PLASMA MICRO-PATTERNING ONTO DIAMOND LIKE CARBON COATING

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PLASMA MICRO-PATTERNING
ONTO DIAMOND LIKE CARBON COATING

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ABSTRACT

Plasma etching using pure oxygen gas without hazardous chemical etchants, is proposed to make fine micro-patterning onto the DLC coating with masking chromium. Through experimental studies, the optimum processing condition is determined; the carrier gas pressure of 40 Pa, the RF-voltage of 250 V and the DC bias of -450 V. Owing to the undercoat by chromium and amorphous SiC, this etching process is terminated after perfect dipping the DLC coating under the non-masked regions. No damages and no deterioration was observed on the substrate and the chromium masking. In addition, the etching rate becomes around 5 μm/H, ten times faster than the conventional beam enhanced plasma ashing process.

1. INTRODUCTION

Micro-patterning onto structural parts has been high-lighted in the tribological aspect. Since those patterned micro-pockets store lubricating oils on the contact surface of materials, friction and wear is significantly reduces in practice. As stimulated by this early success, many R & D works take place to make use of micro-patterning on various fields: electronic devices, sensors, optics and mold/die (Bohm, 2001). Since mold-stamping process takes place above the glass transition temperature, both the substrate material and coating must have sufficient strength and toughness even at high temperature in inert gas atmosphere. Diamond-like carbon (DLC) coatings and glassy carbon materials are suitable for substrate of this micro patterning. Authors (Aizawa-2010) have been concerning with micro-patterning onto diamond like carbon (DLC) coating.

This DLC is usually coated onto tools and dies by physical vapor deposition (PVD) and chemical vapor deposition (CVD) methods. These methods create a unique layer of carbon whose characteristics are just like diamond (Kadilaya,2006); e.g. high hardness (50-80 GPa), high thermal conductivity, nanoscale of atomic structure (<5nm), low friction coefficient (<0.01 to 0.7), high abrasion resistance, chemical stability, and transparency to infrared. Metallic interlayer like chromium together with its graded nano-structure layers is also utilized to improve the toughness against delimitation (Bouzakis-2010, Lukaszkowicz, 2011).

In the present paper, DLC coating with interlayer is employed as a mold-die to be micro-patterned. First, the designed micro-pattern is chemically etched onto the chromium-based top-coat in wet. This sample is subjected to oxygen plasma etching. Precise observation and measurement on the patterned micro-grooves provides us the effect of micro-groove size on the etching behavior of DLC coating. With decreasing the pitch of micro-grooves, isotropic etching turns to be anisotropic. This change in etching behavior is caused by chemical reaction

2. EXPERIMENT

Our developing high dense RF-DC plasma etching system is first introduced. Different from the conventional plasma etching, no chemical agents are utilized in this process. Two types of DLC-coated samples are employed as a test-piece. DLC-coated with chromium interlayer is used to measure the removal rate of coatings. DLC-coated SKD-11 sample with initial micro-pattern is also used to describe the oxygen plasma etching behavior.

2.1 Plasma Etching System

Plasma etching system used in this experiment is shown in Fig. 1. In this etching process, only pure oxygen gas is used to remove the DLC layer together with metallic interlayer. This system has three main processing parameters: i.e. RF-voltage, DC-bias and oxygen gas pressure. In parallel with these parameters, experimental set-up has influence on the etching process; e.g. spatial position of dipole electrode to generate RF-plasmas, distance between this electrode and cathode, and, the distance among the electrode, the cathode and the magnetic lens. Typical experimental set-up is depicted in Fig. 2.

In the following plasma etching experiments, the above parameters are varied to find the optimal feasible range in those parameters for efficient removal of DLC coating. Under optimum selection of parameters, micro-patterning is performed to describe the etching behavior. Spectroscopic analysis of generated plasmas is also made for in-situ plasma diagnosis.

