

Nanomechanical resonant structures in nanocrystalline diamond

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We report the fabrication and the operation of nanomechanical resonant structures in nanocrystalline diamond. For this purpose, continuous diamond films as thin as 80 nm were grown using microwave plasma enhanced chemical vapor deposition. The lateral dimensions of the fabricated structures were as small as 50 nm and the measured mechanical resonant frequencies were up to 640 MHz. The mechanical quality factors were in the range of 2500–3000 at room temperature. The elastic properties of these films obtained via the resonant measurements indicate a Young's modulus close to that of single-crystal diamond. © 2002 American Institute of Physics.
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Nanomechanical and nanoelectromechanical resonant structures (NEMS) are being considered for sensing applications, similar to those demonstrated in micromechanical structures (MEMS)^{1,2} with a hope of gaining even higher sensitivity via the high resonant frequencies achievable in NEMS. One of the motivations for studying NEMS fabricated in different materials is to optimize relevant properties such as hardness, compatibility with operating environment, low dissipation, or the ease of the processing and integration. Another motivation is to study the properties of those materials using NEMS. To date, NEMS have been fabricated and reported, in Si,³ Si_xN_y,⁴ SiC,⁵ AlN,⁶ and spin-on glass.⁷ In this work, we report on nanomechanical resonant structures fabricated in nanocrystalline diamond. For technological purposes, diamond may be one of the most desirable materials for many NEMS applications because it is chemically very resistant, has a high hardness and thermal conductivity. Doping during deposition also allows for a wide range of electrical conductivities.⁸ In particular, the high Young's modulus of diamond E (~ 1000 GPa) as well as relatively low mass density, ρ (3500 kg m^{-3}),⁹ result in a high sound velocity, $\sqrt{E/\rho}$. As will be shown next, a high sound velocity allows us to retain the size of a resonant structure and still obtain a high resonant frequency. To date, MEMS with dimensions an order of magnitude or 2 larger than those reported here have been fabricated in polycrystalline diamond,¹⁰ nanocrystalline diamond,¹¹ as well as in amorphous carbon.¹²

The nanocrystalline diamond films used for fabricating the structures described here have a columnar growth structure with grains that range in size from 5–15 nm on the nucleation side to $\sim 10\%$ the thickness of the film on the growth surface. The deposition is done by microwave plasma enhanced chemical vapor deposition (CVD) using CH₄ and H₂.^{13,14} The substrate is pretreated by exposing it to the deposition conditions in the microwave chamber for 20 min.

Then, the substrate is removed from the chamber and is it treated in an ultrasonic bath of nanodiamond powder dispersed in ethanol, rinsed, and dried. Finally, the nanocrystalline diamond film is grown at 750 °C using gas flows of 900 sccm hydrogen and 3 sccm methane, 800 W microwave power while the thickness is monitored *in situ* using laser interferometry at 677 nm. The diamond films thus obtained have measured density of 3500 kg m^{-3} ,¹⁴ the same as the density of single-crystal diamond.⁹ The Raman spectra of these films using 488 nm excitation shows a well defined 1332 cm^{-1} diamond zone center phonon peak and well as weaker broadband at 1500 cm^{-1} . The deposition process described herein has been used to deposit diamond films on silicon and silicon dioxide. For the purposes of this study, the films were deposited on $1 \mu\text{m}$ of silicon dioxide obtained by thermal oxidation of a single-crystal silicon wafer.

The structures seen in the micrographs in Figs. 1, 2, and 3 are fabricated using electron-beam lithography. The pattern is transferred into the diamond films using a CF₄/O₂ reactive ion etch¹¹ with the diamond film protected by a metal mask. After the removal of the mask, the structures are released in buffered hydrofluoric acid which removes the underlying oxide layer. The fabrication sequence is very similar to the one we use for silicon¹⁵ and silicon nitride⁴ nanomechanical structures. Most of the structures did not need the critical drying point step¹⁶ for their release. The resulting structures have lateral dimensions as small as 50 nm and as large as $6 \mu\text{m}$. Figure 1 shows details of a part of the structure, 200 nm wide and about 200 nm thick. In the same picture, we can see the columnar grain structure and the surface roughness of these films.

