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Preparation of Thick DLC Coating on a Trench Surface

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Uniform coating of the thick diamond-like carbon (DLC) on the aluminum rectangular trench was studied using a new hybrid process of pulsed-plasma-based ion implantation and deposition (PBIID). In the PBIID system, a RF pulse for plasma generation and a negative high-voltage pulse for ion implantation were supplied to a workpiece through a single electrical feedthrough. Then the high-density pulsed plasma was produced along the workpiece, leading to the formation of uniform film in thickness on the three-dimensional workpiece. Ion implantation with the negative high-voltage pulse (-5~-20 kV, 1~4 μs and 1~4 kHz) led to the reduction in compressive residual stress of DLC film and the formation of mixing layer, producing the thick DLC film with more than a few μm in thickness using the toluene gas ($C_6H_5CH_3$). The thickness of DLC film was almost uniform on the all surface of trench structure with the width of 2 cm and the depth of 1 cm. For the trench with the width of 1 cm and the depth of 1 cm, the film thickness of the top surface of trench was about 1.3 times larger than that of bottom and 1.9 times than the sidewall. For the trench with the width of 1 cm and the depth of 2 cm, the hollow cathode plasma was generated inside the trench and the film thickness of bottom became comparable with the top one.
Key words: PBIID, DLC, thick DLC, trench, pulse plasma

1. INTRODUCTION

Diamond-like carbon (DLC) films have many superior properties such as a low coefficient of friction and great mechanical hardness that are very attractive for industrial applications [1]. In spite of their excellent tribological properties, DLC films has not been used widely for industrial parts because of their poor uniformity in thickness and poor adhesion to substrate material. The main cause for poor uniformity is due to the hardness of uniform plasma transport from a plasma source to the whole circumference of the three-dimensional substrate with complicated shapes, because metal plasmas such as vacuum arc tend to flow in the one direction. The poor adhesion is resulted from the presence of strong residual stress of more than several GPa in DLC films. The key technologies for good uniformity and strong adhesion of thick DLC coating films are uniform plasma generation around the substrate surface and the reduction of residual stress.

It is known that ion beam implantation reduces the relaxation of residual stress in films [2-4]. Authors demonstrated successfully the uniform generation of high-density plasma around the three-dimensional substrate by a new hybrid processing system of plasma-based ion implantation and deposition (PBIID) in which the substrate itself worked as an RF antenna for plasma generation [5-7]. The thick DLC film of 1.5 – 2.0 μm in thickness was coated on the surfaces of cylindrical pipe and triangular prism with the angle of 30 – 90 degree. The DLC film thickness profile was extremely uniform on the whole surfaces of cylindrical pipe and triangular prism [8].

In this work, we present the preparation of the thick DLC film on an aluminum-alloy (A5052) rectangular trench surface using the hybrid process of PBIID and the toluene plasma ($C_6H_5CH_3$).

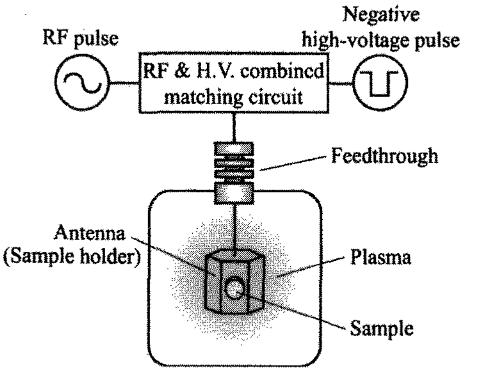

Fig. 1. A schematic diagram of hybrid PBIID system.

2. EXPERIMENTAL

A schematic diagram of the PBIID system is shown in Fig. 1. In this new system, the pulse RF for short pulse plasma generation was supplied to the substrate together with a negative high-voltage pulse for ion implantation through a single electrical feed-through. As the substrate itself was used as a RF antenna, the largest plasma density appeared near the substrate [7].

