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A thermocouple-based remote temperature controller of an electrically-floated sample for plasma CVD of nanocarbons with bias voltage

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ABSTRACT

We report an accurate and easy-to operate instrument for the temperature control of an electrically-floated sample using a thermocouple in direct contact with it. The signal was transmitted via 2.45 GHz WiFi. We measured and analyzed the discrepancy between the thermocouple and a radiation thermometer for the plasma CVD of carbon nanomaterials under sample bias. A successful protection method from abnormal discharge in the plasma is also provided.

1. Introduction

Extension of electrochemical concepts to plasma processes are now gathering considerable attention from both the plasma community and electrochemistry community [1–10]. The distinct feature of plasma electrochemistry is its high energy and high temperature processes in combination with gas-surface interactions, as well as wide potential windows that the gaseous environment allows. From this point of view, the plasma CVD processes of carbon materials, such as bias enhanced nucleation of diamond thin films [11],aligned carbon nanotube forests [12] and their chirality control [13], are involved with a kind of electrochemical process in plasma because they are highly dependent on the bias voltage applied between the substrate and the chamber wall. In order to control the process, it is necessary to precisely measure the sample temperature, as already mentioned in the literature [2]. The temperature of the sample at high potential (>100 V) from the electrical ground level is usually monitored by an infrared thermometer or a pyrometer. However, there is a troublesome parameter, *i.e.*, the emissivity, during operation of the pyrometer. The emissivity is the ratio of the actual radiation from the sample surface at a certain wavelength to that from the blackbody at the same temperature. It is strongly dependent on the composition and morphology of the surface, and thus the value has to be calibrated when a different material is used [14,15]. The difficulty arises when the surface composition and morphology are changing in real time as in the plasma CVD process. On the other hand, if the temperature is not very high (<1500 °C), contact measurement by a thermocouple can be a solution to this difficulty. It is thus highly required to develop a measurement scheme using a thermocouple with an electrically-floated sample. Due to the recent advancement of low power microcontrollers and WiFi technology, it is now possible to do this, without a cumbersome optical cable connection [16] that has been the only recent solution.

Another severe problem during the high voltage experiment in the plasma environment is abnormal discharge events. They produce an intolerable voltage difference between the inputs of the thermocouple signal amplifier and high voltage and current to the heater power supply. We have tried various protection circuits and found a reliable solution.

In this paper, we describe the measurement and protection circuits for the temperature measurement and control of an electrically-floated sample using a thermocouple during the plasma CVD of carbon nanomaterials. We analyzed the difference between the readings of the thermocouple and pyrometer, and demonstrated the bias effect on the formation of carbon nanomaterials.

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2. Experimental setup

Fig. 1 displays the experimental setup. Fig. 1(a) illustrates the complete vacuum system and Fig. 1(b) shows the sample assembly. The sample was insulated by ceramic insulators from the chamber. We used a ϕ 10 mm ultrahigh-vacuum-compatible cartridge heater (Heat Wave Labs Co., Ltd., #101126) because of its high attainable temperature (1200 °C) and low power consumption for small samples. The surface of the heater is insulated from its power leads by ceramics. This feature enables the use of a simple power supply not floated from the electrical ground. The heater was supported from three directions with set screws and 0.1 mm-thick Ta foil. The sample was a 6 mm × 6 mm Si wafer with catalyst nanoparticles. It was attached to the heater surface (made of molybdenum) with clamps. A chromel–alumel (K-type) thermocouple of ϕ 0.1 mm diameter was clamped on the sample surface. Most part of the thermocouple lead was electrically shielded and only small portion for detecting the temperature is exposed to the plasma. The non-shielded portion was affected by the potential deviation in the plasma, but there was no problem because the measurement circuit was electrically floated. The stability of the measured temperature values assures the measurement was correctly performed (see the result below).

