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Phone: (847) 491-3263
Fax: (847) 491-3915

k-ehmann@northwestern.edu

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Technical Program for Tuesday March 26, 2013

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Chair: Jun, Martin Byung-Guk	Univ. of Victoria

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Co-Chair: Li, Kuan-Ming	National Taiwan Univ.

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Park, Simon	Univ. of Calgary

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Pfefferkorn, Frank E.	Univ. of Wisconsin-Madison

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Co-Chair: Maity, Kalipada	NATIONAL INSITITUTE OF Tech. ROURKELA, ODISHA

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Chair: Park, Simon — Univ. of Calgary

WeC1 Pender Island South

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Chair: Bhiladvala, Rustom	Univ. of Victoria
Co-Chair: Park, Simon	Univ. of Calgary

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Aizawa, Tatsuhiko	Shibaura Inst. of Tech.
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Lee, Je-Ryung	Korea Univ.
Woo, Sangwon	Seoul National Univ. of Science and Tech.
Park, Eun-Sok	Korea Inst. of Machinery and Materials
Kim, Hwi	Korea Univ.
Je, Tae-Jin	Korea Inst. of Machinery and Materials
Yoo, Yeong-Eun	KIMM
Whang, Kyung-Hyun	Korea Inst. of Machinery and Materials

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Chair: Araujo, Anna Carla	UFRJ / COPPE
Co-Chair: Annoni, Massimiliano	Pol. di Milano

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Campos, Fabio Oliveira	COPPE/UFRJ

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Biella, Gabriele	Pol. di Milano
Mayor, J.Rhett	Georgia Inst. of Tech.
Rebaioli, Lara	Pol. di Milano
Semeraro, Quirico	Dipartimento di Meccanica, Pol. di Milano

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Chair: Cao, Jian	Northwestern Univ.
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Thiery, Stephane	Arts et Metiers ParisTech
Nyiri, Eric	Arts et Metiers ParisTech
Gibaru, Olivier	Arts et Metiers ParisTech
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Chair: Jeong, Young Hun	Korea Pol. Univ.
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Kulinsky, Lawrence	UCI
Madou, Marc	Univ. of California

Technical Program for Thursday March 28, 2013

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Chair: Ozdoganlar, Burak	Carnegie Mellon Univ.
Co-Chair: Matsumura, Takashi	Tokyo Denki Univ.

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Kashid, Milind	Indian Inst. of Tech. Bombay
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Makino, Takahiro	Univ. of Toyama
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Ohmori, Hitoshi	RIKEN
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Regular Session

Chair: Fassi, Irene	National Res. Council
Co-Chair: Subbiah, Sathyan	Nanyang Tech. Univ.

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Kang, Xiaoming	Shanghai Jiao Tong Univ.
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Zhang, Yanqiao	Univ. of Victoria
Jun, Martin Byung-Guk	Univ. of Victoria

ICOMM
2013
No.

Micro-Imprinting onto DLC and CNT Coatings via High Density Oxygen Plasma Etching

T. Aizawa[1], K. Mizushima[2], T.N. Redationo[3] and M. Yang[4]
[1]Design and Engineering, Shibaura Institute of Technology, Japan; e-mail: taizawa@sic.shibaura-it.ac.jp
[2] Design and Engineering, SIT, Japan; e-mail: y09172@shibaura-it.ac.jp
[3]Mechanical Engineering, University of Brawijaya, Indonesia; e-mail: veneture2@yahoo.com
[4] System Design, Tokyo Metropolitan University, Japan; e-mail: yang@tmu.ac.jp

Key Words: oxygen plasma etching, plasma diagnosis, DLC, CNT, mold-die. Micro-embossing

ABSTRACT

Carbon-based coatings such as DLC (Diamond-like carbon) and CNT (Carbon Nano-Tube) films have been noticed as a structural and functional substrate for micro-forming. First, DLC-coated and CNT-coated silicon substrates were prepared to investigate the micro-imprinting behavior via the high density oxygen plasma etching process. This etching process was also studied by quantitative plasma diagnosis with respect to activated oxygen species and electron density. By using the micro-groove patterning, the etching behavior as well as etching rates, were discussed toward advancement in homogeneous and fast-rate etching. In second, micro-textured DLC-coating was employed as a mother tool for micro-embossing to duplicate these micro-textures onto the aluminum sheets via table-top CNC-stamping system.

INTRODUCTION

Micro-patterning and micro-texturing has grown up as one of main subjects in the mechanical parts and the die and mold technology [1]. The plasma etching methods as well as extremely-short-pulse laser machining provide us a tool to make micro-patterning and micro-texturing onto die-mold materials in addition to the conventional micro-/nano-machining [2, 3]. The chemical etching process has grown up in the semi-conductor technologies; carbon-based materials like diamond-like carbon (DLC) or carbon nano-tube (CNT) are still difficult to be precisely etched for micro-patterning. Originally, DLC coating has been popular as a protective coating of tools and dies for dry forming and stamping at the ambient temperature [4-5]. Hence, mother micro-patterns and micro-textures are once imprinted onto these materials as a mold-die for subsequent micro-forming; then, devices or parts are fabricated in mass-production by duplication of mother patterns and textures via micro-embossing at room temperature [6]. Furthermore, direct micro-texturing onto CNT coating becomes an effective tool to fabricate the electric devices where the patterned CNT plays a role as the electric circuit or the micro-sensor [7].

