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Novel plasma treatment in linear antenna microwave PECVD system

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ABSTRACT

This work reports on hydrogen termination of nano-crystalline diamond films and the behavior of polymer SU-8 as passivating layer after plasma treatment performed at low temperature in a novel linear antenna microwave plasma enhanced system. Nano-crystalline diamond films were grown by microwave plasma enhanced chemical vapor deposition and then hydrogen terminated at different substrate temperatures. The results indicate that a temperature as low as 200 °C is sufficient to reliably attain a diamond surface conductivity of the order of 10^{-7} $(\Omega/\square)^{-1}$. An increase in substrate temperature up to 400 °C results in an increase in surface conductivity up to 1.7×10^{-6} $(\Omega/\square)^{-1}$. The structural changes of the SU-8 passivating layer, before and after plasma treatment, were investigated by FTIR spectroscopy.

1. Introduction

Diamond is a promising material for advanced devices in bio- and electronic applications [1–3]. Considering its extreme electronic, optical, and thermal properties [4], diamond can be specified as ideal material for fabrication of high performance electronic devices [5–7]. Undoped diamond is generally considered to be a very good insulator, but it also exhibits p-type surface conductivity (SC) and negative electron affinity when it is terminated by hydrogen [8–10]. The typical SC value obtained for mono-crystalline diamond (MCD) is in the order of 10^{-4} $(\Omega/\square)^{-1}$ [11]. Presently, nano-crystalline diamond (NCD) shows good enough SC values (10^{-6} $(\Omega/\square)^{-1}$), suitable for fabrication of electronic devices [12,13]. Moreover, NCD is a more desirable material considering its low cost and possibility of deposition on large areas [14].

To achieve hydrogen termination, NCD films are typically exposed either to hydrogen microwave (MW) plasma or to atomic hydrogen produced by a hot filament source. Both methods are commonly used at relatively high substrate temperatures ($T_{sub} \geq 600$ °C) [12]. Decreasing this temperature in a standard microwave system is no simple task, while the main working principle is based on igniting a plasma ball close to the substrate (i.e. approximately 1–2 mm). This arrangement results in a high thermal load of the substrate surface in the range of 400–600 °C. However, the treatment at such temperatures is an undesired technological step in the fabrication of electronic devices, because of partial or complete damage of the metal electrodes or other electronic parts [15]. Therefore, hydrogen termination at low temperature is essential.

Microwave based surface wave-discharge system in linear antenna arrangement represents an alternative plasma source for low temperature processing [16,17]. The main advantage of linear antenna microwave plasma system is a larger distance between the high-density plasma region and the sample (50–100 mm) [18]. Thus, overheating of the substrate from plasma radiation is minimized. Another priority of such system is its ability to ignite stable high-density plasma down to low pressures of 10^{-2} mbar [19]. Additional advantage is its simple scaling-up, i.e. the antenna length could be prolonged up to one meter and the antenna could be multiplied [20,21].

The present work reports on the effect of hydrogen termination on the electrical properties of nano-crystalline diamond films as well as on the behavior of passivating layer (SU-8, 8 μm) after the hydrogen plasma treatment performed at low temperatures in the linear antenna microwave plasma enhanced chemical vapor deposition (PECVD) system. The influence of H-termination on the induced surface conductivity is investigated by I-V measurements. The structural changes of the SU-8, before and after plasma treatment, are characterized by FTIR spectroscopy.

2. Materials and methods

NCD films were deposited on Si/SiO_2 substrates (10×10 mm^2) from a gas mixture of methane and hydrogen in a MW PECVD

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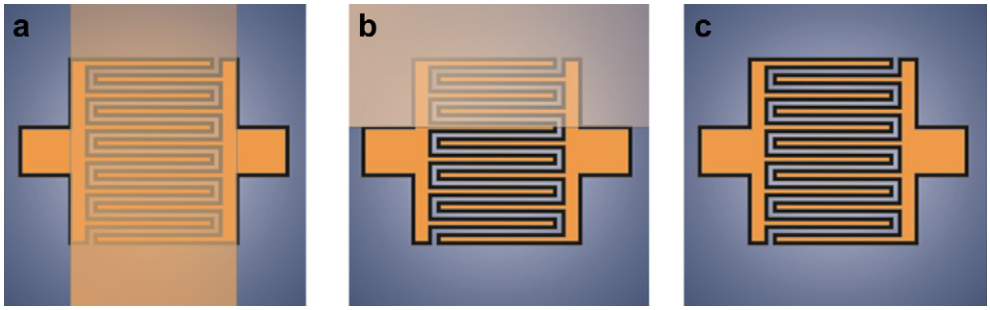

Fig. 1. Schematic drawing of sample sets: (a) 100% SU-8, (b)50% SU-8 and (c) 0% SU-8.

reactor using an ellipsoidal cavity resonator (Aixtron P6, Germany) [22]. Prior to the deposition process, the substrates were seeded in a suspension of nano-diamond powder (5 nm) using an ultrasonic bath. Details on the nucleation parameters can be found elsewhere [23,24]. The NCD film deposition was performed under the following conditions: hydrogen gas flow 300 sccm, methane gas flow 3 sccm, total vacuum pressure 50 mbar, deposition power 2500 W, and substrate temperature 800 °C.

