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Fabrication of DLC films by pulsed ion beam ablation in a dense plasma focus device

Z.P. Wang *, H.R. Yousefi, Y. Nishino, H. Ito, K. Masugata

Department of Electric and Electronic System Engineering, University of Toyama, 3190 Gofuku, Toyama 930-8555, Japan

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ABSTRACT

The pulsed intense ion beam, emitted from a dense plasma focus (DPF) discharges performed with hydrogen gas, has been used to ablate the graphite target depositing diamond-like carbon (DLC) films on Si substrates. The substrates were mounted on a holder, which allowed for deposition at positions between normal and 20° off-normal to the target. The samples were removed for analysis after 10 and 20 shots. Nano-particles were observed in the films by a field-emission scanning electron microscope. Raman spectra indicate that sample deposited at 20° off-normal with 20 shots possesses the highest sp^3 content among the samples. The film deposited at this position was also found has the highest hardness.

1. Introduction

The dense plasma focus (DPF) is well known as a compact and an efficient pulsed source of X-rays [1], neutrons [2,3], relativistic electrons and energetic ion beams [4,5]. The current sheath dynamics in DPF allows the formation of high temperature (1–2 keV) and high-density ($\sim 10^{25}$–10^{26} m^{-3}) plasma column at the end of radial collapse phase [6]. The plasma column then disintegrated due to plasma instabilities, which generate energetic ions and relativistic electrons. The DPF device has been experimentally proved to be able to emit ions of characteristic energy from hundreds of keV to tens of MeV [7,8]. The plasma jet and energetic electrons are responsible for the ablation of target material and the ablated material can be deposited on the substrate [9].

Diamond-like carbon (DLC) is a typical form of amorphous carbon consisting of a mixture of sp^3 and sp^2 fractions. The beneficial properties of DLC are the sp^3 constituents that make DLC mechanically hard, infrared (IR) transparent, and chemically inert [10]. The first DLCs were prepared as thin films by Aisenberg and Chabot using ion beam deposition [10]. So far several techniques have been developed to deposit DLC films by different types of chemical-vapor deposition (CVD) and physical-vapor deposition (PVD) methods, such as filament-assisted chemical vapor deposition [11], ion-assisted deposition [12] filtered cathodic vacuum arc [13], microwave plasma-assisted deposition [14], mass-selected ion beam deposition [15], and pulsed laser deposition [16]. Most approaches to obtain DLC need rapidly cooling process. Polycrystalline diamond films have been deposited using a variety of CVD methods at high temperatures [17]. These methods have achieved high-quality diamond films at deposition rates of up to 1 mm/h on small-area (-cm^2) substrates. Rates drop to below 1 μm/h for larger area substrates (>100 cm^2). CVD methods can create DLC films, if low substrate temperatures (<400 °C) are employed [18,19]. Besides, the wide spread use of DLC has been limited by poor adhesion of these films to substrates because of the large compressive stresses induced in the films during the deposition process [20].

It is well known that DPF is not only a system with rapid cooling processes, but also a promising source in this respect offering room temperature deposition of thin films having better adhesion to the substrate owing to the broad energy spectrum of its ion streams [21]. Moreover, as a source of pulse energy beams, the DPF device shares some common characteristics with respect to pulsed laser deposition (PLD), including high deposition rate, possible film deposition under a reactive background gas pressure [22], direct ion irradiation process [7], deposition of materials having high melting temperature as compared to the continuous deposition techniques [23]. As a consequence, there are good reasons to believe that high deposition rate of DLC films with better adhesion to the substrate can be actualized by DPF device. Besides,

* Corresponding author.
E-mail address: wang_zhiping@yahoo.com (Z.P. Wang).

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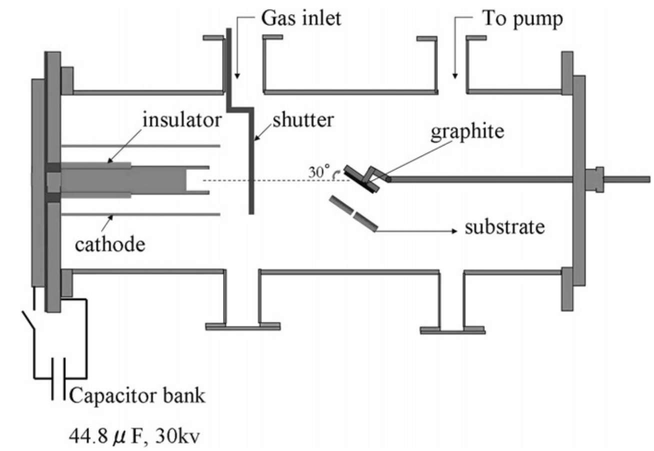

Fig. 1. The schematic of the DPF device employed for DLC film deposition.

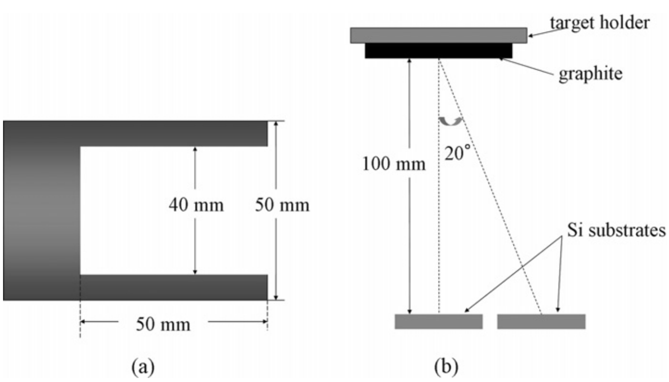

Fig. 2. (a) Featured geometry of anode; (b) featured geometry of target and substrates.

there is also big potential for DPF device to achieve large deposition area.