In the present paper, the high density oxygen etching system is further developed to accommodate the on-line plasma diagnosis and to make uniform and precise etching for micro-patterning onto DLC coating. First, the present system is introduced with some comments on its characteristic features different from the conventional plasma generators. Next, the two dimensional resin-based masks are used to make micro-line-patterning onto DLC coating. Metallic masking is also utilized to make micro-patterning onto CNT coating. Finally, the micro-grid-patterned DLC-coated mold die is fabricated and used for mold-stamping in duplication of these patterns onto aluminum sheet by micro-embossing stamping.

EXPERIMENTAL PROCEDURE

A. TOTAL EXPERIMENTAL PROCEDURE

In the present study, the experimental procedure is depicted in Fig. 1. First, the DLC-coated die material is prepared for micro-texturing onto DLC coating. DLC-coated die with a mother micro-texture is further utilized as an original die for micro-forming. The mother micro-texture is duplicated onto the work materials via micro-forming.

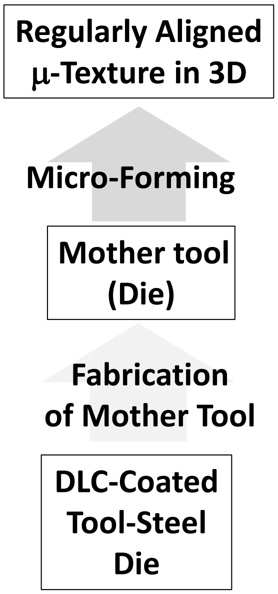

Fig. 1: Microforming procedure to fabricate the micro-textured aluminum sheet in mass-production.

Table 1summarizes the detail design for each processing step in Fig. 1. High density oxygen plasma etching was first invented and developed to make micro-texturing onto the carbon-based materials including DLC coating. Table-top

CNC-stamping system was further invented for micro-printing onto aluminum sheet with sufficiently precise accuracy in geometric dimensions.

Table 1: Design of micro-forming process from mold-die material selection to fabrication of products.

Step of Micro-Forming Process	Present Processing Design
Regular Alignment of Micro-Textures onto Products	Micro-imprinting of plasmonic Patterns onto aluminum sheet
Micro-Forming Process for Micro/Nano Imprinting	Table-top CNC micro-embossing process
Materials Selection of Mother Tool for Micro-Forming	DLC-coating with mother micro-textures
Fabrication of Mother Tool	High density oxygen plasma etching
Materials Selection of Raw Mold-Die Material	DLC-coated AISI420 steel die

B. OXYGEN PLASMA ETCHING

Different from the conventional DC- or RF-plasma generators, where plasmas are ignited and generated in the frequency of 13.56 MHz or its multiples, high density oxygen plasma etching system has no mechanical matching box, as shown in Fig.2. In the present system, input and out powers are automatically matched by frequency adjustment around 2 MHz [2, 8-9]. This difference in power matching reflects on the response time to temporally varying plasma states. The conventional mechanical matching requires for long response time in the order of 1s to 10 s to adjust the RF-power. While, in the present system, it is shortened down to 1 ms; i.e. there is no time delay in power control to drive the etching process. In addition, the vacuum chamber is electrically neutral so that RF-voltage and DC-bias should be controlled independently from each other. RF-voltage is controllable up to 250 V, while DC bias, 0 V to - 600 V.

Fig. 2: High density oxygen plasma etching system to make micro-printing the designed micro-texture onto DLC-coatings. 1: Vacuum chamber, 2: RF-generator, 3: Control panel, 4: RF- and DC-power supplies, 5: Evacuation units, and,6: Carrier gas supply.

A dipole electrode was utilized to generate RF-plasma; DC bias was directly applied to the specimens. Heating unit was also located under this DC-biased table. In the following etching experiments, the specimens were located on the cathode table before evacuation down to the base pressure of 0.1 Pa. Then, a carrier gas was introduced into the chamber to attain the specified pressure. Both oxygen and argon gases were available in this system besides the nitrogen gas for bent. With use of magnetic lens, the ignited RF-DC oxygen plasmas were focused onto the surface of specimens during etching. As had been discussed in Refs. [10-11], the present oxygen plasma etching advanced in the similar manner of RIE (Reactive Ion Etching) where the carbon in DLC or CNT films directly reacted with oxygen atoms in the oxygen radical flux. As illustrated in Fig. 3, un-masked regions were only subjected to this anisotropic etching process, so that the original two-dimensional micro-pattern by masking should be changed to fine three-dimensional micro-texture with stepwise depth profile.

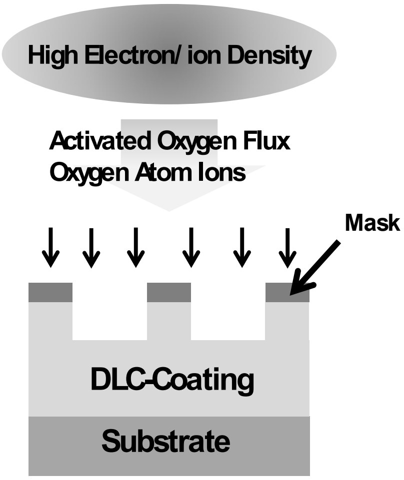

Fig. 3: Reactive ion etching by using the activated oxygen radicals and ions via high density oxygen plasmas.

