Diamond Materials for MEMS and NEMS Structures and Devices

J.E. Butler, T. Feygelson,* L. Sekaric,** H. Craighead,** J. Wang,*** and C.T.-C. Nguyen***

Code 6174, Naval Research Laboratory, Washington DC 20375
*GeoCenters Inc., Fort Washington, MD 20639
**Cornell Center for Materials Research, Cornell University, Ithaca, NY 14853
***Center for Integrated Wireless Microsystems (WIMS), Dept. of EECS
University of Michigan, Ann Arbor, Michigan 48109-2122 USA

ABSTRACT

Diamond materials offer great potential for many micro-electro-mechanical system (MEMS) and nano-electro-mechanical systems (NEMS) applications. Amongst the attractive technological properties of diamond materials are the high stiffness, thermal conductivity, optical transparency range, chemical stability and erosion resistance, and dopant controlled variable electrical resistivity. The nucleation and growth by microwave plasma chemical vapor deposition of conformal nanocrystalline diamond (NCD) films ranging in thickness from 100 nm to 5 microns, and the fabrication of MEMS and NEMS structures integrated with Si based substrates are presented. These nanocrystalline films have been shown to have high optical quality, high thermal conductivity and a Young's modulus nearly that of single crystal diamond. The results of various characterizations of the material quality will be given, along with examples of several MEMS and NEMS structures and devices.

Keywords: Diamond, MEMS, NEMS, Resonators.

1 INTRODUCTION

The exceptional mechanical, electrical, and optical properties of diamond make it the material of choice for many scientific and technological applications.[1] In particular, the high hardness and stiffness of diamond make it desirable for many mechanical applications. For this reason there is great interest in the preparation of thin diamond films which can be used as hard and wear-resistant coatings in a variety of applications, the preparation of diamond cantilevers for scanning force microscopy (SFM), or fabrication of high-Q and high-frequency micro-electro-mechanical resonators. While microcrystalline diamond films with thicknesses in the range of several micrometers have been successfully prepared and characterized, the interest in synthesizing diamond films with grain sizes in the nanometer scale (≤100 nm) is increasing. One reason is to reduce the intrinsic roughness of the diamond surface. However, such novel nanophase materials have grain size dependent mechanical properties that may significantly differ from those of their coarser–grained counterparts.

The changes in the mechanical properties due to reduction in grain sizes are due to the fact that a larger percentage of the atoms are expected to be in grain boundary environments with broken bonds, hydrogen termination, amorphous regions, and/or sp^2 bonded carbon.[2] In addition, the mechanical properties will be affected by other features such as flaws, strains, impurities, etc., that result from synthesis and processing. Therefore it is very important to understand the mechanical and elastic properties before designing and fabricating complex mechanical structures.

The "nanocrystalline" diamond films studied here are similar in their columnar structure to the thicker micro- or polycrystalline diamond films and plates studied previously,[3,4] but very different from the "ultra-nanocrystalline" diamond films reported by Gruen et al.[5,6] The present nanocrystalline films are grown on substrates coated with explosively prepared diamond,[7] which promotes nucleation densities from 10^{10} to greater than 10^{12} nuclei/cm^2. Subsequent growth to a thickness of 100 to 5000 nm results in grain coarsening, polycrystalline texture development, and columnar growth yielding an anisotropic material whose local grain size depends on the distance from the nucleation layer and the growth conditions. The ultra-nanocrystalline films, on the other hand, have much less grain coarsening, significantly higher sp^2 carbon fraction (in fact the evidence for crystalline carbon is barely detectable with conventional techniques) and much higher optical absorption than the columnar nanocrystalline diamond films reported here.[5,6]

2 EXPERIMENTAL

2.1 Nucleation and Growth of Nanocrystalline Diamond Films

The 'nanocrystalline diamond films' studied in this work were fabricated by establishing a nucleation layer on silicon or silicon oxide substrates by: (1) first pretreating the substrate to the diamond growth plasma conditions for a brief period of time (800 Watts microwave power, 720 C substrate temperature, 900 sccm hydrogen flow and 4 sccm methane flow), typically 10 to 30 minutes; then (2), putting

