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DIAMOND AND RELATED MATERIALS

Application of highly oriented, planar diamond (HOD) films of high mechanical strength in sensor technologies[1]

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Abstract

Diamond possesses many characteristics of an ideal material for microsensors, and has indeed emerged as a promising candidate. In comparison to its competitors Si and SiC, large area diamond films are still polycrystalline and inhomogeneous in grain size and orientation. This still determines the material properties, and thus the sensor technology and device performance. However, highly oriented diamond films of high quality have been developed recently, using a modified bias enhanced nucleation method [1]. These films can be described by highly planar, textured surfaces, mirror like backsides, low internal stress and high mechanical strength. Conventional semiconductor processing schemes can now be fully implemented, allowing one to scale high performance micromechanical sensor structures into the lower micrometer range. In this paper, a novel concept based on selective area epitaxy (SAE), pulse doping, reactive ion etching, multilayer contacts and wet chemical backside patterning with micron resolution is presented. The elastic properties and the piezoresistive characteristics of boron doped diamond have both been investigated from diamond cantilever beam deflection measurements. For 15 μm thin HOD-films, a Young's modulus of approximately 830 GPa has been extracted from resonance frequency measurements and nanoindentation measurements. From this data a fracture strength of $\sigma_{fr} = 2.72$ GPa is calculated. To our knowledge, these data represent the highest values reported up to now for such thin films. © 1998 Elsevier Science S.A.

Keywords: Force; Gauge factor; Piezoresistor; Young's modulus

1. Introduction

The properties of diamond, especially the high mechanical strength, the high thermal conductivity, the low thermal expansion coefficient and the chemical inertness against aggressive environment, predestine diamond as an ideal material for sensor applications. Recent developments in diamond nucleation on silicon and advanced outgrowth concepts enabled the large area deposition of "electronic grade", textured diamond films [2]. The smooth, textured surface of these highly oriented diamond (HOD) films allows accurate processing of small dimensions equivalent to silicon technology. The films show low macroscopic stress, resulting in a negligible bending of free standing diamond cantilever beams and diamond membranes. Since the diamond film is grown on ⟨100⟩-oriented Si, the backside patterning (membrane etching) can be performed by standard anisotropic wet chemical etching in KOH solution with a selective etch stop at the diamond interface. The high Young's modulus and the high fracture strain allow the fabrication of micromechanical structures with a very high aspect ratio (length/width), which are expected to yield in improved ranges of force, acceleration and pressure sensing. Furthermore, the chemical inertness of diamond enables the application of these sensors in an aggressive environment. Since boron doped diamond shows piezoresistive effects [3], the active elements for determining tensile and compressive strain can also be fabricated on the diamond structure. Therefore, diamond will serve as a multifunctional material.

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2. Experimental

The highly oriented diamond film was grown on ⟨100⟩-oriented, double side polished 3 in. silicon substrate using AC-bias enhanced nucleation [1]. After outgrowth, the 15 μm thick HOD-film has a surface roughness of approximately 200 nm (peak to valley, measured by profilometry). Backside patterning is performed by a Si_3N_4-mask, standard lithography and wet chemical etching in a 30 wt.% KOH-solution, resulting in rectangular diamond membranes. The boron doped piezoresistors were grown by selective area epitaxy in a MPCVD-system, using a SiO_2-mask for patterning and a solid source boron doping technique [4]. Ohmic contacts were fabricated to highly boron doped diamond layers to facilitate tunneling contacts. For the contact metallization, a silicon based multilayer system is used, which has been reported previously [5]. The top side patterning of the cantilever beams is performed by RIE in an argon/oxygen gas mixture. A multilayer mask consisting of a SiO_2 protective layer and a Ti cap layer was implemented, resulting in a high selectivity between the diamond and mask etching. After etching the 15 μm thick diamond film, the mask is removed in buffered hydrofluoric acid. Fig. 1a shows a SEM-photograph of free standing diamond cantilever beams. The smallest beam has a length of $L=400$ μm, a width of $W=120$ μm and a thickness $d=15$ μm. As seen from the micrograph, no noticeable bending of the free standing beams is observed after etching, which indicates negligible macroscopic stress in the diamond film. To simplify the strain measurements, holes with a diameter of 20 μm were etched into the bars. This allows the application of controlled strain to the identical point of geometry by the nanoindenter or the micro manipulator. All measurements were performed at room temperature.