C. QUANTITATIVE PLASMA DIAGNOSIS

Spectroscopic analyzer of emissive light from plasmas was instrumented in the present etching system. The detector of emissive light was placed onto a silica window of chamber and transferred to the analyzer via the optical fibers. In addition, this spectroscopic analysis was performed to make on-line monitoring of plasmas. Through this analysis, each activated species in the plasmas was identified by its corresponding peak at the specified of wave length. As illustrated in Fig. 4, the Langmuir probe was also equipped to the present etching system. Both the electron density and temperature were also on-line monitored during this etching process. With this information of density and temperature both for electrons, ions and radicals, the plasma state for etching were precisely defined and described to investigate the effect of plasma processing parameters on the etching behavior.

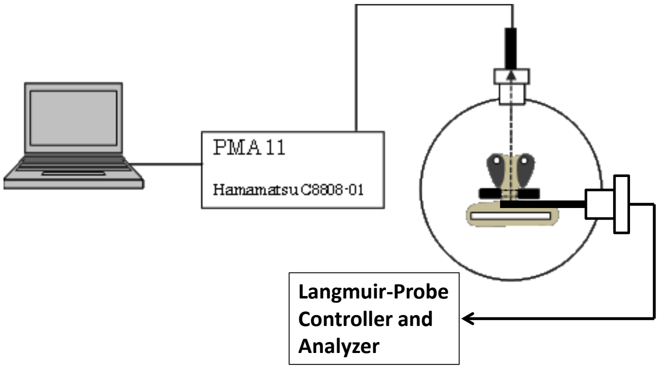

Fig. 4: Quantitative plasma diagnosis to measure the electron density and temperature as well as the spectrum of activated species in plasmas.

D. PREPARATION OF SPECIMENS

DLC-coated silicon substrate with resin-base masking was first prepared to describe the etching behavior of amorphous carbon films via the present oxygen plasmas. As shown in Fig. 5, micro-grooving pattern was imprinted onto the surface of DLC coating by photo-lithography; resin-bases mask with SiO_2 has sufficient toughness against the irradiation of oxygen radical flux. Next, CNT film was directly sputtered onto the silicon substrate with use of catalysis. In this case, metallic mask for micro-grooving was used and located onto the surface of this CNT coating, as illustrated in Fig. 6.

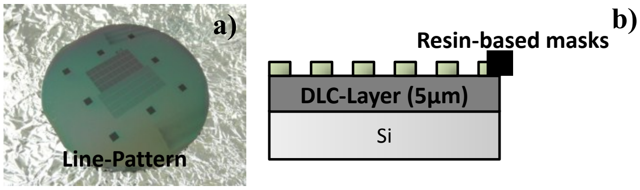

Fig. 5: DLC- coated silicon substrate with the resin-type two dimensional masking; a) Outlook of specimen, and, b) Cross-sectional illustration of specimen.

Fig. 6: CNT- coated silicon substrate before metallic masking.

E. PREPARATION OF DLC-COATED MOLD-DIE

DLC-coated steel die with micro-grid pattern was prepared to fabricate the mother die for micro-embossing. As shown in Fig. 7, AISI420 martensitic stainless steel was employed as a substrate material. DLC film had a multilayered structure; chromium and amorphous SiC films (or a-SiC) were undercoat to be free from over-etching. The mask was made of SiO_2 and a-SiC thin films.

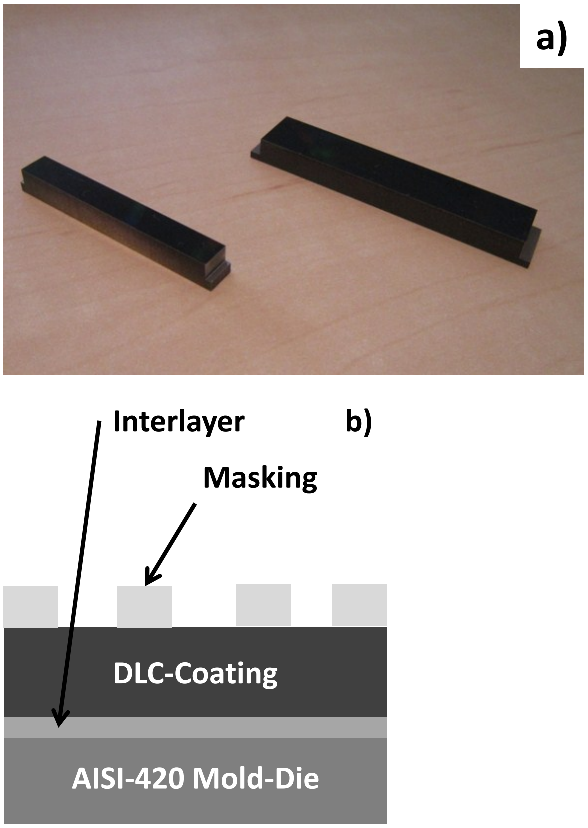

Fig. 7: DLC-coated AISI420 mold-die with initial, two-dimensional masking. a) Outlook of mother tool materials with masking, and, b) Cross-sectional illustration of mold-dies.

