Elasticity, strength, and toughness of single crystal silicon carbide, ultrananocrystalline diamond, and hydrogen-free tetrahedral amorphous carbon

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In this work, the authors report the mechanical properties of three emerging materials in thin film form: single crystal silicon carbide (3*C*-SiC), ultrananocrystalline diamond, and hydrogen-free tetrahedral amorphous carbon. The materials are being employed in micro- and nanoelectromechanical systems. Several reports addressed some of the mechanical properties of these materials but they are based in different experimental approaches. Here, they use a single testing method, the membrane deflection experiment, to compare these materials' Young's moduli, characteristic strengths, fracture toughnesses, and theoretical strengths. Furthermore, they analyze the applicability of Weibull theory [Proc. Royal Swedish Inst. Eng. Res. **153**, 1 (1939); ASME J. Appl. Mech. **18**, 293 (1951)] in the prediction of these materials' failure and document the volume-or surface-initiated failure modes by fractographic analysis. The findings are of particular relevance to the selection of micro- and nanoelectromechanical systems materials for various applications of interest. © *2006 American Institute of Physics*. [DOI: 10.1063/1.2336220]

Increasing demands in performance from micro- and nanoelectromechanical systems (MEMS/NEMS) make accurate characterization of material mechanical properties, at the relevant size scales, a high priority task for the mechanics community. Advances in mechanical testing have made possible the characterization of Young's modulus, Poisson's ratio, fracture toughness, and strength for a variety of thin film materials.^{1–6} However, the results vary for different testing techniques. In this work, we report a comparison of the properties of three MEMS/NEMS materials, 3C-SiC, ultrananocrystalline diamond (UNCD), and tetrahedral amorphone carbon (ta-C), based on a single experimental technique, the membrane deflection experiment,⁷ which makes this comparison highly quantitative and meaningful.

In the membrane deflection experiment (MDE), a nanoindenter is used to deform and stress freestanding fixed-fixed specimens until failure. The result is direct tension, with load and deformation measured independently.^{7,8} In the present investigation, specimens were microfabricated from the three materials under previously developed protocols.⁹ The back sides of the wafers were etched by means of reactive ion etching to obtain freestanding membranes bridging open windows in the silicon wafer. Single crystal 3*C*-SiC from Case Western Reserve University¹⁰ was deposited in an atmospheric-pressure chemical vapor deposition reactor, ta-C from Sandia National Laboratories¹¹ (SNL) was deposited at room temperature via pulsed laser deposition and annealed at 600-650 °C to relieve the high residual stress of the as-

deposited films, and UNCD from Argonne National Laboratory¹² was deposited in a microwave plasma-enhanced chemical vapor deposition reactor at 800 °C.

Specimens with two gauge dimensions, A (20 μ m wide and 200 μ m long) and B (5 μ m wide and 100 μ m long), were stressed until failure under the same conditions using the MDE technique. Identified elastic moduli and strengths for each material are given in Table I (rows 4–6). The strengths are reported at 63% probability of failure.

In interpreting the fracture strength we employ Weibull statistical theory.¹³ Accordingly, the probability of failure P_f can be written as

$$P_{f} = 1 - \exp\left[-\left(\frac{\sigma_{\max}}{\sigma_{0 V}}\right)^{m} V_{e}\right]$$

or $P_{f} = 1 - \exp\left[-\left(\frac{\sigma_{\max}}{\sigma_{0 A}}\right)^{m} A_{e}\right],$ (1)

where *m* is the Weibull modulus and $\sigma_0 V$ or $\sigma_0 A$ are the characteristic strengths of the material in the volume- or surface-initiated failure modes, respectively. V_e and A_e are the effective volume and area of the samples subjected to uniform stress.^{6,8} The area can be the total sample surface area or sidewall area. However, whether the probability of failure scales with volume or area must be determined experimentally for each type of sample. We can determine the scaling parameters by two approaches: (1) test samples with various dimensions, fit the tested values using V_e and A_e (volume, total surface area, or sidewall surface area), and see which fit is best, and (2) examine the fracture surfaces of the specimens to find the failure initiation location.¹⁴

