プラズマ窒化処理ステンレス鋼表面に作成したDLC膜の特性評価

Characterization of DLC Films Deposited on Plasma Nitrided Stainless steel

東京大（院）*アブデルラフマーン　ファルガリー　　表面機能デザイン研究所（非）相澤　龍彦
東京大（正）崔　埈豪

Abdelrahman Farghali*, Tatsuhiko Aizawa**, Junho Choi*

*The University of Tokyo, **Surface Engineering Design Laboratory, SIT

1. Introduction

Stainless steels acquired great importance in both industrial and medical communities over the last few decades since they had excellent corrosion resistance and mechanical strength enough to be utilized in a wide range of applications. However, some grades of stainless steel still had poor tribological properties (e.g., high friction coefficient and abrasive wear rate); they shortened the lifetime of tools and parts in these applications and increased their maintenance cost [1].

Many attempts to overcome these limitations have been conducted by depositing a thin layer to improve the mechanical and tribological properties. In particular, among the diamond-like carbon (DLC) coatings, a hydrogenated amorphous carbon (a-C:H) thin-film material that best satisfies the previous classification. Recently DLC films attracted significant attention due to their ability to be tailored to fit several applications in severe conditions and environments [2].

Nevertheless, depositing DLC film on metallic substrates is often hindered by its poor adhesion caused by different thermal expansion coefficients of both steel and DLC film which drives total delamination. In order to solve this problem, various types of interlayers (Si, Mo, W, CrN, and TiN) have been tested [3].

In this paper, plasma nitriding is utilized as a pretreatment process in an attempt to enhance the adhesion strength of DLC film on a steel substrate. A comparison between DLC film deposited on nitrided steel as opposed to non-nitrided steel substrates in terms of mechanical and tribological behavior is investigated.

2. Experiment procedure

The material used in this study is AISI-SUS420-J2 martensite stainless steel with a chemical composition of Cr 0.08%, Ni 2%, Mn 1%, Si 1%, P 0.04%, C 0.15%, Si 0.03%, and Fe in balance.

A RF-DC high-density plasma nitriding system was employed using N_2 and H_2 gases for 4 h and at 673 K. Detailed nitriding procedures were explained elsewhere [4,5].

Diamond-Like Carbon (DLC) films were deposited by bipolar plasma based ion implantation (PBII) technique using Tetramethylsilane (TMS) and Toluene (C_7H_8) gases at ambient temperature for a total of 3 h under positive and negative voltages of +1.5 and -5, receptively.

After both processes, the samples were cut and polished until a mirror-like surface is obtained in the cross-section. All samples were analyzed and observed by Raman spectroscopy, scanning electron microscope (SEM), and energy dispersive spectroscopy (EDS). The adhesion between DLC films and substrate was tested by using the Rockwell C indentation method with 1471N (150 kgf) load. The indented areas were later observed by digital microscopy.

3. Results and discussion

Figure 1 shows the Raman spectra of a-C:H films deposited on the stainless steel substrate with and without nitrided layer. Both D and G peaks are recorded at 1380 cm^{-1} and 1548 cm^{-1} respectively. The calculated I_D/I_G intensity ratios of both spectra are 0.65.

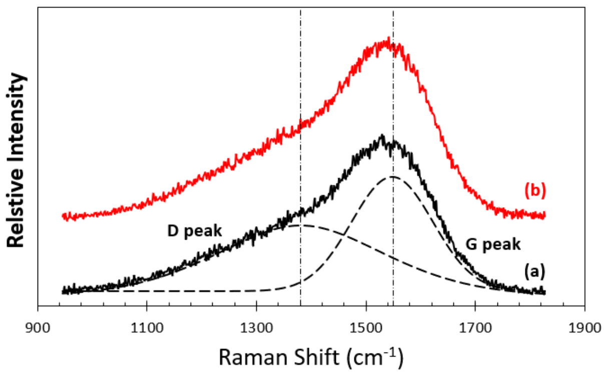

Fig. 1 Raman spectrum of DLC deposited on martensitic stainless steel (a) without nitrided layer and (b) with nitrided layer.

Figure 2a shows the SEM cross-sectional image on the DLC-coated AISI420-J2 substrate including the silicon a-SiCx:H interlayer. The thickness of DLC coating is 2.4 μm and a-SiCx:H interlayer has a thickness of 0.5 μm. The bare nitrided AISI420J2 substrate is also shown in Figure 2b. The nitrided layer thickness is 30 μm with different morphologies than the original substrate material. These different morphologies are obtained due to the nitrogen diffusion into the steel structure which leads to a high percentage of grain refinement and phase transformation from the original martensite (α') phase to the expanded martensite ($α'_N$) phase. This phase transformation is confirmed by the XRD diagram of the nitrided sample.

Concentrations of C, Si, and N elements are detected at each corresponding layer by analyzing the EDS mapping and the SEM images. No trace of oxygen or any other contamination is detected in between the deposited layers which suggests the compatibilities between these dissimilar layers.

Figure 3 illustrates the Rockwell C indentation to test the adhesion of both types of samples. Radial microcracks and partly delaminated areas are spotted around the indented region of the DLC film deposited on the non-nitrided sample as shown in Fig. 3(a). These failures are given a HF2 type of acceptable failures according to the VDI 3198 indentation test [6]. On the other hand, hardly visible few microcracks are spread out of the imprint of the DLC film on the nitrided sample as seen in Fig. 3(b).

Even though the DLC films are deposited under identical conditions, the DLC film on the nitrided sample possesses a more superior adhesion. This can be attributed to the high hardness and stiffness of the nitrided thickness. Furthermore, the 30 μm nitrided layer provides a uniform gradual transition between the substrate and brittle DLC coating which improves the bear loading capacity of the thin DLC film to better withstand high plastic deformation.