The cylindrical vacuum chamber has a diameter of 650 mm and a height of 950 mm. Using a joint matching circuit, the RF pulse and the negative high-voltage pulse were supplied to a substrate through a single electrical feed-through. The RF power supply had the maximum power of 3 kW, the pulse duration of 5~50 μs, and the frequency of 13.56 MHz. A negative high-voltage pulse train with the voltage of -5 ~ -20 kV, duration of 1~4 μs, and repetition rate of 1~4 kHz was applied to the substrate at the time of 20~50 μs later after each RF pulse.

Preparation of DLC coating on the trench was performed with hydrocarbon gases and the pulsed RF plasma of very short pulse duration (5~20 μs). The shorter RF pulse duration can produce the plasma inside the deeper trench.

A four steps procedure for good adhesive DLC coating is as follows: The first step is a sputter-cleaning process using a plasma of mixed gases of Ar and CH_4 (50:50%) with the negative medium-voltage pulse (voltage: -10 kV, duration: 2 μs, repetition rate: 1 kHz). In the second and third steps carbon ion implantation by the high-voltage pulse (-10 ~ -20 kV, 1 ~ 2 μs, 1 kHz) was performed by using CH_4 and C_2H_2 plasmas to produce a mixing layer between the coating film and the substrate. The last step is the deposition process for production of thick film using $C_6H_5CH_3$ plasma by the medium-voltage pulse (-3 ~ -5 kV, 2 μs) with the high repetition rate (4 kHz) and the working pressure is in 1 Pa.

Three type of aluminum-alloy rectangular trench were used as the substrate. The trench A has the top width of 1cm, the trench depth of 1 cm and the trench width of 2 cm. The trench B has the top width of 1cm, the trench depth of 1 cm and the trench width of 1 cm. The trench C has the top width of 1cm, the trench depth of 2 cm and the trench width of 1 cm. All trench has the same length of 4 cm. The trench surface was polished to a mirror finish before DLC coating and set at the center of vacuum chamber. Films were analyzed by Raman spectroscopy. The film thickness was estimated from the cross-sectional SEM observation.

3. RESULTS AND DISCUSSION

Figure 2 shows a typical Raman spectrum of the coating film on the aluminum-alloy plate prepared by using toluene plasma. The broad peak of Fig. 2 consists of two Gaussians centered at 1558 cm^{-1} (G peak due to the graphite carbon) and at 1382 cm^{-1} (D peak due to the disordered graphitic carbon). The Raman spectrum illustrated in Fig. 2 exhibits typical characteristics of conventional hydrogenated amorphous carbon (a-C:H) [9]. The fraction of sp^3 bonds in a-C:H is correlated with an area ratio of two Gaussian fit at D and G peaks (I_D/I_G) by Ferrari and Robertson [10]. As I_D/I_G is approximately 2.7 in Fig. 2, the sp^3 fraction is estimated

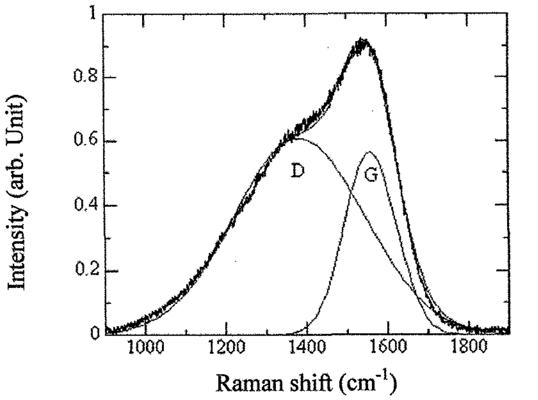

Fig. 2. Typical Raman spectrum of sample prepared by using toluene plasma, where G and D indicate the Gaussian fit for G peak centered on 1558 cm^{-1} and the D peak centered on 1383 cm^{-1}, respectively.

