DOI: 10.1007/s12541-014-0630-4

Micro/Nano-Mechanical Structure Fabricated by Transfer **Printing**

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KEYWORDS: Mechanical structure, Transfer printing, MEMS, Thin-film

A fabrication of micro-electro-mechanical systems (MEMS) generally involves complicated conventional semiconductor processes. Simply micro/nano-structuring process is a technical issue on further wide range of MEMS application. This study proposes to apply transfer printing to fabricate a mechanical structure for MEMS elements. Some metals (Au, Cu and Ni) are previously deposited to be thin-films on a micro-ridged poly-dimethyl-siloxane (PDMS) stamp, while a micro-grooved SU-8 is prepared as a substrate. Contact-and-release process allows the metal thin-film to be locally transferred from the micro-ridges of stamp to the micro-grooved substrate. The transferred Au and Ni thin-films successfully form to be an ultra-thin fixed beam, "cross-linked structure", of which the thickness is less than 100 nm. The fabricated structure is applicable to a nano-scale mechanical oscillator. The production yield of fixed beam increases with thin-film thickness and highly-dense of thin-film. The micro-roughness and hydrophobicity of stamp enhance the production yield of ultrathin fixed beam. These experimental results clearly indicate that the proposed process is dominated by an adhesion force between the stamp and the thin-film as well as the mechanical strength of the thin-film. Nevertheless, it is confirmed the transfer printing process is effective for a fabrication of MEMS element.

Manuscript received: January 31, 2014 / Revised: August 20, 2014 / Accepted: September 5, 2014

1. Introduction

Some micro-electro-mechanical system (MEMS) integrates micro/ nano-scale mechanical elements with electronic circuits for unique sensors, actuators, telecommunication devices, and biochemical analytical systems. A fabrication of mechanical element in MEMS generally involves some complicated semiconductor processes such as sacrificial etching and high-temperature thin-film growth. It restricts type of structural and/or substrate materials because of etching selectivity and heat resistance, so that silicon and glass have been still utilized in most micro-systems. An application of soft material (polymer) to substrate, however, has great interest for novel MEMS application such as bio-medical devices. Development of micro/nanofabrication applicable to soft material is an important technical issue to expand the range of MEMS applications.

Micro/nano-transfer printing has been developed to fabricate spatially patterned thin-film on a substrate.¹ Fig. 1 shows a schematic illustration of transfer printing. In this process, a thin-film of metal, semiconductor, organic molecules, or nano-materials, which is previously covered on a micro-structured stamp, is transferred from the stamp to a substrate. The transferred thin-film forms into a designed two-dimensional pattern. It can be also performed under room temperature and atmospheric environment, so that various materials including soft material are utilized for a structure and a substrate. A transferring mechanism is based on a difference in adhesion force between thin-film, stamp, and substrate in terms of applied force.^{2,3} When adhesion force between thin-film and substrate is higher than that between film and stamp, the thin-film can be transferred to the substrate surface. It means that low adhesion force is preferable for stamp surface. Adhesion force is dominated by surface energy, so that low surface energy material such as poly-dimethyl-siloxane (PDMS) is mainly utilized as a stamp.⁴ With regard to micro/nano-pattern of metal thin-film and its application by transfer printing, many test results have been reported as follows. Law et al fabricated a line pattern of Au with the range from $1 \mu m$ to $10 \mu m$ width, so that its polarization property was confirmed.⁵ Kang et al. located a nano-wire mesh of Cu to demonstrate transparent electrodes.⁶

As described above, these previous works utilized transfer printings for two-dimensional patterning with a focus on an application to electronic devices.⁷ We have proposed fabrications of three-dimensional

nano/micro-mechanical structure using transfer printing. Fig. 2(a) shows schematic illustration of the proposed process, in which micro-beams of thin-film are formed on a substrate. A substrate is previously structured to have an array of micro-ridges by another process such as nano-imprint. A stamp coated with thin-film is contacted to the structured substrate, while the line of thin-film is oriented in a direction perpendicular to the micro-ridges. After the stamp is released from the substrate, the thin-film is transferred and supported on the substrate surface to form a bridge between the micro-ridges. Namely, it produces an array of micro-beams. These micro-beams can be applied to a micro-mechanical sensor to detect specifically-adsorbed gas molecules. Furthermore, it is expected that the transfer printing to structured substrate also enables us to fabricate a multi-functional micro-device, in which some mechanical parts (micro-beam, cantilever, membrane, wire) made of various materials are highly integrated with electronic circuits on a flexible film.