F. TABLE-TOP CNC-STAMPING SYSTEM

Table-top CNC (Computer-Numerical-Control) cold stamping system with the capacity of 200 kN, was developed in order to establish the micro-embossing line in mass production. This system worked on the table top; temporal variation of processing parameters was directly controlled besides data acquisition. Both stroke and load were digitally monitored by load cell and linear scale, respectively. Figure 8 illustrated the stamping operation by using the proto-type, CNC cold stamping system. The stroke range was designed to be wide for various deformation modes in micro-embossing.

A cassette mold-die unit was placed between upper and lower bolster plates; actually working mold-die pair was set-up in this cassette mold-die unit. In parallel with precise positioning control, both the loading and displacement rates were also controllable; then, loading and stroke sequence were both programmed to be run for optimum sequential control to each experimental set-up.

To be noticed, the CNC feeding system of metallic sheets was attached to the above mold-stamping system, not only to control the movement of work material in backward and forward but also to make tension control of thin sheet material for suppression of wrinkling.

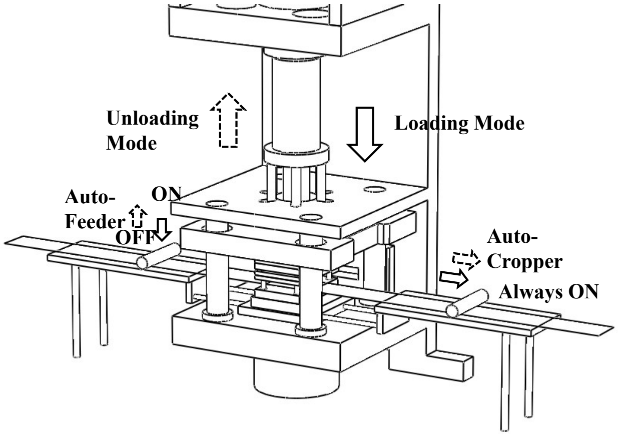

Fig. 8: Table-top CNC-stamping system for duplication of micro-textures onto aluminum sheet via micro-embossing process.

G. OBSERVATION AND MEASUREMENT

SEM (Scanning Electron Microscope), LM (Laser microscope) and LRP (Laser Reflection Profilometer) were utilized to make observation and measurement of imprinted micro-structures on the DLC and CNT films and on the aluminum sheets.

EXPERIMENTAL RESULTS

A. PLASMA STATE FOR ETCHING

Quantitative plasma diagnosis becomes an effective means to investigate the optimum processing conditions for the present plasma etching. Through the spectroscopic analysis of emissive light from oxygen plasmas, the state of activated species is analyzed to optimize the plasma processing parameters. As had been reported in the previous studies [12-14], the activated oxygen species were classified into O_2^+, O* and O (j) for J = III, III, and IV (or O^+, O^{2+}, and O^{3+}). These population distributions were strongly dependent on the RF/DC conditions and plasma pressure. Activation into oxygen ions and radicals was accompanied with increase of electron density.

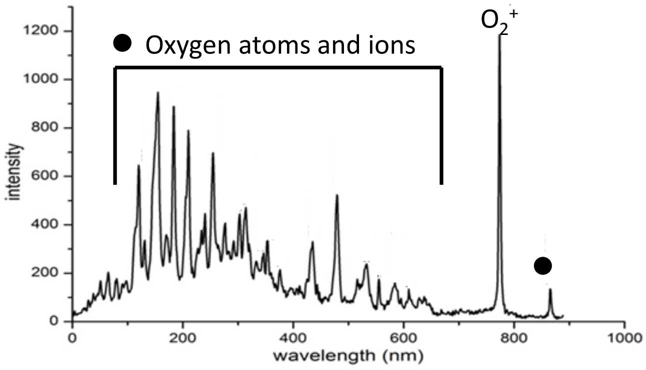

Fig. 9: Measured spectrum of activated oxygen species in the plasmas.

Figure 9 showed a typical emissive light spectrum of the fully developing plasma state. Besides a single, significant peak of O^{2+}, almost all the peaks came from the activated oxygen atoms and ions. This is because of cascading electron detachment reactions in series where $O^{2+} + 2e \rightarrow 2O^*$, $O^* \rightarrow O^+ + e$, $O^+ \rightarrow O^{2+} + e$, and, $O^{2+} \rightarrow O^{3+} + e$. Hence, huge amount of electrons is expected to be present with these activated species of O* and $\{O^+, O^{2+}, O^{3+}\}$.

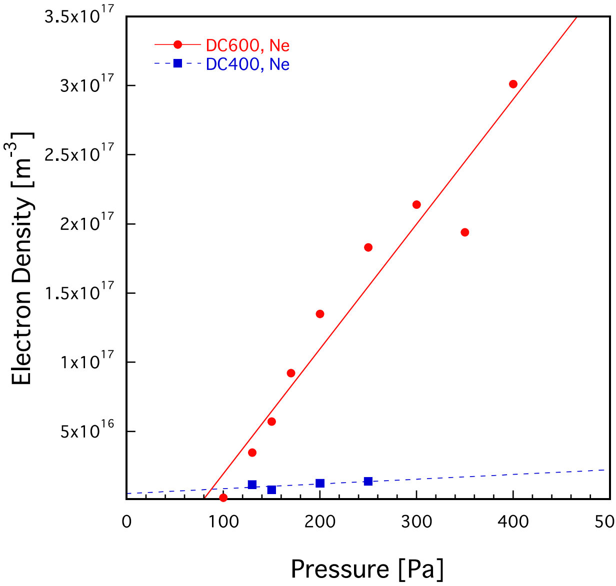

Fig. 10: Variation of electron density with increasing the plasma pressure.

