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Piezoresistivity of *n*-type conductive ultrananocrystalline diamond

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ABSTRACT

Recent developments of a piezoresistive sensor prototype based on *n*-type conductive ultrananocrystalline diamond (UNCD) are presented. Samples were deposited using hot filament chemical vapor deposition (HFCVD) technique, with a gas mixture of H_2, CH_4 and NH_3, and were structured using multiple photolithographic and etching processes. Under controlled deposition parameters, UNCD thin films with *n*-type electrical conductivity at room temperature (5×10^{-3}–5×10^{1} S/cm) could be grown. Respective piezoresistive response of such films was analyzed and the gauge factor was evaluated in both transverse and longitudinal arrangements, also as a function of temperature from 25 °C up to 300 °C. Moreover, the gauge factor of piezoresistors with various sheet resistance values and test structure geometries was evaluated. The highest measured gauge factor was 9.54 ± 0.32 at room temperature for a longitudinally arranged piezoresistor with a sheet resistance of about 30 kΩ/square. This gauge factor is well comparable to that of *p*-type boron doped diamond; however, with a much better temperature independency at elevated temperatures compared to the boron-doped diamond and silicon. To our best knowledge, this is the first report on piezoresistive characteristics of *n*-type UNCD films.

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1. Introduction

The piezoresistive effect is the fundamental of almost all the mechanical force sensors (pressure, acceleration, vibration, etc.) and piezoresistive sensitivity of a sensor is defined as the relative change of the electrical resistivity due to the applied mechanical strain. Silicon is the most common used material for piezoresistive sensors due to its remarkable piezoresistive characteristics and well established fabrication technology; however, silicon based sensors are not functional in harsh environments and exhibit mostly an operation-temperature limit of below 200 °C [1]; although some technologies, such as Silicon-On-Insulator (SOI), could improve the operating temperature of such systems to about 600 °C [2], silicon based sensors still show lack of long term stability at elevated temperatures and under extreme operating conditions, involving oxidation, corrosion, hydrogen exposure or even very high vibration or acceleration. Diamond, on the other hand, owing to its outstanding mechanical properties combined with chemical inertness, large band gap, high carrier mobility, high breakdown field strength and high thermal conductivity is a more attractive material for sensor realization in harsh environments and at high temperatures, compared to silicon. Single crystalline diamond has limited applications due to its restriction in shape, size and infeasibility for integration into the common microfabrication processes. On the other hand, polycrystalline diamond films, grown by CVD techniques, not only possess a wide range of applications, but also could approach some bulk properties similar to those of single crystalline diamond, based on the microstructure [3]. *P*-type conductivity in polycrystalline diamond, based on boron doping, has been intensively investigated over the past years and such films are successfully integrated in different sensor applications [4,5]. On the other hand, *n*-type doping of polycrystalline diamond films using nitrogen and nitrogen-containing gases remains still challenging, since the conduction shows a very different mechanism than in the classical *n*-type doping. Many researchers proposed a grain boundary transport mechanism as a result of increased ordering and amount of sp^2-bonded carbon in the grain boundary phase, resulting in enhanced density of states at the Fermi level, and therefore, increased electrical conductivity [6,7,8]. To this date, no piezoresistive analysis for *n*-type polycrystalline diamond has been reported: therefore, all available experimental results on diamond piezoresistivity refer merely to the *p*-type boron doped diamond. Highest gauge factor was claimed for boron doped single crystalline diamond, although measured experimentally by only a few groups, and proved to be greatly exceeding that of polycrystalline diamond [9]. On the other hand, piezoresistive characteristics of CVD boron doped polycrystalline diamond have been extensively investigated and a relatively wide range of gauge factors (5–250), depending greatly on the grain size, texture, film growth, temperature, electrical resistivity, doping concentration and test structure design and geometry, has been recorded [10–18]. Piezoresistivity in *p*-type diamond is explained mainly as the result of valence bands splitting-off due to the applied strain, while grain boundary scattering reduces the piezoresistive effect in the polycrystalline films, which explains why single crystalline diamond shows more significant gauge factors than the polycrystalline diamond [10,19–21].

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Temperature dependency of gauge factor has not yet been well explained and various behaviors have been reported; it has been observed increasing [12,14,15] and decreasing with temperature [10,22] or even showing almost no temperature dependency, as in heavily boron doped, highly oriented polycrystalline diamond (HOD) [10] and boron doped UNCD films [23]; even various combinations of these behaviors have been recorded [10,13,24–27]. As for doping concentration dependency of gauge factor k, Adamschik et al. observed increasing k with activation energy E_A for high doping concentrations ($E_A < 300$ meV), while for low boron concentrations ($E_A \approx 380$ meV) k decreased. Moreover, they observed a significant decrease of gauge factor in large single-grained piezoresistor followed by existence of only one grain boundary, suggesting deteriorating effect of grain boundaries on the piezoresistivity [10]. In the current paper, for the first time, experimental results of piezoresistive measurements of n-type conductive UNCD films are presented; moreover, the effect of various parameters on the gauge factor is investigated and finally, based on the results, the conduction mechanism is discussed.