To investigate the hydrogen termination of NCD films and characterize its influence on the diamond surface conductivity, interdigital metal contacts (IDCs) were fabricated on the NCD surface. The IDCs were prepared as six electrodes in each patterned plurality with a separation distance of 250 μm. The NCD samples with IDCs were exposed to oxygen plasma (300 W, 3 min), to achieve electrically insulating surfaces (resistivity >10 GΩ), defined as a starting point for further experiments. To investigate the polymer stability during the hydrogen plasma treatment, SU-8 3050 resist material was used in the experiments (MicroChem Corporation, Germany). The SU-8 3050 resist consists of multi-functional, highly branched polymeric epoxy resin dissolved in an organic solvent, cyclopentanone (CP).

For our experiments, three different sets of samples were used: 0% SU-8 (bare NCD), 50% SU-8 (NCD half-passivated with SU-8) and 100% SU-8 (NCD fully-passivated with SU-8). Schematic illustration of the different sets is presented in Fig. 1. The samples with 50% SU-8 and 100% SU-8 were prepared by spin-coating of NCD films with 8 μm layer of SU-8 and consequently baked in an oven at 95°/20 min to remove the solvent from the resist material. Quartz substrate with IDCs, fully passivated by SU-8, was used for reference sample.

Then, all NCD structures were exposed to hydrogen plasma at the same conditions (microwave power 1000 W, vacuum pressure 0.1 mbar, 100 sccm of hydrogen flow, and processing time 30 min), only the substrate temperature varied from 150 to 400 °C for the samples without passivating layer (0%SU-8), and from 200 to 300 °C for the samples with passivating layer (50%SU-8 and 100% SU-8). The hydrogen termination of the samples was carried out in a modified linear antenna MW plasma system (AK 400, Roth and Rau). Schematic drawing of the system is shown in Fig. 2. The linear antenna MW plasma system is a commercially available apparatus used for solar cell technology, which consists of a vacuum chamber equipped with two metal antennas (copper) located in quartz tubes. The system makes use of two microwave generators (2.45 GHz, MX4000D, Muegge) working at pulse-frequency up to 500 Hz and maximum power up to 4.4 kW in pulse at each side of the linear antenna (conductor). In this apparatus, plasma propagates along the two antennas and is generated along the outer part of the quartz tubes. The substrate stage, which is water cooled and up-down moveable, is located below the antenna.

The surface conductivity of hydrogen-terminated NCD patterns was characterized by current-voltage (I-V) characteristics. Electrical measurements were performed at ambient conditions, i.e. atmospheric pressure and room temperature, with a DC bias swept in the range from −1.5 to 1.5 V using the Keithley 237 source-measure unit. The bias voltage sweeping rate was 100 mV/s.

Surface morphology and grain size of the deposited NCD films were investigated by scanning electron microscopy (SEM, Raith GmbH, e_LiNE writer). The diamond character of NCD was investigated by Raman spectroscopy using Renishaw In Via Reflex Raman spectrometer with the excitation wavelength of 325 nm.

Structural analysis of SU-8 before and after the hydrogen plasma treatment was performed by the interference-free reflectance-absorbance spectroscopy of p-polarized IR light at Brewster angle of incidence. The optical infrared absorbance spectra in the spectral region were acquired using a Fourier Transform Infrared (FTIR) spectrometer equipped with MCT detector cooled to 77 K. The spectrometer was purged with dry air to eliminate the optical absorption related to the atmospheric water and CO_2. The

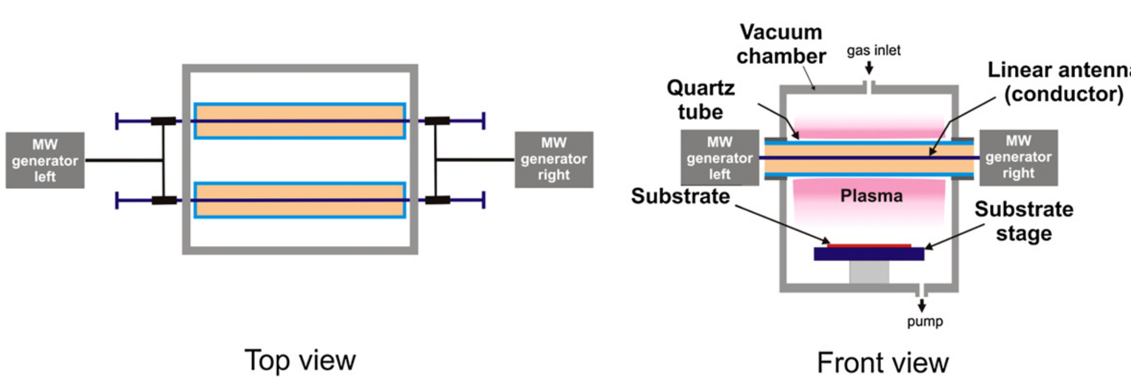

Fig. 2. Schematic drawing of linear antenna microwave PECVD system.