Unlike in the case of two-dimensional patterning shown in Fig. 1, this micro-beam fabrication applying transfer printing to threedimensional substrate is strongly affected by mechanical strength and thickness of thin-film as well as adhesion forces of each interface. An applied in-plane tensile stress is expected in the transfer printing as shown in Fig. 2(b), because the thin-film is partially bonded to the substrate surface. This applied tensile stress may cause undesirable deformation and/or fracture of transferred thin-film. This present paper intends to demonstrate fabrications of micro-beams by the proposed process based on transfer printing, and then shows some investigations about effects of film material, thickness, and surface properties on the process.

2. Experimental

A stamp was fabricated by the following process. This present work utilized PDMS (Slygard 184, Dow-corning) and h-PDMS (VDT-731, Gelest) for stamp material because of its moldability, low surface energy, and mechanical strength. The compression modulus of PDMS and h-PDMS are nominally 2.0 and 9.0 MPa, respectively.⁸ Thick-film photo-resist (SU-8, Micro Chem), which was spin-coated on a Si wafer, was grooved by photo-lithography for a master mold.

The master mold has an array of micro-grooves with 10 or 50 μ m widths. The pre-polymer of PDMS or h-PDMS were mixed with curing agent. The mixture was filled into the master mold, and then thermally cured at 343 K for 70 min in a vacuum oven. Fig. 3(a) shows SEM (VE-9800, Keyence) image of demolded PDMS. The PDMS stamps had an array of micro-ridges, of which the width and height were 10 or 50 μ m and 50 μ m, respectively. The top surface of fabricated micro-ridges was smooth with nano-scale roughness. The surface of PDMS was hydrophobic, where water contact angle (WCA) of the PDMS was typically 107°. Higher WCA of surface is expected to have low adhesion force and surface energy.⁹ The stamp size was typically 1×1 cm.

The fabricated stamps were subsequently coated with metal thinfilms (Au, Ni, or Cu) by vapor-deposition or ion sputtering. These metal thin-films have different yield strength, modulus, and WCA. For example, the yield strengths of Au, Cu and Ni thin-film were reported to be 309, 224, and 770 MPa by some nano-indentation methods.¹⁰⁻¹²

Fig. 1 Transfer printing for two-dimensional pattern

Fig. 2 Schematic illustrations of proposed technique

Fig. 3 Examples of fabricated stamp and substrate

Fig. 4 Transfer printing set-up

In this study, WCA of Au, Cu and Ni thin-films were measured to be 40°, 17° and 6°, respectively. The thicknesses of thin-film were in the range of 30 nm to 140 nm. We employed another SU-8 micro-structured film as a substrate of soft material. The young's modulus and softening point of SU-8 are nominally 2 GPa and 383 K, respectively. The SU-8 substrates were also grooved by photo-lithography. Fig. 3(b) shows SEM image of typical SU-8 substrate. The groove width was 10, 50, or 100 µm. These grooves were arranged on the whole area of the substrate, which size was typically 1×1 cm.

Fig. 4 shows our transfer printing set-up. A substrate is located on a mechanical Z-stage, while a stamp is fixed to the opposite part installed with a force sensor. The contact pressure between the substrate and stamp is measured by a force sensor. The substrate temperature is heated by a ceramic heater. Typical transfer printing process is as follows. The substrate temperature was kept at 423 K due to the softening point of the SU-8. The substrate was moved in vertical direction, and then contacted to the stamp coated with metal thin-film. The contact pressure was adjusted at the range of 1.5 to 3.2 MPa for 30 min. After the substrate was cooled and kept at room temperature for 300 s with unloaded condition, the substrate was released from the stamp.

3. Results and Discussions

3.1 Fabrication of ultra-thin micro-beam

This section describes some demonstrations of fabricating microbeam by the transfer printing. In these experiments, the h-PDMS stamp was coated with 70-nm-thickness of Au thin-films by ion sputtering. The contact pressure was 3.2 MPa. Fig. 5 shows SEM (ERA-9000, ELIONIX) images of the substrate surface after the process. Straight belt-like structures were located across the micro-ridges of the SU-8 substrate. Parts of the structures were completely isolated to the substrate on the grooves, so that it successfully to be bridged between the micro-ridges. This result indicates that the Au thin-film was successfully transferred to form into micro-beams. These micro-beams were arranged on the area of several square millimeters. The fabricated micro-beam was also very thin. The thickness was estimated to less than 100 nm, which was almost same as that of deposited thin-film on the stamp before the process. The micro-ridges of stamp had no residual materials at the opposite face to micro-beams, so that the Au thin-film was processed without delamination. The fabricated micro-beams, however, were slightly deformed upward, although they had little cracks. Fig. 6 shows a surface profile of micro-beam observed by Atomic Force Microscope (VN-8000, Keyence). The maximum deflection of micro-beam was estimated to be approximately 2.5 µm. This result indicates a plastic deformation induced by tensile stress shown in Fig. 2(b). It is considered that a decrease in adhesion force between thin-film and stamp inhibits undesirable tensile stress to cause such a deformation. Nevertheless, it is demonstrated that the proposed process allows us to fabricate an array of ultra-thin micro-beams without any complicated sacrificial etching.