Fig. 2 shows the schematic circuit diagram of the temperature controller using an electrically-floated thermocouple. The electromotive force from the thermocouple is measured by a cold-junction-compensated K-thermocouple-to-digital converter (Maxim MAX6675) through a surge filter composed of coils, capacitors and diodes. The output of the MAX6675 was transferred via an SPI bus to a microcontroller (Arduino Uno) with a ZigBee WiFi unit. The thermocouple converter and microcontroller were powered by a rechargeable lead-acid sealed battery (12 V, 900 m Ah) and electrically insulated from the CVD chamber. The signals can be transmitted through a glass window, which enables the operation in a glove box, if necessary. Thermocouple readings were continuously transmitted to another ZigBee – microcontroller (Arduino Uno) – digital-to-analog (DA) converter (Microchip MCP4725) at one-second intervals. The 0–5 V output of the DA converter was connected to the scalable temperature input (scaled so that 0–5 V corresponds to 0–1000 °C) of a PID controller (Chino DB1110). The output of the PID controller was connected to a remote analog control input of a switching power supply (KIKUSUI PAC 18-20). The output of the switching power supply was connected to the sample heater through another protection circuit. The output protection circuit consisted of a line noise filter (TDK ZRAC2220-11) and two Zener diodes that have a limiting voltage (20 V) slightly higher than the output voltage of the switching power supply (18 V). If a higher heater supply voltage is necessary, we found that gas discharge-type surge arresters (for example, Epcos A81-A230V) were also effective when used in place of the Zener diodes, although they were not needed in the experiments presented here.

The sample bias (0–500 V) was supplied via a 1 kΩ series resistor (*R*) from a switching power supply (KIKUSUI PAC 500-0.6). The current flowing in the sample (*I*) increased superlinearly with the bias voltage and was 10 mA at a 500 V bias voltage. The *IR* drop causes a maximum 10 V voltage drop, which is low enough compared to the bias voltage. The series resistor effectively protected the bias supply from discharge.

The plasma for the CVD was generated by a RF magnetron sputtering gun equipped with a graphitic carbon plate target (Nilaco, Inc., 99.98%, 1-in. diameter) operated in CH_4 gas. The geometry of the substrate and the RF sputtering gun was carefully designed to avoid direct deposition from the sputtering gun [17,18]. Raman spectra were measured by Ranishaw inVia with a 532 nm laser. FE-SEM (JEOL JSM-6500FA) was used for the observation of the surface morphology of the grown materials.

3. Results and discussion

3.1. Thermocouple versus radiation thermometer

Fig. 3 shows the readouts of the thermocouple and the radiation thermometer (Japan Sensor FTZ6-R150, monitoring wavelength 1.95–2.5 μm) focusing on the sample surface during the plasma CVD of carbon at a floating voltage. The source gas was CH_4 (70 Pa, 20 sccm) and the RF power consumed in the plasma was 20 W. The sample bias was −400 V. The substrate was a Si wafer spin-coated with Fe/Ni nanoparticles (Aldrich, diameter ∼ 200 nm) dispersed in ethanol. The catalyst thoroughly covered the surface.

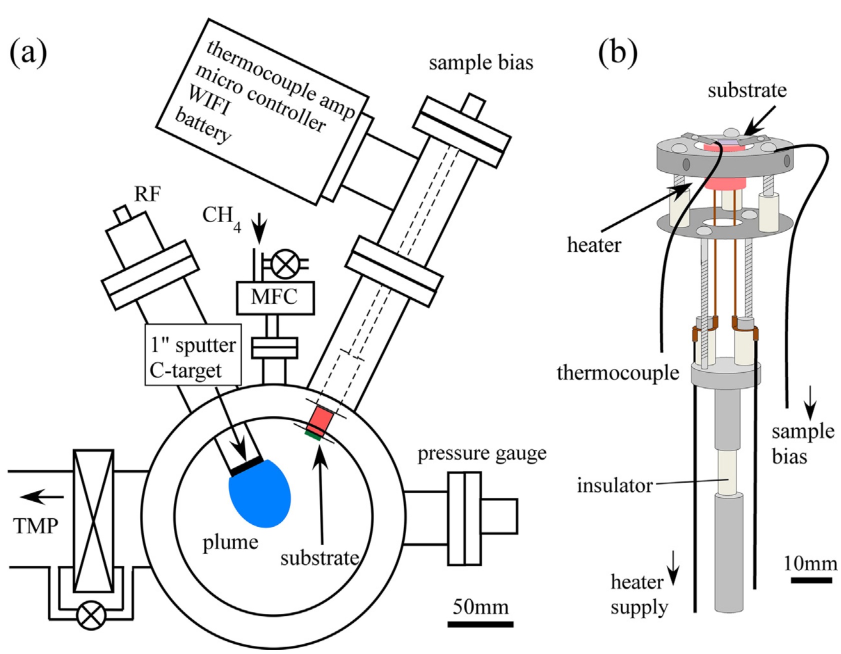

Fig. 1. Experimental setup. (a) Overall schematics and (b) sample assembly.