Figure 8 showed the variation of electron density (Ne) with the plasma pressure for two DC bias conditions. The electron density monotonically increases with the plasma pressure or with increasing the oxygen molecule concentration. This corresponds to monotonic increase of ion density with pressure. It is to be noticed that the above increasing behavior should be affected by the DC bias; e.g. Ne > 5.0×10^{16} /m^3 when the bias voltage (V_B) is -600 V while Ne = 1.0×10^{16} /m^3 at V_B = -400V. This nonlinear dependency of electron density on the DC-bias suggests that plasma parameters must be optimally selected to control the oxygen etching process.

B. ETCHING BEHAVIOR OF DLC COATING

DLC-coated silicon substrate was used as a test specimen for oxygen plasma etching. Two dimensional line-patterns were imprinted onto the surface of DLC coating as a masking pattern for various line widths from 100 μm down to 5 μm. The oxygen plasma processing parameters were fixed in what follows: RF-voltage was 250 V, DC-bias, -600 V, plasma pressure, 40 Pa and the processing time, 2000s. Figure 11 a) depicted the top view of etched specimen by LM. Corresponding to the original line patterns, micro-grooves were successfully etched into DLC-coating.

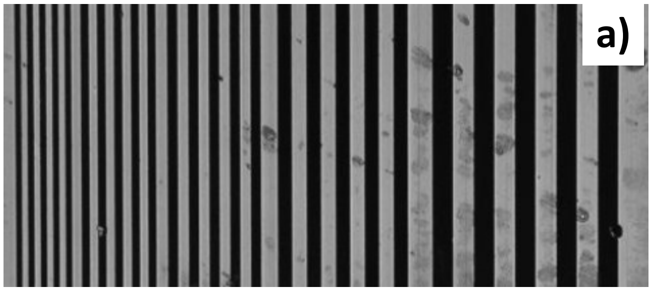

Figure 11 a): micro-grooves etched onto the DLC-coating on the silicon substrate.

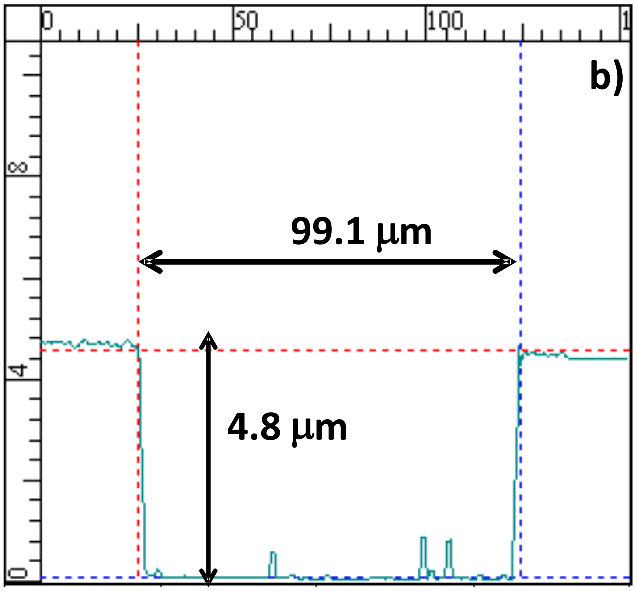

Fig. 11 b): Depth profile of micro-groove with $W_G = 100$ μm.

Figure 11 b) showed the depth profile of a single micro-groove with the width (W_G) of 100 μm. The measured micro-groove width was 99.1 μm and its depth, 4.8 μm, respectively. Both were in fairy good agreement with the initial line width of 100 μm and the DLC film thickness of 5 μm. It was noticed that the depth profile of this micro-groove should be shaped in stepwise to have steep side walls. This well-defined micro-grooving in etching was common to the previous results [7-8] where metallic masks were used as an original pattern. In those reports, the depth profile of narrower micro-grooves than 3 to 5 μm was deteriorated by over-etching; the top of masks was etched and roughened, and, both its side walls and bottom surface were also etched away in part. Let us reconsider this dependency of etching behavior on the line-width of original masking patterns.

Figure 11 c) depicted the SEM micrograph on the cross-sectional view of narrow micro-grooves with the skew angle of 30 degrees. Although slightly over-etched regions were seen on the side walls, narrow micro-grooving patterns were etched in stepwise to have the same line-width and line-pitch as the original masks without deterioration of resin-base masks. This proves that the present resin-base masking technique should be suitable to oxygen plasma etching with sufficient accuracy even to micro-patterning in the order of μm range. Over-etching behavior seen on the side walls could be suppressed by controlling the plasma processing parameters to be discussed in later.

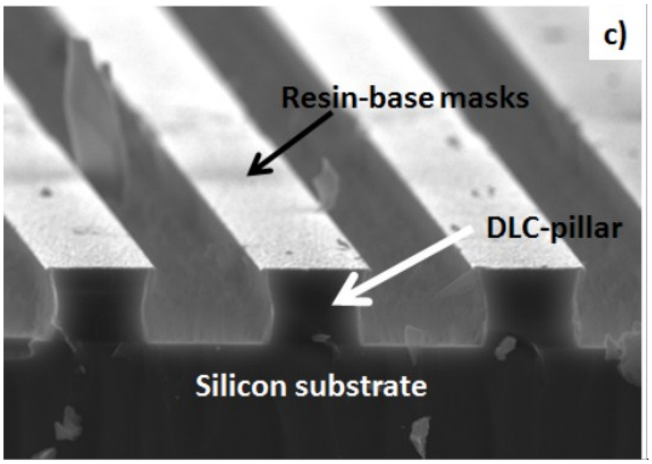

Fig. 11 c): SEM micrograph of narrow micro-grooves.