We also processed 10-µm-wide stamp to form narrower microbeams. In this experiment, the PDMS stamp was coated with Au thinfilm of 100 nm thickness by vapor-deposition, while the SU-8 substrate had 50-µm-wide micro-ridges. Fig. 7 shows SEM image of the substrate after the process. The Au thin-film was also formed into a fixed microbeam. Both edge sides were not straight but irregularly serrated, as they were partially peeled from the micro-ridges. This geometry was mainly caused by inhomogeneous contact pressure, insufficient adhesion force, and undesirable film-coating on stamp side-walls, because it had smaller contact area than that of case shown in Fig. 5. This form accuracy, however, can be improved with adjustments of surface conditions and accurate stamp. It is considered that this process is applicable to fabricating thinner micro-beam with several micrometers width.

3.2 Effect of film thickness and material

This section describes investigations about effects of thin-film

Fig. 5 SEM images of fabricated micro-scale fixed beam

Fig. 6 Cross-sectional view of ultra-thin micro-beam

Fig. 7 SEM image of 10-µm-wide micro-beam

thickness and material properties on micro-beam fabrication. This present study employed three kinds of materials, Au, Ni, and Cu, as a transferred thin-film. As described in section 2, they have different mechanical strength and surface property. Au, Ni, and Cu thin-films were fabricated on the same type of PDMS stamps with 50-µm-wide micro-ridges by vapor-deposition. The thickness of thin-films was the range from 30 to 140 nm. Unlike in the case of previous section and Fig. 5, prepared substrates had 10-µm-wide micro-ridges. The contact pressure and substrate temperature were 1.5 MPa and 423 K, respectively.

Figs. 8(a) and (b) show SEM images of the substrate surface after the transfer printing of Ni and Cu thin-films. These thin-films could be bridged between the micro-ridges of substrate. The transferred Ni and Cu thin-films, however, had many cracks and deformations to cause fractures of micro-beams unlike sputtered Au shown in Fig. 5. It is considered that excess contact pressure and adhesion force resulted in these defects and deformations, as transfer printing conditions were not optimized for these material and thickness of thin-film. This present study defined production yields of micro-beams to quantitatively evaluate effects of thin-film material and thickness. Production yield is the rate of successfully fabricated micro-beams to the transferred sites on the same substrate. Fig. 8(c) shows the relationship between thinfilm thickness and production yield. All materials of thin-films had an increasing tendency of production yield with film thickness. It is considered that an increase of thin-film thickness made applied tensile stress relatively decreased. The production yields of Au and Ni thin-

Fig. 8 SEM images and production yield of fabricated micro-beams using Au, Ni, and Cu thin-films

film, however, were remarkably high as compared to that of Cu thinfilm. The production yields of Au and Ni thin-films reached to 100 % at 140 nm and 100 nm thickness, respectively, while that of Cu thinfilm was no more than 10 % at 140 nm thickness. It was found that the micro-beams fabrication had critical film thickness in the experiment. The critical thickness is related with an applied tensile stress during the releasing step of stamp.

These experimental results were partly consistent with the mechanical strength of thin-film, because there were a few differences in the value of reported yield strength between Au and Cu thin-films. It can be explained by adhesion force between the thin-films and the stamp, because an applied tensile stress was expected to be increased with the adhesion forces. As described above, water contact angle (WCA) is considered to be an index for adhesion force and surface energy. The WCA of Au, Cu and Ni thin-films were measured to be 40°, 17° and 6°, respectively. The Au thin-film has the lowest surface energy, so that it allows the Au thin-film to be easily peeled from the stamp with considerably small tensile stress. Meanwhile, higher surface energy of Ni and Cu thin-films caused strongly adhesion to the stamp. It resulted in larger tensile stress to fracture and deform the thin-film in the transfer printing. These experiments summarized that transfer printing for microbeam fabrication requires enough mechanical strength or thickness of thin-film and low adhesion force between stamp and thin-film.