3. Results and discussion

Due to the negligible transversal piezoresistive effect in boron doped polycrystalline diamond films [3], the piezoresistive structures were implemented for measuring the longitudinal strain at the root of the beam (Fig. 1b). The response was investigated by applying tensile and compressive strain on the cantilever beam by a micro manipulator and measuring the change in resistivity of the piezoresistor. Fig. 2 shows the schematic test configuration. The beam ($L=1500$ μm, $W=570$ μm, $d=15$ μm) was deflected up to $\eta=325$ μm in steps of 5 μm without breakage. As seen from Fig. 3, the curve is linear and fits therefore the elastic deformation theory [6]:

$$\frac{\Delta R}{R}=k\epsilon \qquad (1)$$

k: gauge factor, ϵ: mechanical strain.

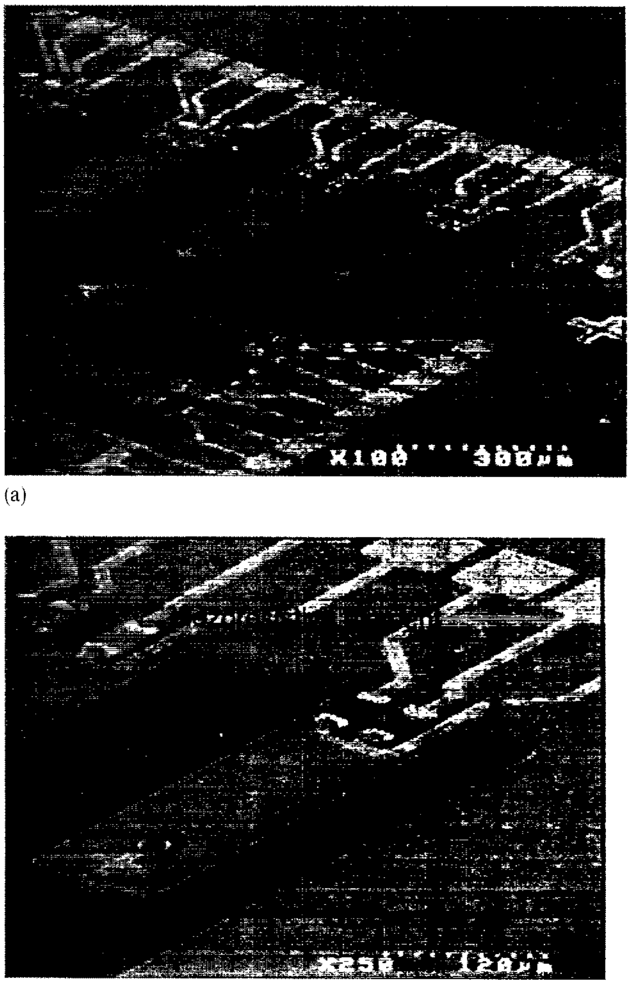

Fig. 1. (a) SEM-photograph of free standing diamond cantilever beams. No bending is observed, indicating negligible macroscopic stress in the HOD-film. (b) Smallest cantilever beam ($L=400$ μm, $W=120$ μm, $d=15$ μm). Boron doped piezoresistors are located at the root of the beam.

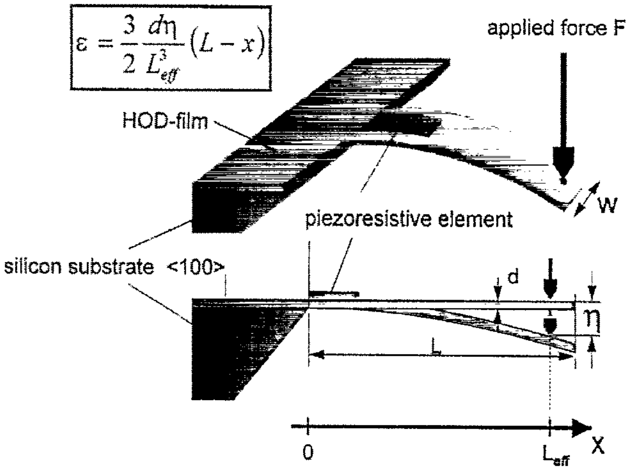

Fig. 2. Schematic test setup. The beam was deflected by a micromanipulator or a nanoindenter.