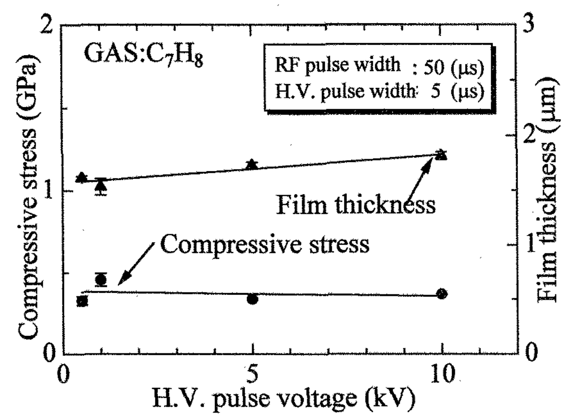

Fig. 3 Dependence of negative pulse voltage on residual compressive stress and film thickness

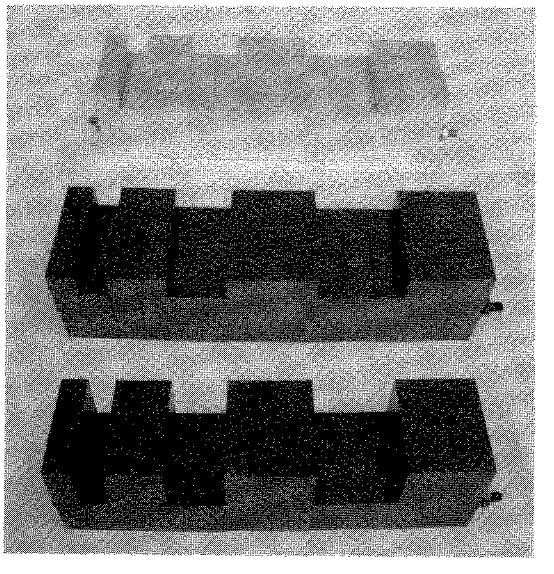

Fig. 4. The photograph of aluminum trench

to be about 25 %.

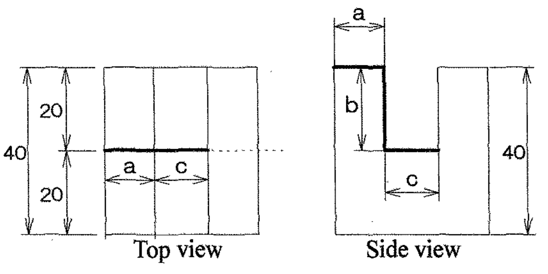

Fig. 5. Schematic diagram of the aluminum trench

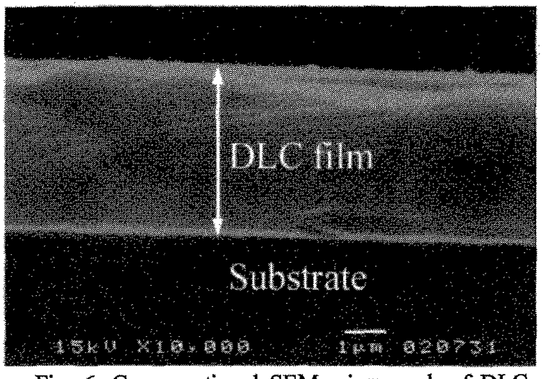

Fig. 6. Cross-sectional SEM micrograph of DLC coating film on the top of trench

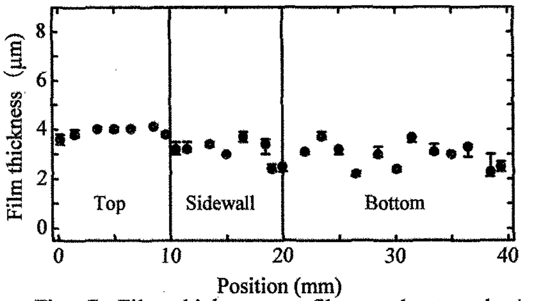

Fig. 7. Film thickness profile on the trench A substrate with the aspect ratio of 2

