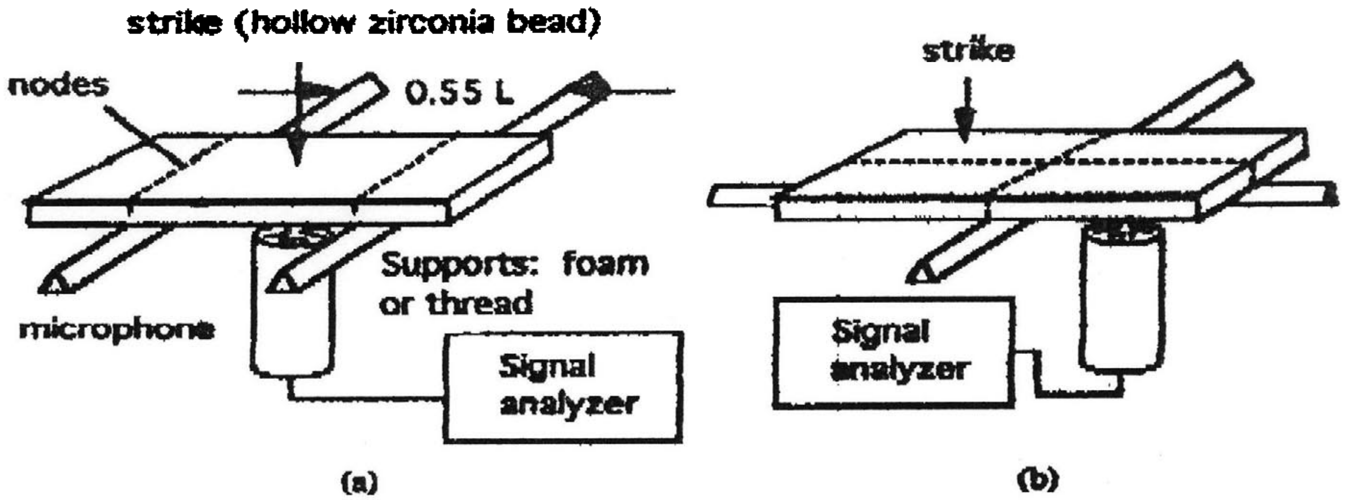

FIG. 1. Schematic illustration of ringing measurements on the (a) flexural mode or (b) torsional mode of rectangular plates. For disks the torsional mode is equivalent to (b), whereas the flexural vibration is a biaxial drumhead mode with a nodal circle at 0.681 of the diameter of the part.

determined by the positions of the supports and tapping rather than by the geometry. The flexural vibration of a round disk is a biaxial drumhead mode, however, with a nodal circle of diameter $0.681d$, where d is the diameter of the disk.[14,15]

We have analyzed free-standing rectangular plates and disks of GE CVD diamond produced by two different growth techniques by the impulse dynamic resonance method. 25-mm square plates were laser cut on one side to produce rectangular plates, and 10-mm diameter disks were laser-cut from larger wafers. The specimens were then supported in a fixture on either thin foam strips or on stretched cotton thread along the nodal lines for flexural or torsional vibrations, as appropriate (see Fig. 1). Oscillations were initiated by dropping a hollow zirconia bead from a height of 6-12 in. through a tube onto the center or one corner of the specimen. The ringing sound was detected by a microphone and preamplifier (Bruel & Kjaer, models 4165 and 2639, respectively), and the resonant frequencies were determined using a digital oscilloscope with Fast Fourier Transform capability.

Placement of the specimen on the fixture, tapping, and acquisition of several ringing measurements requires less than one minute per sample. For the initial experiments we performed, described here, the most time-consuming part of the operation was manual extraction of the resonant frequencies from the digital oscilloscope. For routine measurement in production, however, we use a commercial instrument[16] that performs the signal analysis automatically and reports the resonant frequency on a digital readout.

The approximate expressions used to extract the Young's and shear modulus from the measured flexural and torsional frequencies, f_F and f_T, respectively, and the weights and dimensions of the parts are summarized below. In these equations the length and width (transverse to flexure) of the rectangular plates are denoted l and w, respectively, d is the diameter of the disks, t is the thickness of either type of specimen, and ρ is the density.