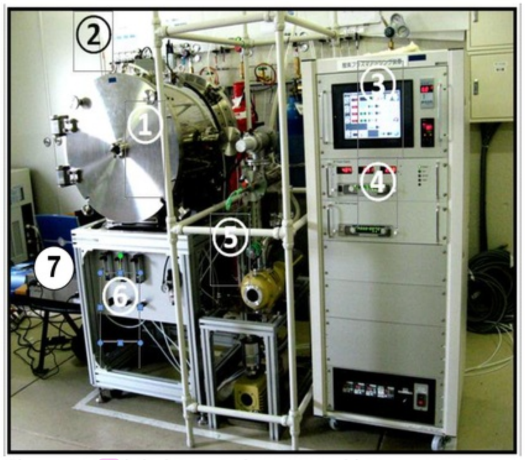


Figure 1. High dense plasma etching system. 1: Chamber, 2: RF-plasma generator, 3: Control-panel, 4: Electric sources, 5: Evacuation system, 6: Gas supply, 7: Plasma Diagnosis (PMA-11)

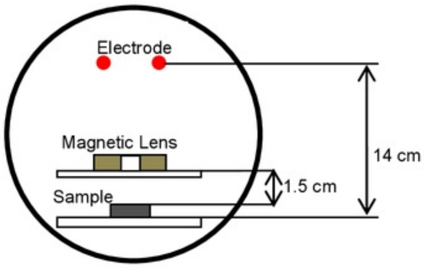


Figure 2. A typical experimental set-up for plasma etching.

2.2 Sample

Two types of samples were prepared to measure the removal rate of DLC coating and to describe the plasma etching behavior.

2.2.1 DLC coated SKD-11 sample

This sample was employed in the preliminary experiments to search for the optimum parameters in plasma etching. SKD-11 is used as a substrate for DLC coating by using PVD RF sputtering. The thickness of DLC film is 1.1 μm.

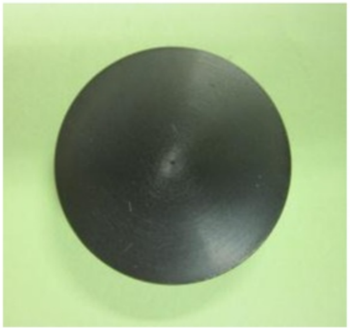


Figure 3. DLC-coated SKD-11 sample.

2.2.2 DLC Chromium Masking

SKD-11 was also used as a substrate for multi-layered coating. Besides the main layer of DLC film, under-coat is made by a pair of amorphous SIC (a-SiC) and chromium to terminate the etching process without loss of anti-delimitation toughness, and, the top-coat, by a pair of chromium layer for chemical etching to make an initial micro-pattern and a-SiC for terminate this chemical etching.

Un-balanced magnetron sputtering was used to form these under-coat, DLC main-film and top-coat, as depicted in Fig. 4.

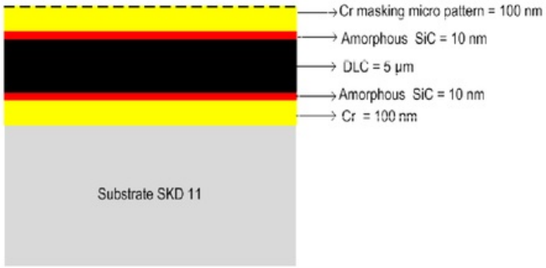


Figure 4. Configuration of multi-layered coating.

Figure 5 shows a chemically etched chromium mask to be utilized as the initial micro-pattern. Using this masking technology, various micro-patterns are formed on this multi-layered coating.

The etching advances first by removal of a-SiC top-coat and then removes the main DLC film. At the presence of under-coat, this etching process is significantly retarded so that the substrate material is free from plasma etching. Micro-groove patterns are varied to have different groove-width from 2 μm to 100 μm with the controlled pitch between micro-grooves.

Figure 5. Chromium-masked DLC-coated SKD 11 before etching

3. EXPERIMENTAL RESULTS AND DISCUSSION

3.1 Etching rate plasma ashing DLC coated SKD 11

The DLC-coated SKD-11 sample, was masked in the left half by polyimide taping. Since only unmasked right-half of sample is ashed by this plasma processing, a step, corresponding to the DLC film thickness (Δx), is formed after etching during the duration time (t). Then, the etching rate is defined by

$$Etching\ rate = \frac{\Delta x}{t}\ nm/s \quad ……. (1)$$

Figure 6 shows the sample after plasma ashing for to measure the etching rate by Eq. (1).