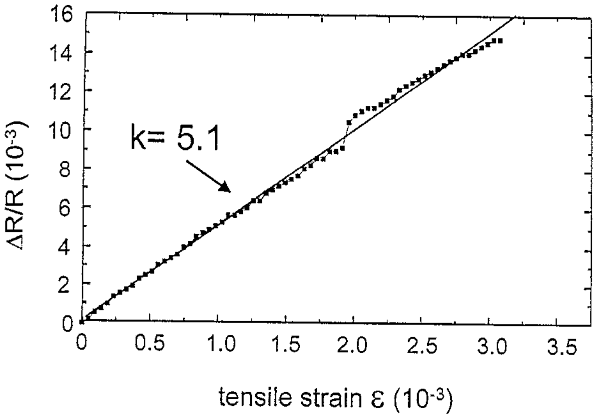

Fig. 3. Beam deflection experiments: mechanical strain versus change in resistance. The beam was deflected by a micromanipulator. The slope yields the gauge factor of $k=5.1$.

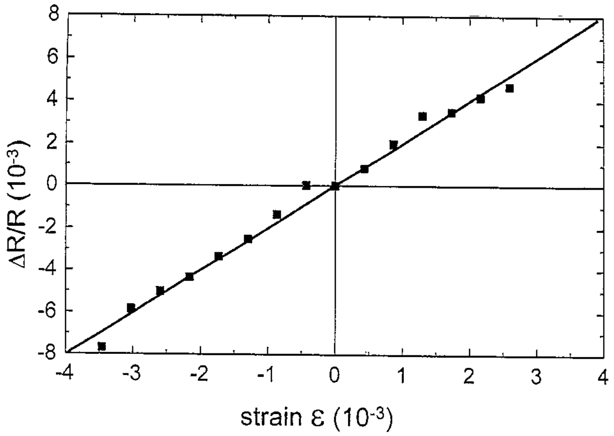

Fig. 5. Beam deflection experiments applying compressive and tensile strain. Compressive strain yields a linear negative change in resitance.

The slope yields a gauge factor of $k=5.1$. It is known that the gauge factor in semiconductors is extremely dependent on the doping concentration [3] (decreasing doping concentrations yield high gauge factors). The boron doping concentration of the investigated piezoresistor structure was determined by temperature dependent conductivity measurements in a temperature range between room temperature and $T=500$ °C. The Arrhenius plot (Fig. 4) yields an activation energy of $E_a=90$ meV for the piezoresistors, which is directly correlated to an effective doping concentration of approximately 7×10^{19} cm^3 [7]. For this doping concentration, the measured gauge factor is in good agreement with literature [8]. Applying compressive strain results in a linear, negative resistance change (Fig. 5). With a controlled doping concentration down to 10^{16} cm^{-3} the gauge factor is expected to increase considerably [3,8]. In this case, to account for the increased thermal sensitivity at low doping concentrations, additional temperature calibration is needed.

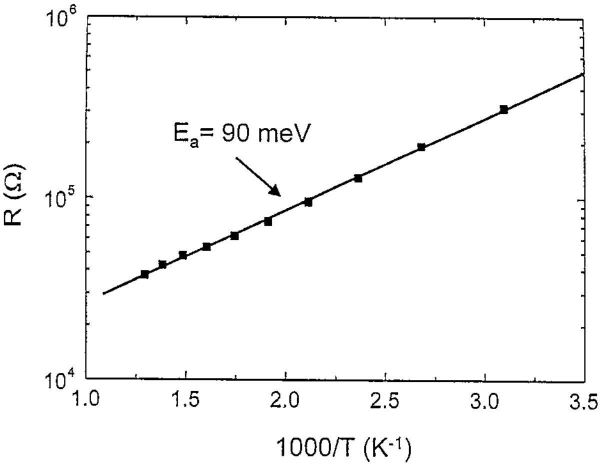

Fig. 4. Arrheniusplot of the piezoresistance in a temperature range between room temperature and $T=500$ °C. The slope yields the activation energy $E_a=90$ meV.

The mechanical properties of the HOD-film were investigated using resonant frequency measurements and nanoindentation measurements. The resonance frequency is a function of the cantilever beams geometry and the Young's modulus can be approximated by [6]:

$$f_{\text{res}}\cong0.16\left(\frac{d}{L^2}\right)\sqrt{\left(\frac{E}{\rho}\right)}$$

ρ: density, E: Young's modulus, d: beam thickness. (2)

Resonance of a beam with $L=1500$ μm, $W=570$ μm and $d=15$ μm was observed at approximately 17 kHz. Assuming the literature value of the diamond density of $\rho=3.5$ g/cm^3 (single crystal diamond), a Young's modulus of $E=890$ GPa was calculated.