C. ETCHING BEHAVIOR OF CNT COATING

Diamond-like carbon coating has an amorphous structure or nano-composite of graphitic planar nano-structure (sp2) and diamond-like tetragonal nano-structure (sp3). Besides DLC materials, carbon has much capability to form other nano-structural configurations in practice. Let us employ the carbon nano-tube specimen in order to consider the effect of carbon nano-structure on the micro-etching behavior via the present oxygen plasma etching. In the similar way to the previous reports [8-9], the metallic mask with the line pattern was glued onto the CNT-coated silicon substrate. The line width was constant by 100 μm.

Figure 12 depicted SEM micrograph of etched CNT coated silicon substrate specimen. The pitch between two micro-grooves was preserved to have the same distance as the line-pitch. However, a micro-groove turned to be dull, round groove. This might be partially because the oxygen flux diffuses along the interface between metallic mask and CNT coating; this leakage of oxygen radicals resulted in dullness in micro-grooving. To be discussed in later, anisotropic etching capacity via oxygen plasma etching might be different between DLC and CNT coatings; the etching process could be more isotropic in case of CNT coating. Furthermore, the vertically aligned nano-structure of CNT had influence on the etching behavior. In fact, comparison between Figs. 11 c) for DLC coating and Fig. 12 for CNT coating revealed that more thick CNT film should be etched away even after the same duration time as etching the DLC coating.

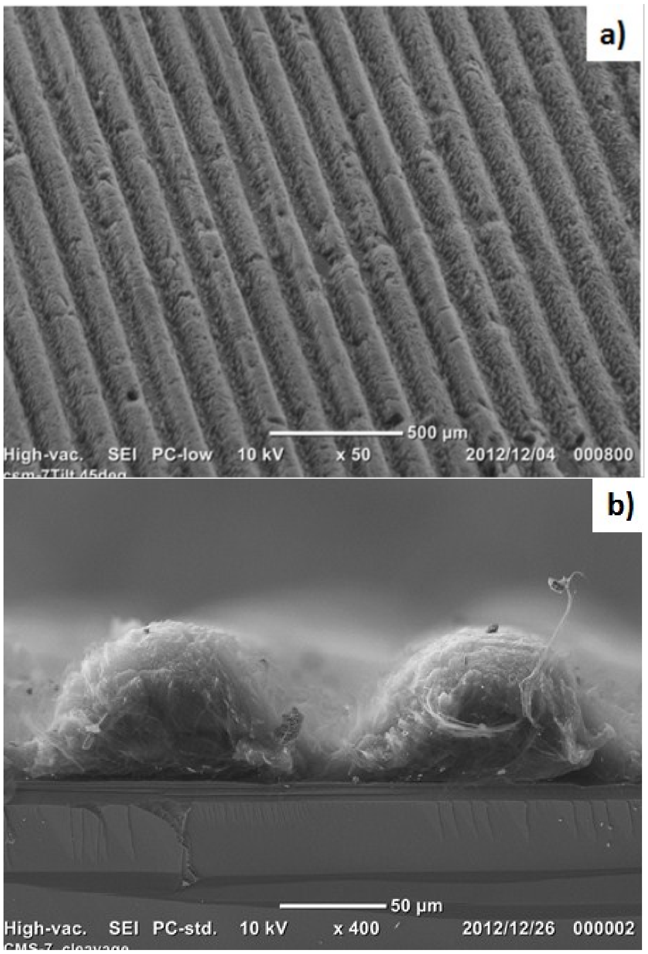

Fig. 12: Micro-patterning onto CNT coating. a) SEM micrograph of etched micro-patterns, and, b) Cross-sectional view of etched micro-patterns.

D. FABRICATION OF MOLD-DIE FOR MICRO-EMBOSSING

DLC-coated AISI420 substrate was employed as a mold-die for micro-embossing as shown in Fig. 7. A resin-type masking with a square-dot pattern was photo-etched onto the DLC coating as the initial mother pattern. Owing to this masking, a square unit-cell was preserved to be in the masked state, and, un-masked grid-lines were selectively etched into the depth of DLC coating. This square unit-cell had around 4 x 3 μm^2; after etching, this two dimensional unit-cell turned to be a rectangular cylinder and the whole patterned surface of AISI420 substrate, to have micro-punches with the area of 10 x 50 mm^2.

Figure 13 depicted the SEM micrograph on the surface of etched DLC-coated AISI420 die. Rectangular cylinders with its top of 4 x 3 μm^2 and its depth of 5 μm were aligned with the same pitch. In the following micro-stamping, this mother die worked as the upper die to make micro-embossing.