3.3 Effect of thin-film property

This section describes effects of thin-film properties on micro-beam fabrication. The properties (roughness, strength) of thin-film depend on deposition method and conditions. This present study coated two kinds of Au thin-films on PDMS stamps by vapor-deposition or ion sputtering. The thickness was 70 nm. The authors experimented two-dimensional transfer printings on a flat SU-8 substrate to investigate a difference in transferring applicability between two Au thin-films, and then applied two kinds of Au thin-film to transfer-printing on a micro-structured substrate.

Fig. 9 Relationship between production yield and pressure in twodimensional transfer printing of Au thin-film on a flat SU-8 surface

Fig. 10 SEM images of thin-film surfaces transferred from the stamp

Firstly, the results of two-dimensional patterning were described as follows. The authors prepared a flat SU-8 substrate without micro-ridges. Meanwhile, the stamp had an array of 50-µm-wide micro-ridges, which was coated with vapor-deposited or sputtered Au thin-film. In this experiment, the contact pressure varied with the range of 150 Pa to 1 MPa. The transfer printing made the Au thin-films patterned into an array of lines on the flat SU-8 surface. Some Au thin-films were partially transferred with fractures onto the substrate in some conditions, as the substrates had incomplete and discontinuous patterns. Fig. 9 shows rate of transfer and typical SEM of fabricated line-patterns of Au on the flat SU-8 surface. Rate of transfer is defined as the area of transferred thinfilm divided by the contacted area of micro-ridge of stamp. In the case of vapor-deposition, the Au thin-films were almost completely transferred on the substrate for all contact pressures. Meanwhile, in the case of sputtered Au thin-film, the rate of transfer increased with contact pressure. It reached to 100%, when contact pressure was more than 1 MPa. This difference in rate of transfer was attributed to surface property of thin-film. Fig. 10 shows SEM images of thin-film surfaces coated by vapor deposition and ion sputtering. In the coating conditions, the surface of vapor-deposited film had dense and coalescent islands to roughen surface, while the sputtered film was very smooth. These surface morphologies were attributed to growth mode of thin-film, which was three-dimensional (3D) nucleation growth or two-dimensional growth. The growth of 3D nucleation suggests that the stamp surface had poor wettability for Au thin-film to generate small adhesion force. Therefore, the thin-film coated by vapor-deposition could be easily peeled and transferred at a lower contact pressure in the experiments. Meanwhile, the sputtered Au thin-film needed larger contact pressure to increase real contact area between the thin-film and the substrate, because it made the thin-film have larger adhesion force to the substrate. The

Fig. 11 Production yield of micro-beams fabricated by transfer printing of vapor-deposited and sputtered Au thin-films onto micro-ridges of substrate

mechanical strength of vapor-deposited thin-film, however, was lower than that of sputtered thin-film owing to its homogeneity.

As described above, the surface morphology of thin-film strongly affected the transferring accuracy for two-dimensional transfer printing. Two type Au thin-films were also applied to transfer printings on a micro-structured substrate, which had 10-um-wide micro-ridges. These Au thin-films were previously deposited on the stamp with $50-\mu m$ wide micro-ridges. The thickness of thin-film was 70 nm. The contact pressure was 1.5 MPa for the vapor-deposited film or 3.2 MPa for the sputtered film. For both vapor-deposited and sputtered thin-films, it was found that some micro-beams were bridged between the microridges of substrate similar to that shown in Fig. 6. There, however, was a remarkable difference in the number of fabricated micro-beams. Fig. 11 shows their production yields. The production yield of sputtered film was much higher than that of vapor-deposited film. This result was attributed to thin-film homogeneity shown in Fig. 10. The vapordeposited thin-film consisted of nano-scale grains (islands) and boundaries. These grain boundaries decrease an allowable stress of thin-film to be strongly related with production yield. Therefore, this structure of vapor-deposited thin-film caused to be fractured under applied tensile stress by transfer printing, although it had effective function of peeling from the stamp shown in Fig. 9.

The above experimental results summarized that accurate fabrication of micro-beams requires the following specific thin-film properties. The surface structure should have lower adhesion force at an interface between the thin-film and the stamp, while the film structure also have homogeneity with high mechanical strength. The present study also processed transfer printing of bi-layered thin-film to achieve higher production yield with smaller contact pressure and thinner film thickness as shown in Fig. 12(a). The first layer was coated by vapordeposition to generate lower adhesion force induced by 3D nucleation. The second layer, which is the same material as the first layer, was coated by ion sputtering to improve film homogeneity. Both thickness of first layer and second layer were 50 nm, so that the total film thickness was 100 nm. The thin-film material was Au or Cu. The stamp and the substrate had 50-µm-wide and 10-µm-wide micro-ridges, respectively. The contact pressure was 1.5 MPa. Micro-beams of bi-layered thin-film were successfully fabricated in the same way as that of mono-layered thin-film as shown in Fig. 5. Fig. 12(b) shows their production yields of micro-beams for the bi-layered and the mono-layer thin-films. In both materials of thin-films, the production yield of bi-layered thin-film was greatly improved as compared that of mono-layer. Especially, in the case of Cu, the bi-layered thin-film might enhanced its mechanical