This result was verified by nanoindentation measurements of the beam deflection η and the corresponding force F. Fig. 6a shows the measured beam deflection versus applied force. Fig. 6b shows, that the Young's modulus is almost independent of applied force and no hysteresis between increasing and decreasing force was observed. A linearity of approximately 99% was measured over the entire strain regime, which is in good agreement with the literature [6].

$$\eta=4\,\frac{L_{eff}^3}{E\cdot W\cdot d^3}\cdot F \tag{3}$$

Eq. (3) yields a Young's modulus of $E=825$ GPa and verifies the value obtained from the resonant frequency measurement. To investigate plastic deformation of the material, a hold time of 10 min at the maximum force was applied. No effect on the diamond film was observed. The Young's modulus of 825 GPa represents approx. 80% of that observed for single crystal diamond ($E=1050$ GPa). From this data, a fracture strength of $\sigma_{\text{fr}}=2.72$ GPa is calculated for a diamond cantilever

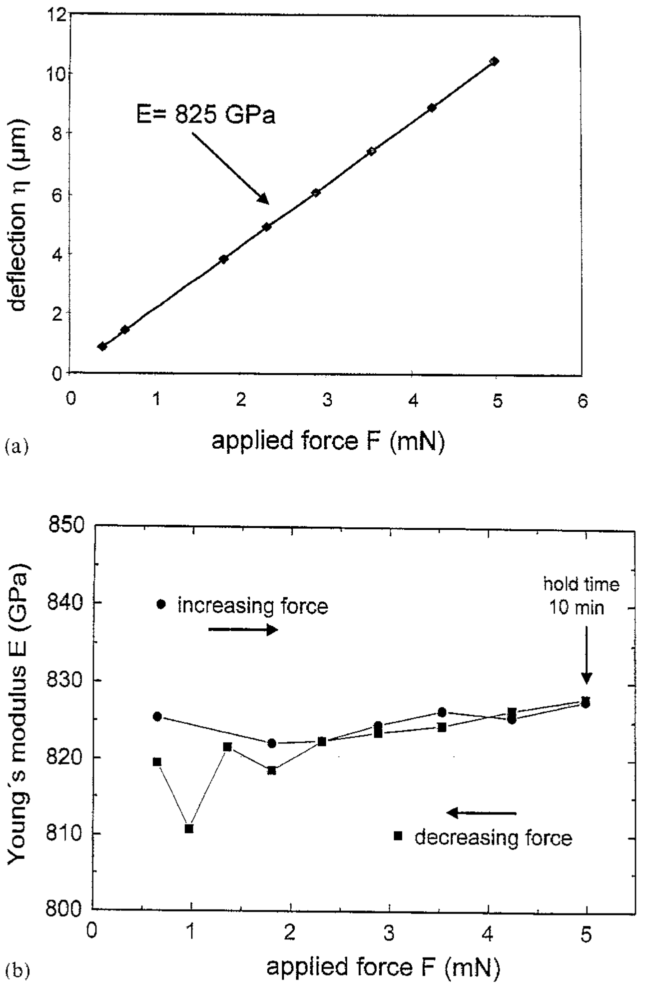

Fig. 6. (a) Nanoindentation measurement. A linearity of 99% is observed in the strained regime. The slope yields the Young's modulus of $E = 825$ GPa. (b) Young's modulus versus applied force. The maximum force ($F = 5$ mN) was kept constant for 10 min to investigate plastic deformation. Neither plastic deformation nor hysteresis effects were observed.

beam of $d = 15$ µm thickness, which fractured at a deflection of $\eta = 325$ µm. To our knowledge, these are the highest values reported up to now for such thin films.

4. Conclusions

The improved growth of highly oriented diamond (HOD) films on Si-substrate enables for the first time diamond microsensor device processing with micron resolution, utilizing techniques equivalent to Si-processing. Diamond cantilever beams with high aspect ratios have been fabricated using wet chemical etchback and reactive ion etching. Boron doped piezoresistors have been integrated, using selective area epitaxy, solid source boron doping and silicon based ohmic contacts. The HOD film of 15 µm thickness shows negligible macroscopic stress, thus no bending of the free standing diamond film has been observed. The elastic and piezoresistive properties of diamond cantilever beams have been investigated by beam deflection measurements. A Young's modulus of 825–890 GPa has been measured, which is more than 80% of that of single crystal diamond and still approximately two times that of 3C–SiC. A gauge factor of $k = 5.1$ has been extracted. This value is limited by the high boron doping concentration of approximately $7 \times 10^{19}\text{cm}^{-3}$. Thus, thin large area HOD films show excellent mechanical properties which can now be implemented into micro mechanical sensor structures determining force, acceleration or pressure.

Acknowledgement

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