the treated substrate into an ultrasonic bath of nanocrystalline diamond powder in methanol or ethanol for 10 to 60 minutes; followed by (3), immediate rinsing and washing with ethanol and nitrogen blow dry. This is an implementation of the novel nucleation process reported by Rotter.[8] For film growth, the seeded substrate is returned to the growth reactor and diamond is grown to the desired thickness (800 Watts microwave power, 720 C substrate temperature, 900 sccm hydrogen flow and 3 sccm methane flow). The growth rate and film thickness were monitored *in situ* by diode laser reflectometry at 677 nm. Uniform and conformal nucleation densities in excess of 10^{12} cm^{-2} were observed on Si and SiO_2. The growth reactor was a commercial microwave plasma reactor (Model PDS-17, Astex Inc., Woburn, MA) operating at 2.45 GHz with a maximum power of 1.5 kW. Purified hydrogen and methane (99.999%) were used as reactants. The substrate was placed on an inductively heated susceptor and the pressure of the flowing gases was maintained at 15 torr. This reactor had been used previously for growth of boron doped diamond films using diborane as a reactant. Analysis of films similar to the ones studied in this work has indicated residual boron doping levels of 10^{19} to 10^{20} boron atoms per cubic centimeter.[9]

2.2 Device Fabrication

The structures used in this work were fabricated using optical or electron-beam lithography. The pattern is transferred into the diamond films using a CF_4/O_2 reactive ion etch[10,11] with the diamond film protected by a metal mask (Cr or Al). 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 that used for silicon[12] and silicon nitride[13] nanomechanical structures. Most of the structures did not need the critical drying point step[14] for their release.

3 RESULTS

A typical nanocrystalline diamond film is shown in figure 1. This film was grown on a silicon substrate which was subsequently etched away to reveal the freestanding film of thickness ca. 800nm. The combined micro-Raman and photoluminescence spectra of a nanocrystalline diamond film on a silicon substrate excited with 488nm laser irradiation are shown in figure 2. The zone center phonons of diamond and silicon, at 1332 and 521 cm^{-1} respectively are evident, along with a weak resonance Raman band at 1550 cm^{-1} due to amorphous sp^2 bonded carbon. The Raman features are superimposed on a broad photoluminescence spectra ranging from 0 to 8000 cm^{-1}.

An example of a MEMS resonator structure is shown in figure 3. This folded beam MEMS resonator was constructed using p-type conducting nanocrystalline diamond doped with boron. Such structures have demonstrated very high quality factors, Q, of 36,460 for a device resonating at 27,352 Hz.[10]

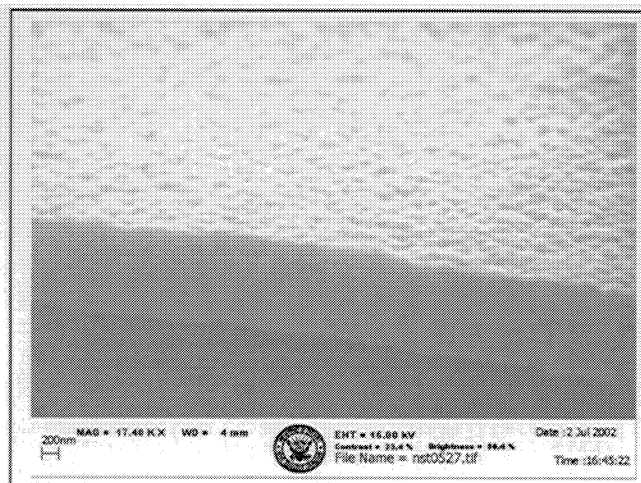

Figure 1: A scanning electron micrograph showing the columnar structure and surface roughness of a ca. 800 nm nanocrystalline diamond film.

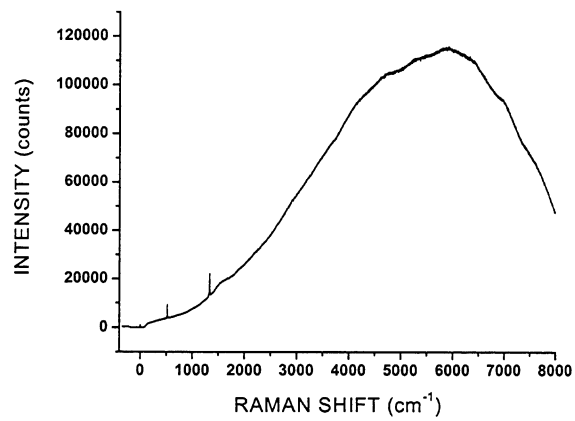

Figure 2: The micro-Raman and photoluminescence spectra of a nanocrystalline diamond film on a silicon substrate excited with 488nm laser irradiation is shown.