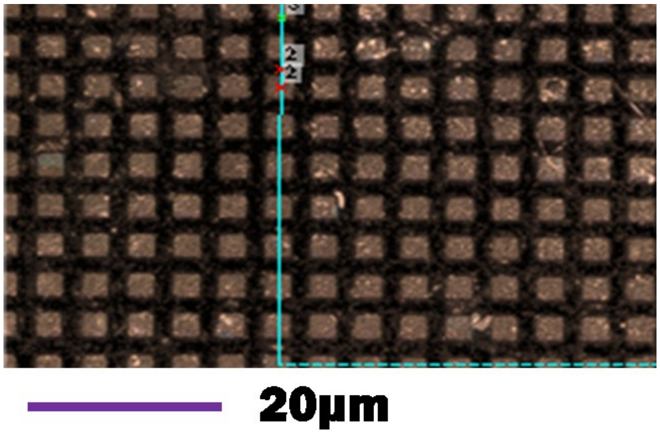

Fig. 13: Micro-textured, DLC-coated mold-die for micro-embossing.

DISCUSSION

A. COMPARISON OF ETCHING RATE

The measured depth of micro-grooves in Fig. 11 divided by the processing time provides us the average etching rate both for DLC and CNT coatings. Table 2 compared both the etching rates (or T_{DLC} and T_{CNT}) at the bias voltage of 400 V and 500 V, respectively. In case of DLC coating, T_{DLC} was about 1.6 nm/s, or 5.8 μm/H; while T_{CNT} reached about 25 nm/s or 90 μm/H in case of CNT coating. This difference might be attributed to microstructures both in DLC and CNT coatings.

Table 2: Comparison of the oxygen plasma etching rate between DLC and CNT films

DC bias	Rate of DLC	Rate of CNT
400 [V]	1.57 [nm/s]	24.09 [nm/s]
500 [V]	1.642 [nm/s]	25.981 [nm/s]

DLC is an amorphous carbon where tetragonal cluster of carbon (or sp3 substructure) is mixed with planar carbon cluster (or sp2 substructure) and hydrogen atoms. Oxygen atom diffusion path is narrowed by the network of these sp2 – sp3 substructures. This retardation in oxygen atom diffusion results in slow etching rate. On the other hand, the vertically aligned CNT film to the substrate surface has much lower density than DLC film; oxygen atoms and ions easily diffuse into the depth of CNT coating along the carbon-network walls. This smooth diffusion path of oxygen atoms accelerates the etching rate of CNT coating.

B. MICRO-ETCHING BEHAVIOR

A shown in Fig. 11, the present oxygen plasma etching has practical feasibility to micro-etching in the order of micro-meters. Since the geometric accuracy is strongly dependent on the anisotropic etching behavior, the effect of unit-size in the micro-patterns on the accurate etching must be studied by experiments. Micro-grooving etching was employed here to investigate the effect of micro-groove width on the reactive ion etching behavior.

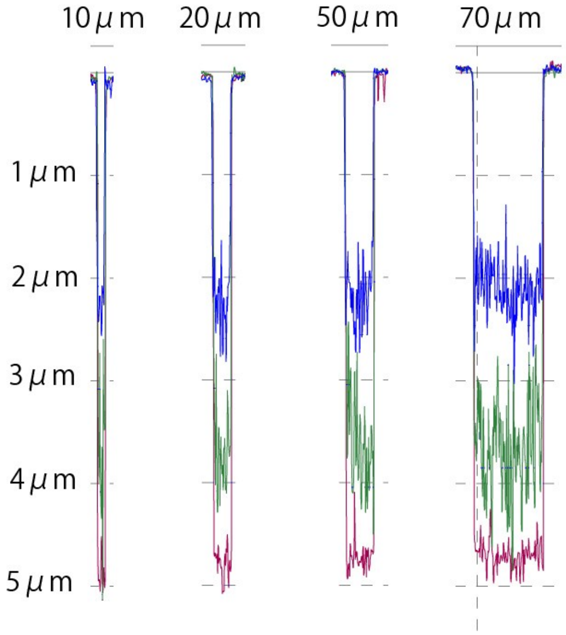

Fig. 14: Time evolution of depth profiles during oxygen plasma etching,

When the micro-groove width (W_G) was 70 μm, the etching front profile was uniform so that the etching rate at the top-front in the anisotropic etching should be nearly equal to the average etching rate. With decreasing W_G down to 10 μm or less than, the top etching front was rather insensitive to W_G. As stated before, however, the average etching rate decreased with W_G. This difference between macroscopic and microscopic etching behavior might be attributed to local change of oxygen pressure in the etching process. With decreasing W_G, the partial pressure of oxygen flux in the micro-groove channel could be reduced so that the etching rate at the front should be constant but the average etching rate should be retarded.

C. MICRO-EMBOSSING BEHAVIOR

CNC-pressing system was utilized to duplicate the micro-textures on the DLC-coated mold-die onto the aluminum sheet with the purity of 99.94 %, the average grain size of 8 μm, and the thickness of 80 μm. As illustrated in Fig. 8, the aluminum sheet was transferred and fed to the stamping position; then, the micro-textured die was upset onto the aluminum sheet and loaded for duplication of micro-textures. With aid of minimum quantity of dry lubricating oils, the stamped aluminum sheet under tension control was easily demolded and transferred for next stamping operation. A typical continuously stamped aluminum sheet was shown in Fig. 15.