4. Conclusion

The supersaturated nitrided thickness of 30 μm is proven to have a tremendous impact on the adhesion strength of DLC film on martensitic stainless steel. Both the a-SiCx:H layer along with nitrided thickness are believed to be a perfect combination to improve the mechanical and tribological properties.

Acknowledgments

The authors would like to express their gratitude to Mr. Hiroshi Morita (Nano-Coat Laboratory, LLC) and Mr. Fukukawa from the microstructure analysis platform for their help in measurement.

References

1) X. Yun-tao, L. Dao-xin, and H. Dong: Improvement of erosion and erosion-corrosion resistance of AISI420 stainless steel by low-temperature plasma nitriding, Appl. Surf. Sci. 254 (2008) 5953–5958.

2) J. Robertson: Diamond-like amorphous carbon, Mater. Sci. Eng. R 37 (2002) 129–281.

3) J. Choi, K. Soejima, T. Kato, M. Kawaguchi, W. Lee: Nitriding of high-speed steel by bipolar PBII for improvement in adhesion strength of DLC films, Nucl. Instrum. Methods Phys. Res., Sect. B 272 (2012) 357–360.

4) A. Farghali, T. Aizawa: Phase transformation induced by high nitrogen content solid solution in the martensitic stainless steels, Material Transactions 58 (2017) 697-700.

5) A. Farghali, T. Aizawa: Nitrogen supersaturation process in the AISI420 martensitic stainless steels by low-temperature plasma nitriding, ISIJ International 58 (2018) 401-407.

6) Verein Deutscher Ingenieure Normen, VDI 3198, VDI-Verlag, Dusseldorf, 1991.

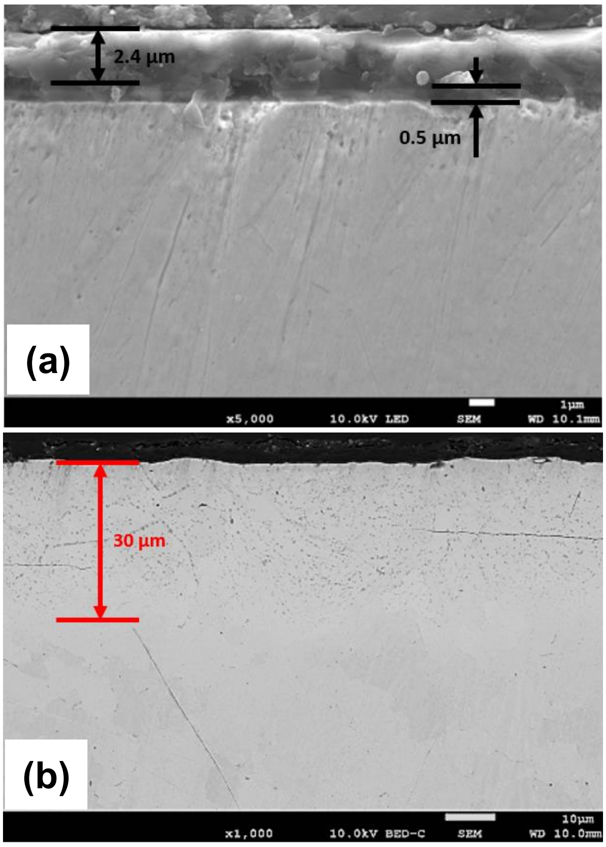

Fig. 2: SEM cross-section micrograph of DLC films deposited on both (a) AISI420-J2 and (b) nitrided AISI420-J2 samples.

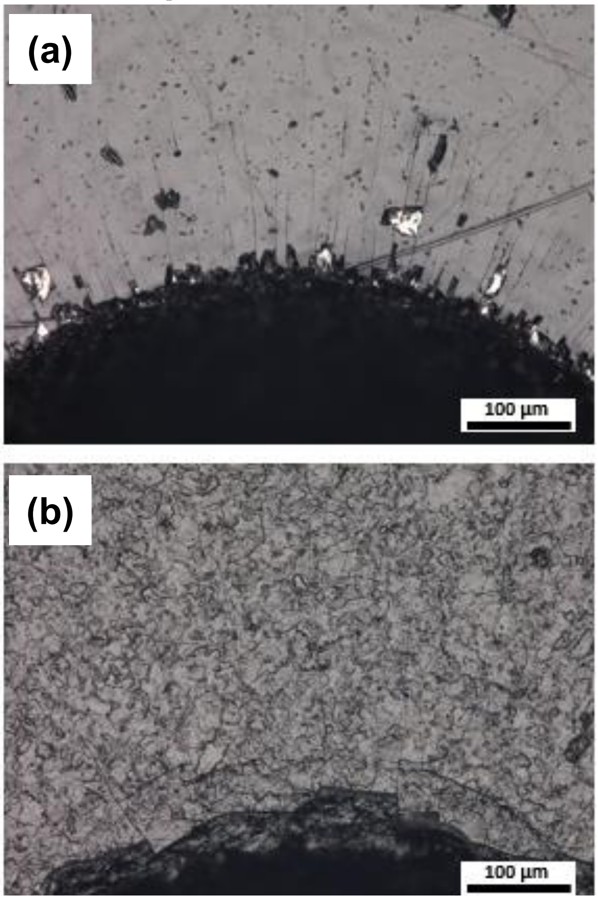

Fig. 3 Optical images on the DLC films indented in Rockwell C with the load of 1471 N. (a) DLC film on the non-nitrided sample, (b) DLC film on nitrided sample.