Preparation of diamond-like carbon films by plasma-assisted chemical vapour deposition in the open atmospheric system

H. Yagi[1], H. Motomura[1], M. Jinno[1]

[1]*Ehime University, Faculty of Engineering, 3 Bunkyo, Matsuyama, 7908577, Japan*

Reactive plasma has been generated under the open air condition by microwave excitation of a downstream flowing mixture of hydrogen and methane. The plasma torch having the shielding gas flow was employed to eliminate the effect from the atmospheric gas such as nitrogen and oxygen. The diamond-like-carbon films were deposited in the methane concentration 10%(CH_4/H_2). The films have DLC properties from the Raman spectra. The morphology of the film was smooth and flat (roughness was less than 0.02 μm). The hardness of the films were about 20-22 GPa.

1. Introduction

In recent days, plasma phenomena and its process under atmospheric pressure have been widely researched. The plasma process under atmospheric pressure is expected to be high speed in deposition and etching in spite of its controllability of plasma. In this situation, if the process under atmospheric pressure realizes in “the open-air”, the processing system becomes simple and the controllability of substrates such as the processing area and the handling of substrates, and also the processing rate becomes high in proportion to the pressure. This enhances the availability of plasma process under atmospheric pressure. In this experiment, we deposited DLC (Diamond-like-Carbon) films by the plasma CVD process under entire open-air condition.

2. Instructions

2.1. Design of torch

Experimental setup employed in this study is schematically shown in Fig.1. Microwave power is supplied by a semiconductor generator at 2.45 GHz from a coaxial cable. The waveguide section of the torch opposite the power input was terminated by a movable contactless plunger. The microwave in the rectangular waveguide is guided to the co-axial waveguide by a waveguide-to-coaxial line transition. The electrode has coaxial structure and the inside diameter of the outer cupper electrode is 12 mm and the outside diameter of the inner tungsten electrode is 4 mm. It is attached to the waveguide vertically. Apparatus of the torch is shown in Fig.1. This torch has the shield gas. The shield gas separate between the plasma and the atmosphere. The process gas is supplied through the gap between outer electrode and inner electrode to the end of nozzle. The plasma is torched between the end of inner electrode and the substrate caused by the high electric field between them as shown in fig.1.

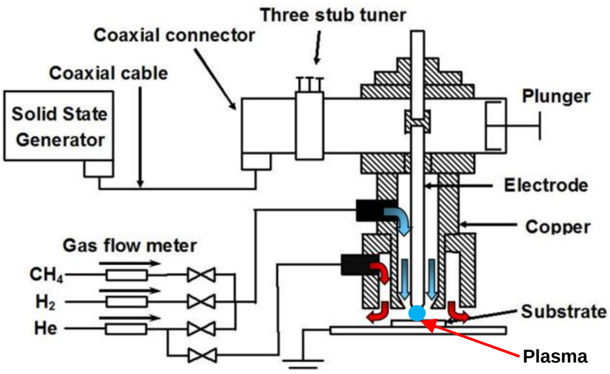

Fig.1 Apparatus of the plasma torch with the flow of shield gas.

2.2 Experimental conditions

There two stages to torch the plasma in the open-air. The first stage is that helium gas was used to torch the plasma. After the plasma was torched, the inlet gas was gradually changed from the helium gas to the mixed gas of hydrogen and methane for depositing films. The plasma still remain ignited after the gas changed. Experimental conditions employed in the

Table 1. Experimental conditions

Incident power	30-50 W
Pressure	atmospheric pressure (open-air)
Gases	He, H_2, CH_4
Flow rate of deposition	CH_4:10-20 (sccm), H_2:100-200 (sccm)
Concentration of methane (CH_4/H_2)	10%
Flow rate of initially torching plasma	He:110-220 (sccm)
Flow rate of shielding plasma	390-780 (sccm)
Substrate temperature	600-680 K (on Si)

present study are summarized in Table 1.

The silicon substrate was placed on a quartz glass plate, the gap between the nozzle and the substrate was 1.0 mm. The temperature of substrate was controlled by the difference between the incident power and the reflected power. The reflected wave can be varied by the three stub unit. The flow rates of the gases were precisely controlled by the mass flow meter.