Figures 1 and 2 show mechanical structures that were actuated using a piezoelement.¹⁷ All of the measurements were done in a vacuum of about 10^{-6} Torr. The motion is detected interferometrically^{15,18} with the structure efficiently modulating the light at 633 nm. The measured resonant frequencies of the doubly clamped paddles (Fig. 1) range from 6–30 MHz for their flexural (out-of-plane) mode of

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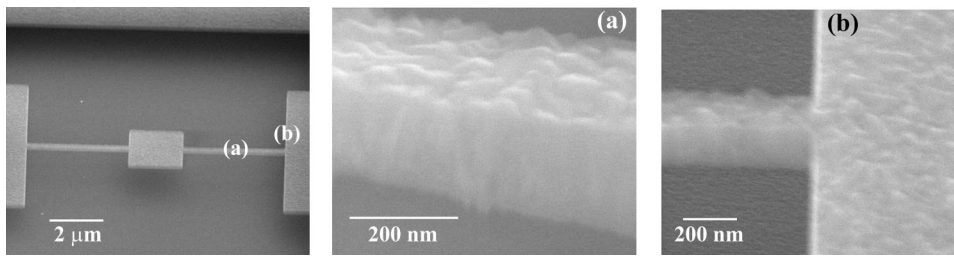


FIG. 1. A $2 \times 2 \mu\text{m}$ diamond paddle with the details of the supporting arms [in (a) and (b)].

motion.¹⁸ Their quality factors, $Q = f/\Delta f$ are 2400–3500. The measured resonant frequencies of the structures in Fig. 2 were 8–20 MHz and their quality factors were around 3000. The set of structures whose resonant frequencies we use to derive materials properties are shown in Fig. 3. They are doubly clamped beams varying in length from 8 to $1 \mu\text{m}$, in steps of $0.5 \mu\text{m}$ and resonant frequencies 34–640 MHz. The width of the fabricated beams was as small as 50 nm and as wide as $1 \mu\text{m}$ and thicknesses of about 180 nm. The beams were actuated using an applied rf voltage between the structure and the substrate.¹⁸ In order to use this electrostatic driving scheme, a wetting layer of chromium and a 35 nm layer of gold for conducting the electrical signal were evaporated on the structures and the substrate. Because the volumetric fraction of the gold layer was significant, we expect it to affect the mechanical properties of the beams.

The data in Fig. 3 show the frequency dependence on the length of the beams. The angular frequency, ω , of the first resonant mode of a doubly clamped beam is given by:¹⁹

$$\omega = \frac{4.73^2}{L^2} t \sqrt{\frac{E}{12\rho}}.$$

Here, L is the length of the beam, t its thickness, E is Young's modulus of the material, and ρ is its density. For a bilayer diamond/gold structure such as this one, ρ is replaced by $\rho_{\text{effective}}$ where

$$\rho_{\text{effective}} = V_{\text{diamond}}\rho_{\text{diamond}} + V_{\text{gold}}\rho_{\text{gold}},$$

and V_{material} is the relevant volumetric fraction. Assuming the density of gold to be $19\,300 \text{ kg m}^{-3}$,⁹ and the density of the diamond $3\,500 \text{ kg m}^{-3}$ we arrive at an effective Young's modulus of about 700 GPa. Taking into account the elastic

contribution of the gold layer, we derive the value of Young's modulus for the diamond to be 840 GPa. The value obtained from this analysis is about 20% lower than that obtained for some of the diamond films grown under the same conditions and measured using surface acoustic waves¹⁴ and is also outside the range of values of other CVD films⁸ or single-crystal diamond.⁹ Considering that the beams are released by an isotropic etch which results in an undercut to the base that may contribute to the motion, this additional released material may result in a lower-frequency relative to a rigidly clamped beam. A simple addition of the overhang length to the length of the structure does not give an accurate treatment. The overhang is however somewhat clamped and an exact solution of the mode shape is necessary as it has been demonstrated in other analyses of similar beams.⁶ One would not predict a very different value of Young's modulus in fully dense nanocrystalline films compared to the bulk Young's modulus. A polycrystalline material would have to have very large grain boundaries and very small grains (much smaller than in this material) for its elastic properties to differ drastically from the crystalline material as results from Baker *et al.*²⁰ demonstrate that a 10% difference is barely achievable.

The resonant frequency of a $2 \mu\text{m}$ long, 180 nm thick diamond beam is 640 MHz. For comparison, the frequency of a 200 nm thick silicon beam of the same length is 380 MHz.²¹ The most important factor in the selection of a material with which one can obtain the highest resonant frequency for a given size is the sound velocity of the material, given by $\sqrt{E/\rho}$. Diamond has a high sound velocity, $18\,000 \text{ m s}^{-1}$ compared to AlN (5500), Si (7500), or SiC (11 400).