Figure 6 DLC-coated SKD 11 sample after plasma ashing

This etching rate is significantly dependent on the plasma processing parameters and set-up configuration. In the case of 40Pa for pressure, -450 V for DC bias, 250 V for RF, and 15 mm for distance between magnetic lens and sample, DLC coating with the thickness of 1.1 μm, was ashed away for 290 seconds; the etching rate is 3.8 nm/s or 13.7 □m/H. This high rate only for removal of DLC films is characteristic to the present high dense oxygen plasma etching.

3.2 Plasma Diagnosis

In plasma diagnostics, this plasma etching process is characterized by in-situ measurement of time variation of CO peak intensity at the wave-length of 256 nm since removal of DLC coating is driven by chemical reaction between carbon in DLC and activated oxygen atoms in plasmas, With advancement of etching, this peak intensity gradually decreased and became nearly zero. This indicates that the carbon removal process is completed (Aizawa, 2011).

3.3 Optimization of processing parameters

The carrier gas pressure is one of the factors affecting the plasma etching process. Low pressure makes the ionization process faster so that the electron density increases. This leads to much bombardment of activated species onto the coating. Then, lowing pressure is thought to accelerate the etching process (May, 2006). In order to investigate this pressure effect on etching process, the pressure was varied by 40, 55 and 75 Pa with –voltage kept constant by -450 V and 250 V, respectively. The distance between magnetic lens and sample was also fixed by 15 mm. The thickness of DLC of 1.1 μm. Figure 7 depicts the variation of etching rate with pressure.

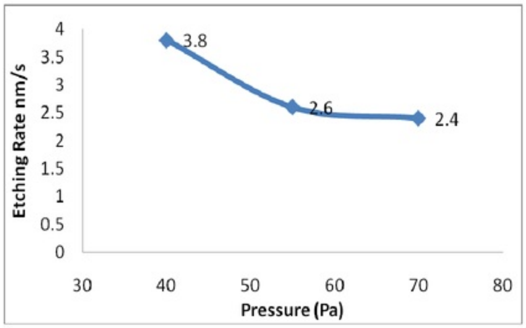
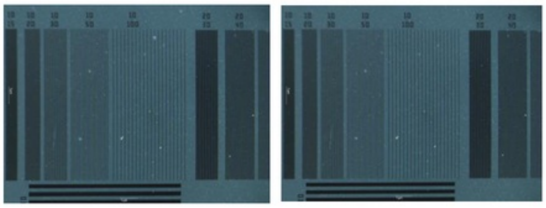


Figure 7. Relationship of pressure and etching rate.

As theoretically predicted, the etching rate is enhanced with lowering pressure. Since the lowest pressure is limited down to be 10 to 20 Pa in the present etching system, the pressure is selected to be 40 Pa in the following experiments.

3.4 Micro-patterning

Multi-layered SKD-11 specimen which was shown in Fig. 5 was employed for micro-patterning. Figure 8 compares the surfaces before and after plasma etching for 3450 s. No change was distinguished between two. This implies that no damage or no deterioration in the original chromium mask tool place during plasma etching.



(a) (b)

Figure 8. Chromium-masked DLC-coated SKD 11 before etching (a) and after etching (b).

The laser reflection profilometer (Mitaka-Kohki NH 3SP) was used to precisely measure the surface profile of etched specimen. As shown in Figure 9, un-masked regions were selectively etched away from the original surface of specimens. On the other hand, the DLC film under the chromium mask was left as columns. Although noisy signals were included in the bottom profiles, the average depth of etched groves were nearly the same; the depth of grooves is 5 μm. This means that the whole thickness of DLC film was successfully etched away in the chromium masked regions. In addition, side-surfaces of each groove is found to be smooth and straight against the bottom. This geometric sharpness is preferable to mold-die to imprint this micro-pattern onto optical polymers and oxide glasses (Aizawa, 2011c).

Figure 9. Depth length of 15 μm micro pattern

CONCLUSION

Dry etching process using plasma etching at 40 Pa pressure, DC bias -450V, RF 250 V and between magnetic lens and sample 15 mm is very effective for removing DLC and DLC make Cr micro patterning.

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