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	3 <i>C</i> -SiC		UNCD		ta-C	
Material	Size A	Size B	Size A	Size B	Size A	Size B
No. of tests	30	30	30	30	30	30
Thickness (nm)	855±53	527 ± 29	1050 ± 77	515±21	896±33	501 ± 12
Hardness (GPa)			98		70-80	
Young's modulus (GPa)	422±18	435±15	960±25	955±21	801±22	795 ± 34
Fracture strength (MPa)	2090±519	2680 ± 556	3990 ± 428	5080 ± 555	4680 ± 540	5260 ± 459
Weibull modulus	5.4 ± 0.27		11.6 ± 0.58		12.0 ± 0.60	
$\sigma_{0~\mathrm{V}}$ or $\sigma_{0~\mathrm{A}}$	6780±339 MPa μm ^{2/m}		$8560 \pm 428 \text{ MPa} \ \mu \text{m}^{3/m}$		8020±401 MPa μm ^{2/m}	
Scaling parameter	Sidewall		Volume		Sidewall	

TABLE I. Measured elastic modulus and fracture strengths for the three tested materials.

If the Weibull statistics adequately describes the behavior of the material, $\sigma_{0 V}$ or $\sigma_{0 A}$ are material and fabrication parameters. In other words, they will be the same for various sample dimensions. In Fig. 1 we plot the values of $\sigma_{0 V}$, $\sigma_{0 A}$, and $\sigma_{0A,S}$ (corresponding to sidewall area) as calculated for two sample geometries (A and B) in the case of the three tested materials. The results show that the normalized characteristic strengths are closest for the two sample sizes when volume is used in the case of UNCD, and sidewall area is used in the cases of 3C-SiC and ta-C. Thus, from the fracture statistics, the scaling parameter is most likely volume in the case of UNCD and sidewall area for both 3C-SiC and ta-C. As will be shown later, the fractographic analysis¹⁵ supports this conclusion.

For a known Weibull modulus, characteristic strength, and scaling parameter (Table I, rows 7–9), different sets of experimental data (P_f , σ_{max}) can be transformed into one single data set for each material. This was performed for the three materials in Fig. 2, with points corresponding to the two geometries labeled in the legend, to track their progression. From the plots, it is clear that the points line up quite well on one curve and, as a result, the Weibull statistics [Eq. (1)] with the chosen scaling parameters describe the experimental data quite well.

To confirm that the failures are volume or surface initiated as predicted by the statistics, the fracture surfaces of the three tested materials were examined by high resolution electron microscopy. Figure 3(a) shows a typical fracture surface corresponding to an UNCD sample. The features observed in Fig. 3(a) suggest that failure most likely initiates from interior (volume) defects introduced during the film deposition process, although the origin of the failure cannot be determined precisely. Figure 3(b) is a plain view transmission electron microscopy (TEM) micrograph of the UNCD sample. The high resolution image reveals the existence of amorphous carbon among well-defined, normal-sized diamond grains. The inset shows the ring pattern obtained by a fast Fourier transform. The size of the amorphous region is about $4 \times 6 \text{ nm}^2$, which is of the same order as the grains.



FIG. 1. The estimated Weibull parameters based on volume ($\sigma_{0 V}$), total surface area ($\sigma_{0 A}$), and sidewall area ($\sigma_{0 A,S}$), normalized to the values

corresponding to sample B

Such amorphous regions were observed in several parts of the sample and constitute potential sources of volumeinitiated mechanical failure. More details of the fractographic analysis and TEM study can be found in Ref. 14.

Figure 4(a) is a typical fracture surface of ta-C. As expected from the material's amorphous structure, the fracture surface is smoother than that of UNCD and similar to that observed in glasses, which exhibit more or less circular fracture *mirrors*. The center of the circular mirrors is very smooth and it is commonly attributed to the origin of the fracture.¹⁶ This feature appears at the edge of the left sidewall and is perpendicular to the maximum tensile direction. Thus, the image confirms that sidewall roughness may act as a stress concentrator, and therefore initiate fracture. The fracture surface of single crystal 3C-SiC is similar to that of ta-C [Fig. 4(b)] The surface is very smooth and a mirror region can be clearly observed on the sidewall. Hence, the failure origins for ta-C and 3C-SiC are found to be on the sidewalls, which is consistent with the scaling parameters identified based solely on statistics.