An example of a NEMS structure is shown in figure 4. The 'ghostliness' of the SEM image is due to the electron transparency of the ca. 130nm thick nanocrystalline diamond film and the ca. 60 nm wide bars of the structure. The 'tennis racquet' structure is suspended ca. 300 nm above the silicon substrate after removal of the underlying silicon dioxide layer. To the resolution of this and similar images the diamond is fully dense with no breaks or pinholes observed. Nanomechanical clamped beam structures made films 180 nm thick have been demonstrated to resonate at 640 MHz for a beam of 2 microns length.[15]

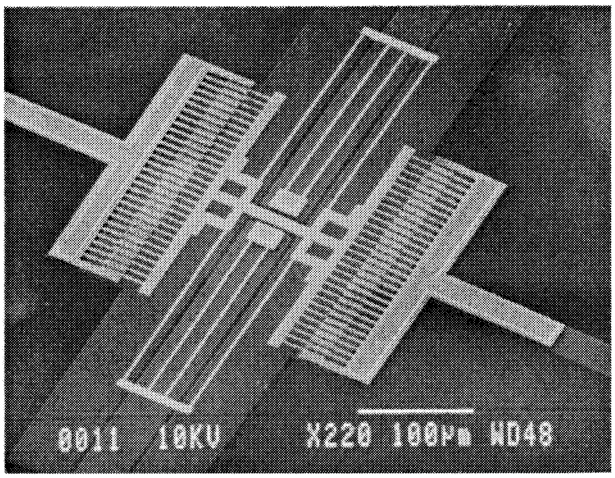

Figure 3: A scanning electron micrograph of boron doped nanocrystalline diamond folded beam MEMS resonator.

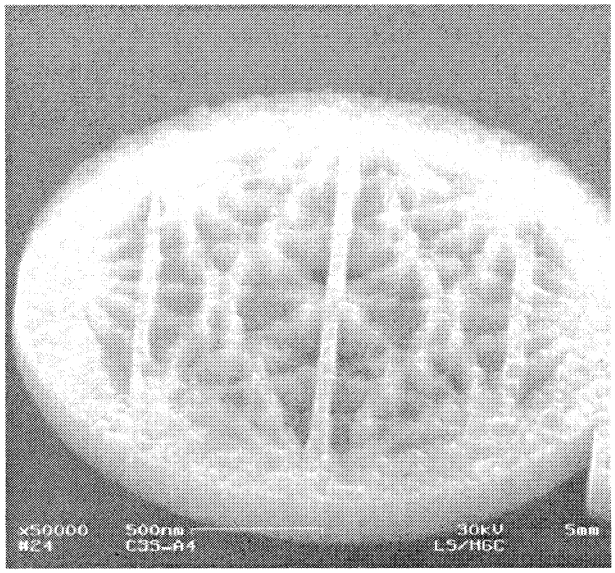

Figure 4: A scanning electron micrograph of boron doped nanocrystalline diamond folded beam MEMS resonator.

4 DISCUSSION AND CONCLUSIONS

Nanocrystalline diamond films of varying doping levels have been shown to be useful materials for MEMS and NEMS applications and compatible with processing steps used in the more traditional silicon MEMS fabrication. This enables the use of diamond as a conductor or an insulator. The films are fully dense and features as small as 40 nm have been fabricated. Structures are fabricated using traditional lithography and etching tools of silicon processing with some modification to the recipes.

The deposition temperature of the NCD film may be an issue in the thermal budget of some complicated device structures where the NCD film is one of the last layers. In our experience, deposition temperatures of ca. 800 C are optimal, but some of the NCD films for MEMS structures were deposited at temperatures between 500 and 600 C.

Approximate mechanical analysis of these resonant structures indicate a Young's modulus of NCD films between 300 and 700 GPa.[10,15] Recent surface acoustic wave measurements on similarly grown NCD films measured between 450 and 980 GPa for Young's modulus and found the value only slightly dependant on films thickness, but very sensitive to the initial nucleation density.[16]

The initial nucleation density is generally as high as $10^{12}/cm^2$, starting from seeds of ca. 3-5 nm dimensions. Subsequent growth coarsens the grain size, leading to the faceting observed in figures 1 and 4. The quality of the NCD is quite high, as determined by the Raman linewidth of 5 to 10 cm^{-1} depending on film thickness. The optical transparency is evident in the detection of the Raman spectra of the underlying silicon and in the oscillations in the broad luminescence background emission (due to interference between the reflections on each side of the diamond film.

In summary, we have demonstrated the nucleation and growth of nanocrystalline diamond films with controlled doping, and have fabricated structures with characteristic dimensions ranging from micrometers to nanometers. Resonant frequencies as high as 640 MHz have been demonstrated in NEMS devices, while quality factors exceeding 36,000 were observed in MEMS devices.

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