2.3 Experimental procedure

The DLC films were deposited on the substrate by microwave plasma enhanced chemical vapour deposition (MPECVD) process. The procedure of the experiment in the beginning was as follows; 1) the helium gas was supplied, 2) the microwave power was supplied and the three stub tuner was tuned to torch the plasma, 3) after plasma was torched, the three stub tuner was tuned again to stay predetermined temperature, 4) the supplied gas was changed to the mixture of hydrogen and methane gas.

Flow rate of the helium and the hydrogen gas was about100 to 200 sccm, and the methane gas was varied 10 to 20 sccm. The concentration of methane to hydrogen is 10%. The microwave power was varied from 30 to 50 W and the period of process was 10 minutes. All the processes were executed under the open-air.

3. Results

The deposited films were characterized by the morphology, the structure, the roughness of surface and the hardness. Fig.2 shows the image of deposited film by scanning electron microscope. The film is smooth and homogeneous from the photograph. Fig.3 shows the image of Raman spectroscopy. It shows DLC characteristics[1] of the films. Fig.4 shows the profile of the films by the Surface Roughness Measurement instrument (Mitsutoyo Inc.). The films is very smooth and flat. The height of the roughness (ripple) seems to be 0.02 μm from fig.4. Hardness of the films was measured by the nano-indentation method (Nano Indenter G200, Agilent Technologies Ltd.) for thin films. The hardness of the films was 20-22 GPa. This value is equivalent to DLC films deposited by the conventional method in the low pressure plasma CVD process.

4. Conclusions

The plasma torch having the shielding gas flow was employed to eliminate the effect from the atmospheric gas. The diamond-like-carbon films were deposited. The films have DLC properties from the Raman spectra. The morphology of the films were smooth and flat. The hardness of the films were about 20-22 GPa.

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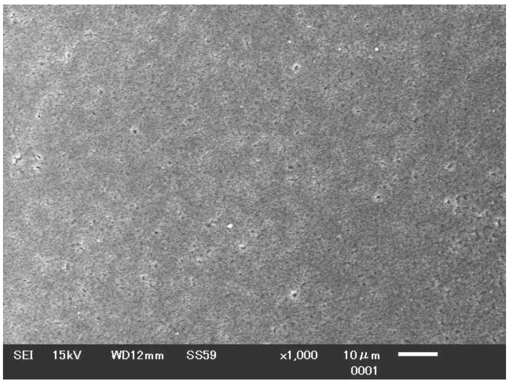

Fig. 2. Scanning Electron microscope image of the deposited films in the open-air. The flow rates of H_2 and CH_4 are 100 sccm and 10 sccm, respectively.

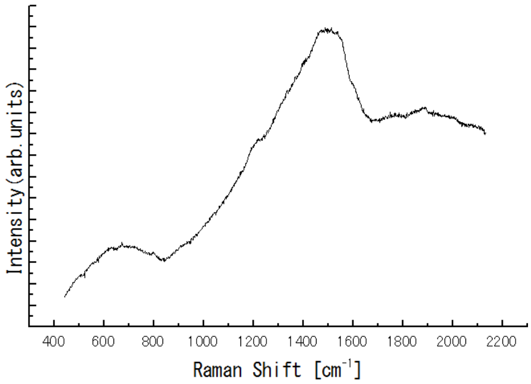

Fig. 3. Raman spectra of the deposited films in the open-air. The flow rates of H_2 and CH_4 are 100 sccm and 10 sccm, respectively.

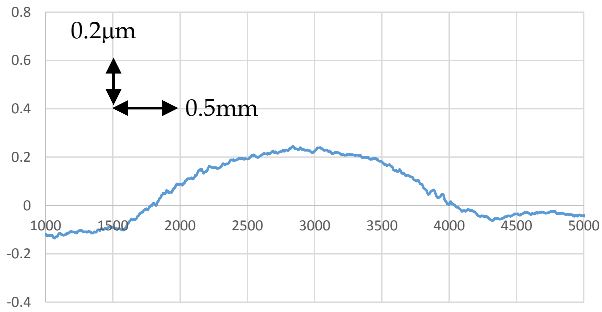

Fig. 4. Surface profile of the deposited films in the open-air. The flow rates of H_2 and CH_4 are 200 sccm and 20 sccm, respectively.

5. Reference

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