GA12 / P24

Hard Coating of Nanodiamond Composite Films Deposited on Silicon Substrate by Coaxial Arc Plasma Deposition

Ali M. Ali[1,2], Mohamed Egiza[1,3], Koki Murasawa[4], Yasuo Fukui[4], Hidenobu Gonda[4], Masatoshi Sakurai[4], and Tsuyoshi Yoshitake[1]
[1] Dept. of Applied Science for Electronics and Materials, Kyushu University, Kasuga, Fukuoka, 8168580, Japan,
[2] Dept. of Physics, Al-Azhar University, Cairo 11884, Egypt,
[3] Dept. of Mechanical Engineering, Kafrelsheikh University, Kafrelsheikh 33516, Egypt
[4] OSG Corp. 2-17 Shirakumo-cho, Toyokawa, Aichi 442-0018, Japan
Email: abdelgawad_ali@kyudai.jp

Abstract: *Nanodiamond Composite (NDC) films deposited on Si substrates with smooth surfaces by coaxial arc plasma deposition are easily peeled off with increasing film thickness and enhancing hardness of the films. To enlarge the film thickness at which the films are not peeled off, we suggested to employ NDC buffer layers deposited at a substrate temperature of 550 ℃. It was demonstrated that NDC films with thicknesses of more than 1.5 μm, whose hardness and Young's modules are 55 and 438 GPa, can be deposited at room temperature. This thickness is as least 4 time larger than the maximum thickness of comparably hard hydrogen-free diamond-like carbon films deposited at low temperatures by arc ion plating and is comparable with that of ultrananocrystaline diamond films deposited at substrate temperatures of higher than 500 ℃ by CVD. The NDC buffer layers might play roles in enhancing the adhesion strength due to atomic interdiffusion at interfaces with the Si substrates and in releasing the internal stress of the upper hard NDC films.*

Keywords: Nanodiamond; hard coating; coaxial arc plasma; buffer layer.

1. INTRODUCTION

Nanodiamond Composite (NDC) films [1 , 2] have received much attention since they possess physical properties similar to those of diamond and relatively smooth surfaces [3]. They are promising candidates for a variety of applications such as electrical devices, detectors, hard coating, microelectromechanical systems (MEMS), and nanoelectromechanical system (NEMS) [4,5]. MEMS and NEMS devices are currently fabricated on Si substrates, and they are used in applications involving bending and flexural motion, such as cantilever accelerometers and vibration sensors [6]. Although Si substrates are the core of the devices, they have poor mechanical and tribological properties. To improve their properties, hard coating with diamond and related hard materials are beneficial to Si substrates in the applications. In our previous studies, the formation of NDC films by coaxial arc plasma deposition (CAPD) on Si substrates at a substrate temperature of 550 °C with a thickness of approximately 0.4 μm was carried out and their hardness and Young's modulus were at most 23 and 184 GPa, respectively [7].

To increase the thickness at which films can be deposited without peeling off from substrates, an enhancement in the adhesion strength between films and substrates and the release of the internal stress generated in films are the main key factors. Generally, the internal stress in films increases with increasing hardness. In this study, NDC buffer layers deposited at a substrate temperature of 550 °C were employed for hard NDC films deposited at different substrate temperatures, and the effects of the buffer layer insertion on the film thickness at which the NDC films can be deposited without peeling off were investigated.

2. EXPERIMENTAL PROCEDURES

470 nm-thickness NDC buffer layers were deposited on Si substrates at the substrate temperature of 550 °C and a base pressure of less than 10^{-4} Pa by CAPD with graphite targets. A voltage of 100 V was applied to the arc plasma gun equipped with a 720 μF capacitor. The deposition was carried out at a repetition rate of 1 Hz. Posterior to the deposition of the buffer layers, they were cooled down to room temperature at the base pressure. After that, NDC films were deposited on the buffer layers at different substrate temperatures ranging from room temperature to 550 °C.

The films thickness was measured by a surface roughnessmeter at edge-steps between the film and substrate, which were formed by using a mask during the film deposition. In addition, the thickness was also evaluated from cross-section SEM images. The top-view images were taken by scanning electron microscopy (SEM). The crystalline structures of the buffer layer were examined by powder X-ray diffraction (XRD) using 12 keV X-rays from synchrotron radiation at beamline 15 of Kyushu Synchrotron Light Research Center/Saga Light Source. For its measurement, the film was peeled off from the substrate and powdered. The powder was filled in a borosilicate glass capillary with an inside diameter of 0.3 mm. The hardness and young's modulus of the NDC films were estimated by nanoindentation method with applied indentation load 0.5 mN.

3. RESULTS AND DISCUSSION

The hardness of the buffer layers was estimated to be approximately 23 GPa. Figure 1 show the hardness of the films deposited at different substrate temperatures. The hardness increases with decreasing substrate temperature. At the substrate temperature of 550 °C, the hardness become the same value as the buffer layer. The degradation of the hardness and Young's modulus with increasing substrate temperature might be because the sp^2 content in an a-C matrix in the NDC films increases with increasing substrate temperature, in other words, graphitization is induced thermally.