The previous experiments did by C.R. Kant et al. [23], which deposited carbon films on various substrates using argon ions of a 3.3 kJ DPF, reported that crystalline graphite thin films were achieved on silicon and quartz substrates but the films are amorphous in nature when deposited on glass substrates. Deposition of DLC films on silicon substrates by ablation of graphite anode material with the help of pulsed electron beam in a 1.45 kJ DPF device under nitrogen environment has been reported by S. Zeb et al. [24]. In the present experiment, it is the first time to utilize ion beam in a DPF device to deposit amorphous carbon films on silicon substrates. Crystal structure, bonding structure, morphology, and mechanical properties of deposited films are studied.

2. Experimental procedure

The pulsed ion beam, produced by a 20 kJ Mather-type DPF device, has been used in the present experiment. The arrangement of the experiment set-up is shown in Fig. 1. The DPF device consists of an inner electrode (anode) and an outer electrode (cathode). The anode is made of copper with diameter and length of 50 mm and 243 mm, respectively. The top of the anode is made into hollow geometry. The detailed parameters of the shape of the anode are shown in Fig. 2(a). It is necessary to mention that the anode material will be ablated by the electrons that emitted from the pinch [25]. As a consequence, the impurity will be deposited on the substrates. In this experiment, the hollow in the anode is made as big in diameter and deep in depth as possible to reduce the deposition of anode impurity. The cathode has a cage-like structure and it consists of 24 copper rods with diameter of 8 mm. The length and inner diameter of the cathode are 230 mm and 100 mm, respectively [4]. The DPF device is powered by a capacitor bank of 44.8 μF. At 30 kV operating voltage the capacitor bank delivers a maximum current of 600 kA with 1.7 μs quarter period.

High purity graphite disc of 50 mm diameter and 5 mm thickness was used as a target and was placed into the chamber at 150 mm from anode tip along axial direction. The carbon films were deposited on commercially available one sided polished Si (100) wafer, which was cut into square shape biscuits of 15 mm × 15 mm and then was cleaned ultrasonically in acetone. The substrates were blown-dry and then set into the chamber with the help of a sample holder, bearing two samples at angular position of normal and 20° off normal to the target. By this design, deposition of varied carbon ion energies in the carbon ablation plasma was analyzed. Detailed parameters are shown in Fig. 2(b). Generally after each fresh loading of samples for thin-film deposition, it takes some shots to condition the DPF device for optimized efficiency. In order to prevent the samples from being exposed to weak focusing, a shutter is used before proper focusing was obtained [7].

For films deposition, the chamber was first evacuated up to 4×10^{-5} mbar and then H_2 was introduced as working gas at the optimum pressure of 3 mbar. The work of optimization of the DPF device at various working gas pressures has been done by monitoring the Rogowski coil current and ion current density. When the pulse trigger is switched on, the discharge starts over the insulator surface and then the plasma sheath comes off and is accelerated axially by the magnetic field auto-generated by the current (Lorentz force). After the current sheath runs over the upper end of the central electrode (anode), the plasma is compressed in a small region (the focus or pinch). According to the plasma instabilities, the pinch column collapsed and beams of ions (proton in our case) and electrons were generated [5].

The number of focus deposition shots is believed to play an important role to alter the stress in the deposited films [21]. As a result, after DPF system reached the optimum condition, 10 and 20 shots were chosen for DLC formation. In this experiment, energetic pulsed ions emitted from DPF were used to ablate the graphite target and creating the carbon ablation plasma. The ionic, atomic, and molecular carbon species in the ablation plasma offered a good carbon ion source for DLC formation. The as-deposited samples were investigated by X-ray diffraction (XRD, XRD-6000, Shimadzu), field-emission scanning electron microscopy (SEM, JSM-6700F JEOL), Raman spectroscopy (NRS-1000, Japan Spectroscopic Co., Tokyo, Japan) and nano-indentor (Fisher scope, H100C).

3. Results and discussion

The sample treated at 20° off-normal for 10 shots wasn't selected to characterize in this section because the film thickness is too thin for Raman and nano-indentor measurements. Besides, the film deposited at this position has poor adhesion to the substrate, which is inferred that 10 shots is not enough to supply sufficient ion energy flux to break the barrier of activation energy of silicon substrate.

3.1. XRD results

Samples treated at normal position for 10 and 20 shots along with sample at 20° off-normal for 20 shots were chosen for characterization. XRD spectra were taken over the range of $2\theta = 20$–$80°$ at a scan rate of 0.2°/min. Fig. 3 shows the XRD patterns of as-deposited samples under different conditions of 10 shots normal, 20 shots normal and 20 shots off-normal. The diffraction peaks which observed at $2\theta = 43.3°$ and 50.4°, corresponding to (111) and (200), of copper. The presence of these copper peaks