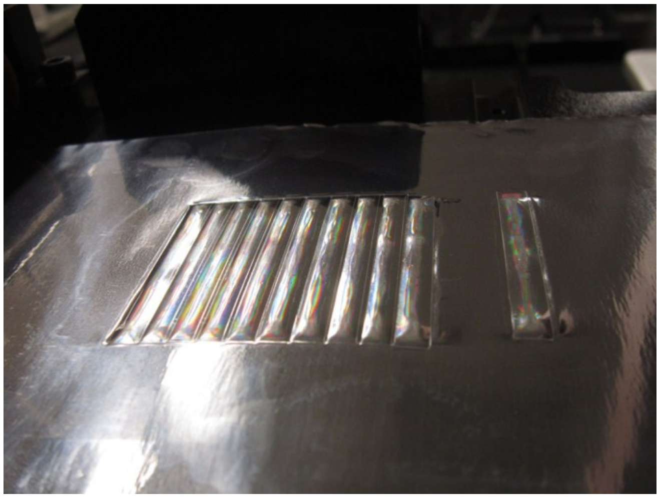

Fig. 15:Progressive stamping of micro-textured aluminum sheets by using the table-top CNC-controlled stamping system.

Figure 15 depicted a micro-embossed aluminum sheet. Original micro-textures on the DLC-coated mold-die were successfully imprinted onto the area of 10 x 50 mm^2 by one single stamping shot. During the unloading step, the work sheet was moved by automatic feeding control in Fig. 8; large area product was fabricated in the progressive manner. No wrinkling was seen on the surface of micro-textured aluminum even after ten stamping shots. Rainbow-shining in the micro-embossed are in Fig. 15 was attributed to surface plasmon, trapped in the micro-textures.

Laser microscope was utilized to measure each micro-grid pattern at the center and at the edge of the micro-embossed aluminum sheet. As shown in Fig. 16, no significant difference in the geometry and dimension was noticed between two images; that is, homogeneous imprinting took place by using this micro-embossing process. In both cases, the concave micro-grid patterns were homogeneously imprinted onto aluminum, corresponding to the original convex micro-textures of rectangular DLC cylinders in Fig. 13.

CONCLUSION

High density oxygen plasma etching provides us an efficient tool to make precise etching onto both DLC-coated and CNT-coated substrates. With aid of quantitative plasma diagnosis, the plasma state in etching is insitu monitored to describe the etching behavior. The etched DLC coating has high loading ratio or aspect ratio of depth to width in micro-textures. In addition, their sharp edge corner is preferable to the mother tool to make micro- or nano-imprinting onto metallic or plastic sheets. Table-top CNC pressing system is successfully applied to duplicate the original micro-patterns on the DLC coating onto the pure aluminum sheet with sufficient accuracy. This success proves that micro-patterned metallic sheets should be fabricated in mass production by using the present approach.

Micro-patterning onto fragile CNT coating requires for sophisticate treatment other than metallic masking to prepare for masking before the present oxygen plasma etching. With aid of new masking technique, micro-texturing of CNT coating might well be put into practice because of its high etching rate.

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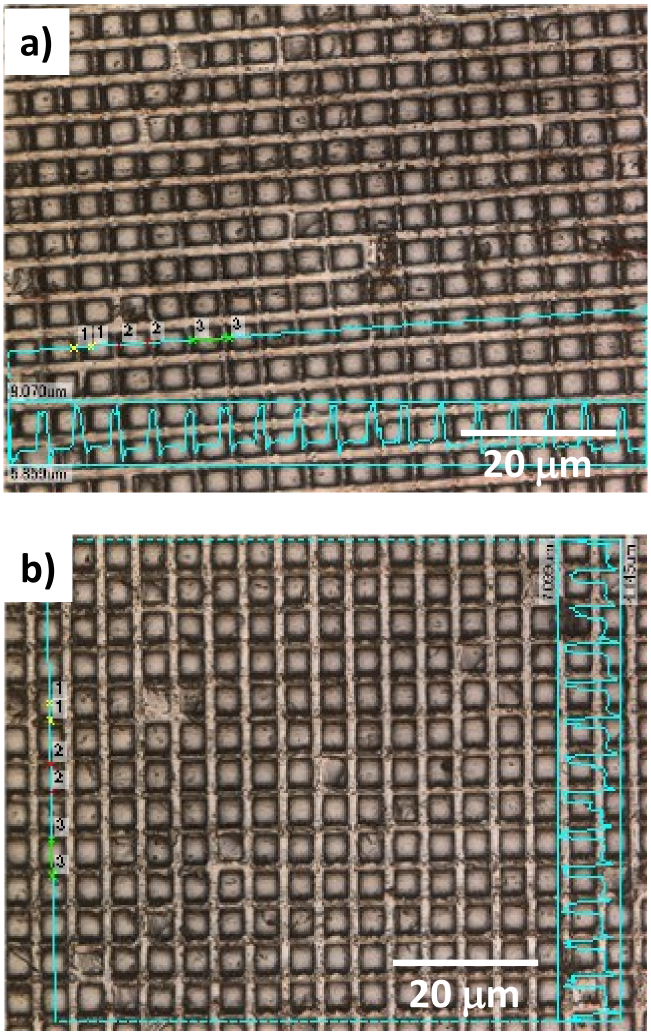

Fig. 16: Micro-textures imprinted onto the aluminum sheet by CNC-pressing. a) Micro-textures measured at the center of micro-embossed area by 10 x 50 mm^2, and, b) Micro-textures at the edge of micro-embossed area by 10 x 50 mm^2.

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