Fig. 12 Micro-beam fabrications by transfer printing of bi-layered thin-

strength to prevent its fracture, as one could hardly fabricate microbeams of mono-layer Cu thin-film regardless of transfer printing conditions as shown in Fig. 8. It is also considered that multi-layering allow micro-beam of thin-films to have various mechanical properties for specific MEMS applications.

3.4 Effect of surface wettability of stamp

film

To investigate an effect of stamp surface energy on a fabrication of micro-beams, we prepared some surface-modified h-PDMS stamps.

The h-PDMS stamps with 50-µm-wide micro-ridges were processed by plasma treatment (PDC-32G, Harrick Plasma) and air exposure. The h-PDMS stamps were exposed to air plasma, where the plasma treatment time was the range of 0 to 30 min. This process made the h-PDMS surface hydrophilic, while it also etched the surface to be rougher. Furthermore, the surface was gradually re-modified to be hydrophobic with air-exposure after plasma treatment, while the surface roughness (Ra) was unchanged. For example, the water contact angle (WCA) of h-PDMS previously treated by plasma was changed from 0 degree to 64 degrees after 3.5 hours of air-exposure. The authors prepared some surface-modified stamps with different WCA and Ra by changing both plasma treatment time and air-exposure time. The wettability (WCA) of h-PDMS could be adjusted to the range of less than 5 to 105 degrees, while the surface roughness was 21 nm or 127 nm Ra. Au thin-films were sputtered on these surface-modified h-PDMS stamps. The thinfilm thickness was 75 nm. Transfer printing process using the surfacemodified stamp was applied to the SU-8 substrates with 50-µm-wide micro-ridges at contact pressure of 3.2 MPa.

Fig. 13 shows SEM images and production yields. With larger WCA and roughness of stamp, it prevented plastic deformations and fractures of transferred thin-films (a). The production yield also increased with contact angle and surface roughness (c). As the larger contact angle (lower surface energy) helped the thin-film to be peeled from the stamp, it resulted in the higher production yield. This result also indicated that surface roughness of stamp was preferred for the transfer printing of Au thin-film. It is not reasonable from the perspective of contact area, because contact area is generally increased with surface roughness. Therefore, the authors also observed surface morphologies of the stamp and the transferred thin-film. Fig. 14(a) shows cross-sectional profiles of the thin-film and the stamp obtained from AFM observations. The surface roughness of thin-film was smaller than that of stamp, although the surface of thin-film had been contacted to the stamp before transfer

Fig. 13 Effects of wettability and surface roughness

(b) Model of contact condition between thin-film and stamp

Fig. 14 Surface roughness and model of adhesion

printing. It is reasonable to suppose that there were many small voids at the interface between the thin-film and the stamp as shown in Fig. 14(b). The real contact area decreased with the surface roughness due to the voids in this experiment. Therefore, the rougher surface with smaller contact area also made lower adhesion force to improve the production yield. These effects achieved good form accuracy and high production yield of micro-beam fabricated by transfer printing without multi-layering of thin-film. The formation of voids was attributed to the growth mode of Au on h-PDMS surface such as nucleation growth. It is required to find applicable range of surface roughness and/or spatial frequency of stamp to achieve desired transfer printing. They will be investigated by our future works.

4. Conclusions

This present study is concluded as follows.

- (1) It is demonstrated that an array of ultra-thin fixed beams can be fabricated by the process based on transfer printing.
- (2) The fabricated micro-beams have ultra-thin thickness less than 100 nm and several tens micrometer long.
- (3) It is clarified that production yield and form accuracy of microbeams is affected by surface and film properties.
- (4) Accurate fabrication of micro-beams can be achieved by multilayering of thin-film and surface modification of stamp.
- (5) It is confirmed that transfer printing is effective process as a MEMS fabrication process.

ACKNOWLEDGEMENT

The authors wish to thank Ms. Omata of ELIONIX INC for SEM works. Fruitful discussions with Mr. Murakami and Mr. Miyazaki of Tokyo Metropolitan University are greatly appreciated. This work was supported by the Japanese Grant-in-Aid for Scientific Research (C) (22560130) and JKA promotion funds from KEIRIN RACE (25-114).

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