The Young's modulus of the rectangular plates was determined from[12,13]

$$E = 0.9464\rho(l^2 f_F / t)^2 T_1 \qquad (1)$$

$$T_1 = 1 + [1 + 0.0752\nu + 0.8109\nu^2](t/l)^2 - 0.868(t/l)^4 - \frac{8.340[1 + 0.2023\nu + 2.173\nu^2](t/l)^4}{1 + 6.338[1 + 0.1408\nu + 1.536\nu^2](t/l)^2} \qquad (2)$$

These equations are due to an approximate solution by Pickett[17] and have been shown to be accurate (probably to better than 1%) by Spinner et al.[18] The correction term T_1 contains the Poisson's ratio ν and so in principle requires iterative solution along with the determination of the shear modulus. However, for the aspect ratio ($t/l \ll 1$) of the CVD diamond specimens examined here T_1 is nearly equal to one and insensitive to the assumed value of ν, and iteration of the solution is unnecessary.

The shear modulus of the rectangular plates was determined from the equation[12,13]

$$G = 4\rho l^2 f_T^2 R \qquad (3)$$

where, for the shape factor R we have used the equation recommended by Spinner and Tefft:[13]

$$R = \left[\frac{1 + \left(\frac{w}{t}\right)^2}{4 - 2.521\left(\frac{t}{w}\right)\left(1 - \frac{1.991}{e^{\pi w/t} + 1}\right)}\right]\left[1 + 0.00851\left(\frac{w}{l}\right)^2\right] - 0.060\left(\frac{w}{l}\right)^{3/2}\left(\frac{w}{t} - 1\right)^2 \qquad (4)$$

For relatively thin plates, such as employed here,

$$R \approx (w/t)^2/4$$

The Poisson's ratio then follows from the usual relation

$$\nu = E/(2G)-1 \tag{5}$$

With disks the flexural and torsional frequencies are each strongly influenced by both the Young's and shear modulus. Conveniently, the Poisson's ratio can be derived directly from the ratio of the two frequencies.[14,15] In general ν also depends on the t/d ratio, however, for relatively thin disks ($t/d \leq 0.05$) the latter dependence is negligible. In this case a quadratic fit to tabulated values[14,15] gives, to an excellent approximation,

$$\nu = -3.3382+3.834(f_F/f_T)-(f_F/f_T)^2 \tag{6}$$

Given the determined value of ν, the Young's modulus is determined from

$$E = 14.8044\rho(1-\nu^2)(d^2/t)^2[(f_F/K_F)^2+(f_T/K_T)^2] \tag{7}$$

The correction factors K_F and K_T depend weakly on ν and the t/d ratio and are tabulated.[14,15] For the parts analyzed here K_F and K_T are approximately equal to 8.4 and 5.9, respectively. The value of the shear modulus then follows from

$$G = E/[2(1+\nu)] \tag{8}$$

RESULTS AND DISCUSSION

The flexural and torsional frequencies, f_F and f_T, respectively, measured on representative samples are summarized in Table 1, along with the weights and dimensions of the samples. The rectangular bars were lapped on both faces and polished on one; the first disk was polished on both faces, and the second was as-grown (after removal from the substrate). These samples are all of high quality, as indicated by thermal conductivities between 9 and 20 W/cm-K.

TABLE 1. Summary of dimensions, weights, resonant frequencies, elastic moduli, and Poisson's ratio of free-standing CVD diamond plates in rectangular or circular form.