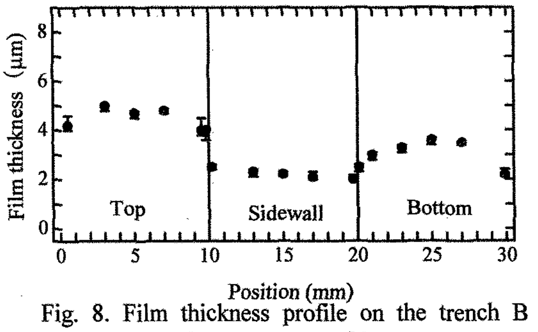

Fig. 8. Film thickness profile on the trench B substrate with the aspect ratio of 1

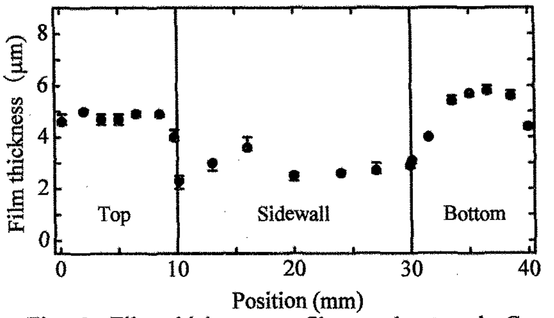

Fig. 9. Film thickness profile on the trench C substrate with the aspect ratio of 0.5

Figure 3 shows the residual compressive stress and the thickness of in the DLC film as a function of pulse voltage at the forth stage (deposition process). As seen in Fig. 3, the film thickness increases slightly with increasing implantation voltage. On the other hand, residual stress is one or two order of magnitude less than conventional DLC films and little dependent on ion implantation voltage. This reason of low residual stress is not clarified now.

Figure 4 shows a photograph of an aluminum trench. The top and the side views of trench are drawn in Fig. 5, where a is the top width, b is the trench depth, and c is the trench width.

Figure 6 shows the SEM micrograph of cross-section of film on the center of top surface of trench A, where a = 1 cm, b = 1 cm, c = 2 cm, and the total process time was 5.5 h. As seen in Fig. 6, the film thickness is approximately 4 μm, indicating the deposition speed of about 0.73 μm/h. From the similar results of cross-sectional SEM observation of DLC film on various parts of trench, the thickness profile of DLC film on the surface of trench A at the center of trench length of 40 mm is shown in Fig. 7. As seen in Fig. 7, the film thickness on the each surface of top, sidewall, and bottom is quite uniform and the thickness of each face is almost uniform. Figure 8 shows the thickness profile of DLC film on the surface of trench B, where a = 1 cm, b = 1 cm, and c = 1 cm. In this case, the film thickness at the top surface is about 1.9 times of that at the sidewall and 1.3 times of bottom. Figure 9 shows the thickness profile of DLC film on the surface of trench C, where a = 1 cm, b = 2 cm, and c = 1 cm. In this case, the film thickness at the top surface is about 2 times of that at the sidewall, while the thickness of bottom is almost same thickness as the top one. The observed increase in thickness of the bottom is resulted from the plasma generation inside the trench by hollow cathode effect.

4. SUMMARY

The hybrid process of plasma-based ion implantation and deposition, in which the substrate itself was used as a RF antenna for plasma production, resulted in the thick DLC coating of 4 ~ 5 μm in thickness on the top of three-dimensional substrates. The DLC film thickness profile was almost uniform on the whole surfaces of the trench with the aspect ratio of 2 and the trench depth of 1 cm. For the case of the trench with the aspect ratio of 1 and the trench depth of 1 cm, the film thickness of top surface was about 1.3 times larger than that of bottom and 1.9 times than the sidewall. For the case of the trench with the aspect ratio of 0.5 and the trench depth of 2 cm, the film thickness of bottom surface was is almost same thickness as the top one. The observed increase in thickness of bottom is resulted from the plasma generation inside the trench by hollow cathode effect.

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