The fracture toughness K_{IC} of the three materials under investigation was determined using the membrane deflection fracture toughness technique developed by Espinosa and Peng.¹⁷ Sharp cracks were achieved by placing a Vickers indent (at maximum load of 200 g) into the silicon and emanating corner cracks into the thin film specimen prior to its release (for details see Espinosa and Peng¹⁷). Alternatively, blunt notches with finite tip radii were fabricated using focused ion beam micromilling.¹⁷ The equivalent stressintensity factor K'_{IC} from a blunt notch was computed by⁸

$$K_{IC}' = \sqrt{1 + \frac{\rho}{2d_0}} K_{IC},$$
 (2)

in which ρ is the root notch radius and the finite length d_0 is a characteristic dimension given by⁸



FIG. 2. Weibull plots showing fracture strength transformed to unit size in uniaxial tension. 3C-SiC and ta-C are transformed to unit sidewall area and UNCD is transformed to unit volume.

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FIG. 3. (a) SEM micrograph of the overview of the fracture surface of an UNCD specimen and (b) HRTEM plain view of an UNCD sample, showing amorphous carbon in front of grains, with one grain presenting twins. The inset shows the fast Fourier transform (FFT) of an area within the amorphous region.

$$d_0 = \frac{2}{\pi} \frac{K_{IC}^2}{\sigma_u^2}.$$
 (3)

In this equation, σ_u is the ideal or theoretical strength of the material associated with the characteristic length d_0 (Novozhilov fracture criterion). Note that d_0 can be obtained by matching two different experimental results performed on notches with different root radii ρ , as suggested by Eq. (2). In this work we matched the experimental results corresponding to a value of ρ =200 nm and used the toughness identified from specimens containing atomically sharp cracks.

The measured fracture toughness, equivalent stressintensity factor of a blunt notch, characteristic length, and computed ideal strength for 3*C*-SiC, UNCD, and ta-C are given in Table II.

In summary, we studied the applicability of Weibull theory, which can help us predict micro- and nanosystem reliability. Specifically, it was used to find the material's failure strength for a certain geometry, although the theory is applicable to complex geometry through proper three dimensional stress analysis. The fracture toughness (the material's resistance to crack propagation) and the theoretical strength on the scale of a defect-free characteristic length were also identified. Therefore, the investigation is particularly relevant to the problem of selecting a MEMS/NEMS material for a particular application. For instance, the results show that of the two forms of diamond, ta-C has the highest fracture toughness, 6.2 MPa $m^{1/2}$, and theoretical strength, 25.4 GPa, despite its amorphous nanostructure and defect-free characteristic length similar to UNCD. However, ta-C cannot be easily doped and requires an annealing step which must be



FIG. 4. (a) Fracture surface of a ta-C specimen. (b) Fracture surface of a single crystal 3C-SiC specimen. The arrow points to the mirror region where the failure originated.

TABLE II. Measured fracture toughness, equivalent stress-intensity factors, characteristic defect-free length, and theoretical strengths.

Material	K_{IC} (MPa \sqrt{m})	K'_{IC} (MPa \sqrt{m})	d_0 (nm)	σ_u (GPa)
3C-SiC	3.2	$5.3(\rho = 200 \text{ nm})$	58	10.6
UNCD	4.5	$8.7(\rho = 200 \text{ nm})$	37	18.6
ta-C	6.2	$11.8(\rho = 200 \text{ nm})$	38	25.4

incorporated in the microfabrication process. By contrast, UNCD can be doped in order to make it conductive. Furthermore, the as-grown films exhibit very low residual stresses. Hence, in applications where wear resistance is paramount, diamond in either crystalline or amorphous form is the material of choice.¹⁸ In applications where high temperatures are expected, 3C-SiC is best because of its high temperature stability.

The investigation also points at direction for improving the materials. For instance, the results suggest that weak or defective interfaces, likely between clusters of grains, are present in UNCD. Clusters of grains and corresponding interfaces are the result of the existence of multiple growth sites, arising from ultrasonically seeded diamond nanoparticles, during film deposition.⁸ It remains to be demonstrated that advances in seeding and plasma characteristics can lead to UNCD strengths closer to the theoretical strength of single crystal diamond.

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