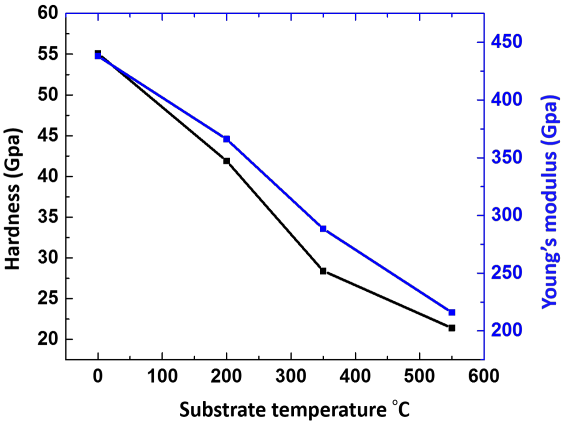

Fig. 1. Dependence of hardness and Young's modulus on substrate temperature.

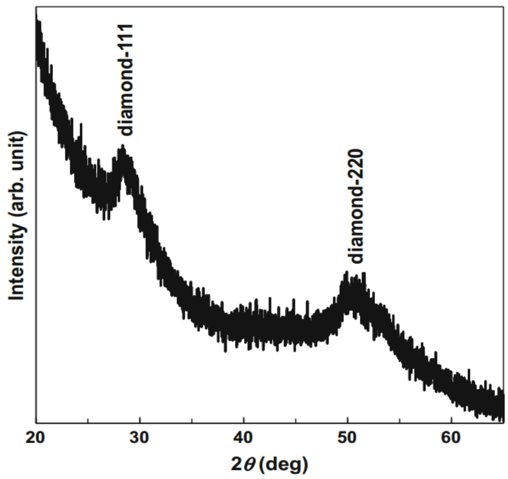

Fig. 2. XRD pattern of NDC film deposited at room temperature.

To confirm the existence of diamond grains in the films, XRD measurements with synchrotron radiation were employed. An XRD pattern shows peaks due to diamond-111 and 220. They are extremely broad, which is owing to a small grain size. The size was evaluated to be approximately 2.4 nm by using Scherrer equation. There are no peaks due to graphite, which indicated that the carbon source is almost completely decomposed into atomic species by arc plasma.

Since the internal stress in films increases with the hardness, the maximum hardness films deposited at room temperature should naturally easily be peeled off from the substrates. However, the film could be deposited at a thickness of 1.5 µm at least. This is the drastic improvement of the film thickness at which the film can be deposited without peeling off. As reasons for the improvement, the following two are considered. One is that the hardness of the buffer layer is not so large. The buffer layer is expected to release the internal stress in the upper films effectively. The other is that atomic interdiffusion at interfaces between the Si substrates and buffer layers are actively enhanced since the buffer layers are deposited at 550 °C, which results in an enhancement in the adhesion strength. In the case of the deposition directly on the Si substrates at room temperature, the interdiffusion hardly occurs and the adhesion might predominantly be due to van der van der Waals adsorption.

By employing the buffer layers, the hardness and thickness are comparably enlarged as compared with those of films prepared by CVD. In the case of deposition at elevated substrate temperatures, an internal stress is induced after coolog down to room temperature due to a difference in the thermal expansion coefficient between the substrate and film materials, which facilitates the peeling-off of the films. On the other hand, atomic interdiffusion at interfaces between substrates and buffer layers are enhanced at elevated temperatures. Both thermal expansion coefficients of Si and diamond are small, thus the effects of the activated atomic interdiffusion at elevated temperatures might preferentially appears for the buffer layers in this study.

4. CONCLUSION

Both high hardness and large thickness of NDC films were simultaneously realized by deposition employing NDC buffer layers deposited at 550°C. The NDC buffer layers might play important roles in enhancing the adhesion strength due to atomic interdiffusion at interfaces with the Si substrates and in releasing the internal stress of the upper hard NDC films.

5. ACKNOWLEDGEMENT

This research was partially supported by Osawa Scientific Studies Grants Foundation, JSPS KAKENHI Grant-in-Aid for JSPS Fellows (Grant No. 15J00654), Grant-in-Aid for Young Scientists (B) (Grant No. 26790019), and JST A-STEP (Grant No. AS2511118 M). The experiment using synchrotron radiation was performed at SAGA-LS with the approval of the Kyushu Synchrotron Light Research Center (Proposal Nos. 1505044S/BL15 and 1410105S/BL12).

REFERENCES

[1] Gruen, D. M. (1999). Annual Review of Materials Science, 29(1), 211-259.
[2] Naragino, H., Egiza, M., Tominaga, A., Murasawa, K., Gonda, H., Sakurai, M., & Yoshitake, T. (2016). Applied Physics A, 122(8), 761.
[3] Spear, K. E., & Dismukes, J. P. (Eds.). (1994). Synthetic diamond: emerging CVD science and technology (Vol. 25). John Wiley & Sons.
[4] Krauss, A. R., Auciello, O., Gruen, D. M., Jayatissa, A., Sumant, A., Tucek, J., ... & Gardos, M. N. (2001). Diamond and Related Materials, 10(11), 1952-1961.
[5] Espinosa, H. D., Prorok, B. C., Peng, B., Kim, K. H., Moldovan, N., Auciello, O., ... & Mancini, D. C. (2003). Experimental Mechanics, 43(3), 256-268.
[6] Krauss, A. R., Auciello, O., Gruen, D. M., Jayatissa, A., Sumant, A., Tucek, J., ... & Gardos, M. N. (2001). Diamond and Related Materials, 10(11), 1952-1961.
[7] Yoshitake, T., Nakagawa, Y., Nagano, A., Ohtani, R., Setoyama, H., Kobayashi, E., ... & Nagayama, K. (2010). Japanese Journal of Applied Physics, 49(1R), 015503.