Rectangular bars:

CVDD Lot #	l (mm)	w (mm)	t (mm)	weight (g)	f_F (kHz)	f_T (kHz)	E (GPa)	G (GPa)	ν
B965	26.19	10.11	0.316	0.291	8.61	15.01	1145	530	0.08
B966	25.68	12.52	0.302	0.339	8.76	12.11	1212	547	0.11
B986	25.68	17.37	0.398	0.613	10.76	11.00	1037	467	0.11
B988	25.68	9.06	0.312	0.248	8.24	15.78	980	461	0.06
						mean:	1093	501	0.09

Disks:

CVDD Lot #	d (mm)	t (mm)	weight (g)	f_F (kHz)	f_T (kHz)	E (GPa)	G (GPa)	ν
C535	10.24	0.260	0.075	69.81	49.41	1161	536	0.08
C603	10.08	0.365	0.102	100.41	71.00	1154	532	0.08

The accuracy of the elastic constants of CVD diamond determined by the dynamic resonance method is typically limited mainly by the accuracy of the dimensional measurements, particularly the thickness. The weight and frequencies can easily be determined 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} (ρ = weight/t) and therefore an accurate thickness is particularly important. For unpolished plates an approximate average thickness can be determined by assuming the ideal density of 3.515 g cm^{-3} for diamond.

The measured values of the Young's and shear modulus and Poisson's ratio are in good agreement with theoretical values for pure polycrystalline diamond. Two groups have shown independently that the elastic constants for single-crystal natural diamond, when rotationally averaged as appropriate for polycrystalline diamond with no preferred orientation, yield a Young's modulus of 1143 GPa, a shear modulus of 534 GPa, and a Poisson's ratio of 0.07.[4,19] Thick-film, free-standing CVD diamond normally exhibits a significant fiber texture, and all of the samples examined in this study have a preferred (110) orientation. The effective elastic constants–all experimental measurements of the elastic constants of CVD diamond of which the authors are aware assume isotropic elastic behavior–depend on the texture. The Young's modulus exhibits only modest orientational dependence, and both Klein and Cardinale[4] and Werner *et al.*[19] calculate a value of 1151 GPa for (110)-oriented diamond. These authors differ somewhat on the effective shear modulus for (110)-textured diamond, obtaining 536 and 524 GPa, respectively.[4,19] These values correspond to Poisson's ratios of 0.07 and 0.10, respectively. Not all the grains in textured diamond have a fiber axis precisely aligned along the sample normal, of course, and a more complex rotational averaging procedure is probably required for precise specification of the effective elastic constants of textured CVD diamond. In any case, we have not investigated the relationship between the fiber texture of CVD diamond and the elastic constants.

In conclusion, we have demonstrated that the impulse dynamic resonance method as applied to free-standing CVD diamond is simple, fast, and apparently yields accurate values for the Young's and shear modulus and the Poisson's ratio, and is therefore suitable for routine characterization of commercial material. In addition, we have shown that GE CVD diamond produced by two different growth methods routinely achieves values of the Young's and shear modulus in good agreement with the corresponding values for pure polycrystalline diamond.

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IDENT.	Length	Height	Width	Mass	Dens.	POISSON		R[FLEX]	R[LONG]	R[TORS]	E[FLEX]	E[LONG]	G[TORS]
	mm	mm	mm	g	g/cm3	EF,G	EL,G						
B965	26.19	0.32	10.11	0.29	3.478	0.08		8.610 KHz		15.01 KHz	**********		532.7 GPa
~~B966~~	~~25.68~~	~~0.30~~	~~12.52~~	~~0.34~~	~~3.491~~	~~0.11~~		~~8.760 KHz~~		~~12.11 KHz~~	**********		545.7 GPa
B986	25.68	0.40	17.37	0.61	3.453	0.11		10.76 KHz		11.00 KHz	**********		467.9 GPa
B988	25.68	0.31	9.06	0.25	3.416	0.06		8.240 KHz		15.78 KHz	981.8 GPa		462.6 GPa

IDENT.	Diam.	Thick.	Mass	Dens.	POIS.	R[FLEX]	R[TORS]	E[FLEX]	G[TORS]
	mm	mm	g	g/cm3					
1	10.24	0,26	0.08	3.503	0.08	29	40	**********	533.4 GPa
2	10.08	0.37	0.10	3.502	0.08	20	28	**********	529.2 GPa
1 density	10.24	0.26	0,08	3.515	0.08	29	40	**********	535.3 GPa
2 density	10.08	0.37	0.10	3.515	0.08	20	28	**********	531.2 GPa