2. Experiments

Intrinsic diamond film was grown on <100> oriented double side polished silicon substrate, using HFCVD technique with a mixture of H_2 and CH_4 as the process gas. Before the deposition, the silicon substrate was seeded ultrasonically in a solution containing nanodiamond powder [3]. Approximately 20 μm thick intrinsic diamond film, with electrical resistivity of $>10^6$ Ωcm and surface roughness of about 20 nm, serves as the electrical insulating layer (between the silicon substrate and the n-type diamond), as well as the mechanical carrier of the test structure. Afterwards, conductive UNCD film was grown on top of the intrinsic diamond film in the same reactor with the addition of about 0.7% ammonia (as a nitrogen containing source) to the total gas mixture of H_2 + (5%) CH_4. Under strictly controlled deposition parameters, including deposition temperature and ammonia addition to the gas phase, UNCD films with electrical resistivities as low as 0.02 Ωcm and activation energies in the range of meV (much smaller than that of substitutional nitrogen in the diamond lattice) could be grown. The n-type nature of the electrical conductivity has been confirmed by Hall and Seebeck effect. More details on the n-type conductive UNCD and their characteristics were published previously [6]. The piezoresistors (conductive UNCD) were then selectively etched using a reactive ion etching (RIE) process in an argon/oxygen plasma at 200 W and 0.09 mbar. Evaporated Ti (100 nm)/Pt (40 nm)/Au (200 nm) multilayer was used as the ohmic contact system for the piezoresistors. Current-voltage characteristics measured at room temperature showed an excellent linearity, approving the ohmic character of the electrical contacts. Besides, the Ti/Pt/Au contact system shows high thermal stability that enables high temperature electrical measurements.

To achieve freestanding microcantilevers, the backside of the silicon substrate was etched using standard photolithography and ICP (Inductive Coupled Plasma) etching process resulting in a rectangular intrinsic diamond membrane. Top side patterning of such membrane in the form of freestanding microcantilevers was achieved by a RIE process. As shown in Fig. 1 (left), the freestanding microcantilevers show no visible bending, indicating negligible internal stress in the intrinsic diamond film. Diamond cantilevers with three different lengths of 500, 1000 and 1500 μm (same width and thickness of 400 μm and 20 μm, respectively) were used to investigate the effect of the test structure geometry on the gauge factor. Piezoresistors (active area: 50 × 50 μm, thickness: 1 μm), are located at the root of the cantilevers where the highest mechanical strain is applied. Electrical resistance measurements were performed by connecting four resistors in a Wheatstone bridge configuration and measuring the voltage across the bridge. Longitudinal and transverse arrangements of the sensitive elements enabled direction-dependency investigations of the piezoresistive effect. In the longitudinal arrangement, current density, electric field and mechanical strain are all in parallel; while in transverse configuration, current density and electric field are perpendicular to the applied mechanical strain (see Fig. 6 (right)).

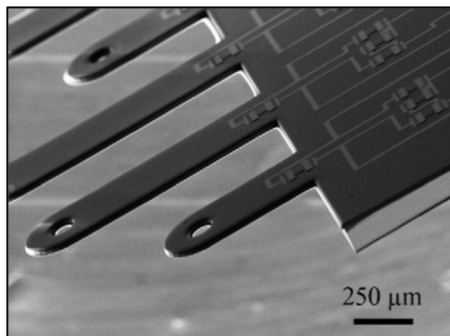
Fig. 2 shows the schematic layout of one test structure. One end of the cantilever is fixed on the silicon substrate, while the free end is deflected stepwise under the optical microscope in 10 μm increments, using a tungsten needle (with a 25 μm tip radius) attached to a micrometer head. The holes etched at the free end of the cantilevers help accurate and stable positioning of the needle. After each step of deflection, resistance change as a function of applied tensile strain is calculated by measuring the Wheatstone bridge voltage output. Gauge factor k, which defines the piezoresistive sensitivity of the piezoresistor material, can be derived from the relative change in the resistance of the piezoresistor $\Delta R / R_0$ (R_0: resistance at zero strain) as a function of applied tensile strain ε, as presented in Eq. (1) [10], where ν is the Poisson's ratio of the intrinsic diamond and ρ is the electrical resistivity of the conductive UNCD film.

$$k = \frac{\Delta R}{R_0} \cdot \frac{1}{\varepsilon} = 1 + 2\nu + \frac{1}{\varepsilon} \cdot \frac{\delta\rho}{\rho} \quad (1)$$

Applied tensile strain at the root of the bending cantilever, where the piezoresistor is located, can be calculated as defined in Eq. (2) [10], assuming that both length and thickness of the cantilever are much larger than those of the sensing elements ($l \gg x$), ($h \gg a$).

$$\varepsilon = \frac{3}{2}\delta\left(l - \frac{x}{2}\right)\frac{h}{l^3} \quad (2)$$

In the calculation of ε, contribution of any intrinsic film strain is neglected. Taking into account that the piezoresistor is much thinner and much smaller than the intrinsic diamond cantilever, one can assume that the above-calculated ε is almost equal to the tensile strain applied on the thin piezoresistor during the cantilever deflection.



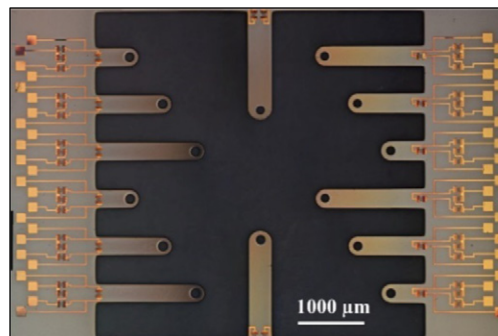


Fig. 1. (left) SEM micrograph of freestanding diamond microcantilevers with longitudinally arranged piezoresistors. (right) Top view optical micrograph of the layout of one testing chip.

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