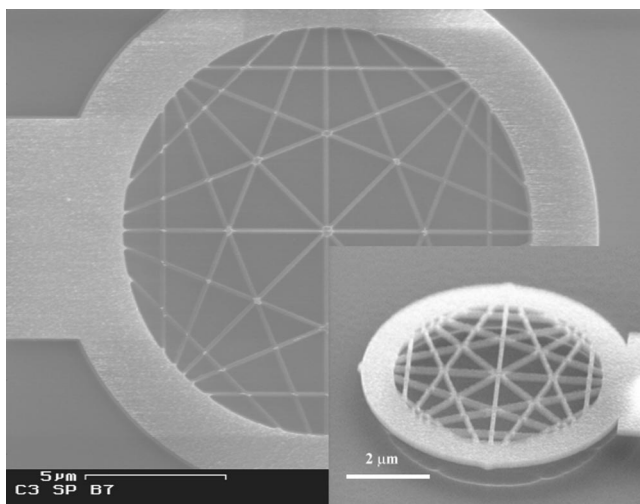


FIG. 2. An $8 \mu\text{m}$ and a $2.5 \mu\text{m}$ radius mesh membrane structure.

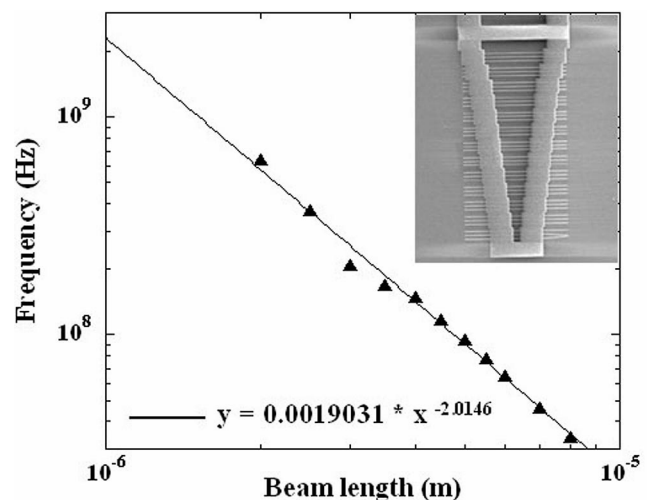


FIG. 3. Resonant frequencies of the doubly clamped diamond beams, 1–8 μm long (inset) plotted vs their length. The shortest beam measured was 2 μm long.

Another advantage of using diamond for NEMS is its relatively low losses. The lower dissipation gives a sharper resonant response and translates into higher sensitivity for applications. Considering that these films have many grains and considerable surface roughness, one would expect Snoek (grain boundary) damping and possible surface-related losses to be large (surfaces are thought to be a significant source of dissipation in this size scale).²¹ The quality factor values we obtain for the diamond structures are comparable to those of structures in silicon¹⁸ and silicon nitride⁴ of similar dimensions and at room temperature. These comparable quality factors in the aforementioned materials (a single-crystal, a polycrystalline, and an amorphous one, all chemically different) imply that at room-temperature bulk material processes may not be the limiting dissipation mechanism and thus the choice of material may not limit the width of the resonant frequency. If, for example, thermoelastic dissipation²² and/or surface losses indeed dominate room-temperature losses in NEMS, then temperature dependent studies of the diamond may give us more answers about relevant dissipation processes in this material. High-*Q* resonators at lower frequencies have already been demonstrated in MEMS structures made from films such as these¹¹ and their *Q*-factor values also compare to those of identical polysilicon structures.²³ It is likely that the *Q*-factor value for NEMS could be improved with further optimization of grain sizes and crystalline texture in these films.

In summary, we have fabricated, using standard lithographic and semiconductor processing techniques, nanocrystalline diamond structures with dimensions as small as 50 nm. The CVD diamond films used in this fabrication have been deposited on a variety of substrates, with thicknesses as low as 80 nm. The measured resonant frequencies of these devices range up to 640 MHz. The elastic properties of the CVD diamond films obtained from these measurements show elastic compliance close to that of single-crystal diamond.

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- ¹H. J. Mamin and D. Rugar, Appl. Phys. Lett. **79**, 3358 (2001).
- ²B. Ilic, D. Czaplewski, H. G. Craighead, P. Neuzil, C. Campagnolo, and C. Batt, Appl. Phys. Lett. **77**, 450 (2000).
- ³D. W. Carr and H. G. Craighead, J. Vac. Sci. Technol. B **15**, 2760 (1997); A. Erbe, R. H. Blick, A. Tilke, A. Kriele, and J. P. Kotthaus, Appl. Phys. Lett. **73**, 3751 (1998); A. N. Cleland and M. L. Roukes, *ibid.* **69**, 2653 (1996).
- ⁴L. Sekaric, D. W. Carr, S. Evoy, J. M. Parpia, and H. G. Craighead, Sens. Actuators A **101**, 215 (2002).
- ⁵Y. T. Yang, K. L. Ekinci, X. M. H. Huang, L. M. Schiavone, M. L. Roukes, C. A. Zorman, and M. Mehregany, Appl. Phys. Lett. **78**, 162 (2001).
- ⁶A. N. Cleland, M. Pophristic, and I. Ferguson, Appl. Phys. Lett. **79**, 2070 (2001).
- ⁷D. M. Tanenbaum, A. Olkhovets, and L. Sekaric, J. Vac. Sci. Technol. B **19**, 2829 (2001).
- ⁸T. A. Ralihar, W. P. Kang, H. Windischmann, A. P. Malshe, H. A. Naseem, J. L. Davidson, and W. D. Brown, Crit. Rev. Solid State Mater. Sci. **25**, 163 (2000).
- ⁹*CRC Handbook of Chemistry and Physics*, 62nd edition (CRC Press, Boca Raton, 1982).
- ¹⁰R. Ramesham, Thin Solid Films **340**, 1 (1999); J. L. Davidson, R. Ramesham, and C. Ellis, J. Electrochem. Soc. **137**, 3206 (1990).
- ¹¹J. Wang, J. E. Butler, D. S. Y. Hsu, and C. T. C. Nguyen, *Proceeding of the 15th IEEE MEMS Conference, Las Vegas, Nevada, 22–24 January 2002* (IEEE, New York, 2002), p. 657; J. Wang, J. E. Butler, D. S. Y. Hsu, and C. T.-C. Nguyen, *Proceedings Solid-State Sensor, Actuator and Microsystems Workshop, Hilton Head, S. Carolina, June 2002*, p. 65.
- ¹²J. P. Sullivan, T. A. Friedmann, and K. Hjort, MRS Bull. **26**, 309 (2001).
- ¹³S. Rotter, *Applied Diamond Conference/Frontier Carbon Technology (ADC/FCT) Joint Conference, 31 August–3 September 1999, Tsukuba, Japan*, edited by M. Yoshikawa, Y. Koga, Y. Tzeng, C.-P. Klages, and K. Miyoshi (1999), p. 25.
- ¹⁴J. Philip, P. Hess, T. Feygelson, and J. E. Butler (unpublished).
- ¹⁵D. W. Carr, L. Sekaric, and H. G. Craighead, J. Vac. Sci. Technol. B **16**, 3281 (1998).
- ¹⁶G. T. Mulhern, D. S. Soane, and R. T. Howe, *Proceedings of the Seventh International Conference on Solid-State Sensors and Actuators (Transducers 93)*, 7–10 June 1993, Yokohama, Japan (1993), p. 296.
- ¹⁷K. Y. Yasumura, T. D. Stowe, E. M. Chow, T. E. Pfafman, T. W. Kenny, B. C. Stipe, and D. Rugar, J. Microelectromech. Syst. **9**, 117 (2000).
- ¹⁸D. W. Carr, S. Evoy, L. Sekaric, A. Olkhovets, J. M. Parpia, and H. G. Craighead, J. Appl. Phys. **86**, 6072 (1999).
- ¹⁹S. Timoshenko, D. H. Young, and W. Weaver, *Vibration Problems in Engineering*, 4th ed. (Wiley, New York, 1974), pp. 425–427.
- ²⁰S. P. Baker, R. P. Vinci, and T. Arias, MRS Bull. **27**, 26 (2002).
- ²¹D. W. Carr, S. Evoy, L. Sekaric, H. G. Craighead, and J. M. Parpia, Appl. Phys. Lett. **75**, 920 (1999).
- ²²R. Lifshitz and M. L. Roukes, Phys. Rev. B **61**, 5600 (2000).
- ²³C. T. C. Nguyen and R. T. Howe, IEEE J. Solid State Circuits **34**, 440 (1999).