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Nanoelectrode Lithography of Silicon surface by Brass Stamp

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Abstract:

The stamps used in the nanoelectrode lithography (NEL) process require conductive layer deposition, which makes them a bit expensive. This paper reports the feasibility of using brass materials as the conductive stamps for NEL to shorten the process step and reduce the production cost. In this paper, the fabrication of nanostructures on the brass stamp was performed on a single point diamond turning (SPDT) machine. Some burrs were formed during the machining process, that prohibit the stamps from achieving a homogeneous contact with the substrates. Introduction of a thin layer of polymer (PS-OH) on the silicon substrate showed an improvement in contact uniformity so as the oxidation. However, some areas of the substrate remained unoxidized as few of the burrs were quite large. The brass stamps could be advantageous as they show no degradation after many uses. Nevertheless, the issues of the burr formation and non-uniformity should be alleviated first to make these stamps appropriate to the NEL process.

Keywords: Nanolithography, Anodic oxidation, SPDT, Burrs, Polystyrene

1. Introduction

Surface modification by using local oxidation has become a promising method after the first use of this technique with a scanning tunneling microscope (STM) by Dagata

et al. [1]. In the last decade, the use of atomic force microscopes (AFMs) as an efficient tool for the oxidation lithography has opened the way to manufacture nanometer-sized devices on the various materials [2-5]. Because of the slow processing speed of AFM, a great number of studies have been carried out to modify these approaches using conductive stamps with multiple protrusions that facilitate the large-area fabrication [6-8]. This method is commonly known as nanoelectrode lithography (NEL).

One of the most important factors in the NEL system is the requirement of a conductive stamp that must allow the nanostructures fabrication on it. Previous studies show that the researchers have used several stamps with multiple protrusions in NEL experiments. T. Mühl et al. utilized a conductive stamp with few protrusions to demonstrate the feasibility of this technique [9]. Other researchers used digital versatile disc (DVD) coated with Au film evaporated in high vacuum [3, 7, 10, 11]. Albonetti et al. fabricated a conductive stamp using Au/cr-5 or platinum metals, which was coated on a polydimethylsiloxane (PDMS) with multiple protrusions [12]. Silicon materials (Si, SiC) have also been used as stamps where the surfaces were modified by electron beam lithography and an etching process [8, 13]. To make these patterned surfaces conductive, a Ni or Au layer was deposited on the surfaces.

However, the use of gold/platinum as a conductive layer of the stamp makes it a bit expensive. Other conductive materials such as brass can be useful as a stamp for the NEL process due to their high elastic modulus and high breaking strength. They can also reduce the process steps such as deposition of the conductive layer. Brass is a copper-zinc alloy, with copper (60 - 63%), zinc (35.5%), Fe (max 0.35%), Pb (2.5 - 3.7%) and other components (max 0.5%). Good corrosion & wear resistance and good electrical & thermal conductivity make brass materials suitable for electric and electronic industries. In this article, we examined the feasibility of brass materials as a conductive stamp for the NEL process. It is also shown how the oxidation is improved by placing the polystyrene (PS-OH) layer on the sample surface.

2. Experimental Details

2.1 Fabrication of the brass stamp

Single-point diamond turning (SPDT) has been exploited to fabricate the structures on the brass surfaces. The machining processes were carried out on a 3-axis ultra-

precision diamond turning machine, which is equipped with an aerostatic bearing spindle and liner slides. Two different single-crystal diamond tools from Contour Fine Tooling were used in the facing cut to generate an ultra-precision substrate surface and nanostructures on the surface, respectively. The diamond tool used for the facing cut has a nose radius of 0.5 mm, a rake angle of 0˚, and a clearance angle of 15◦, respectively. A zero-rake sharp point diamond tool (V-shaped) was used for the fabrication of microstructures. The tool geometry and the machining conditions are listed in Table I. No coolant lubricant was used during the machining process. As shown in Figure 1, the turning was performed on the brass stamp with a dimension of 10 x 10 x 5 mm^3. The shape of the structures was spiral lines with 2 μm apart.

Table I: Machining condition and tool geometry

Conditions	Flat turning	Patterning
Tool material	Diamond	Diamond
Workpiece material	Brass	Brass
Tool radius	0.5 mm	0.1 mm
Rake angle	0 degree	0 degree
Clearance angle	15 degree	57 degree
Feed rate	2 μm/rev	2 μm/rev
Depth of cut	2 μm	2 μm
Spindle speed	500 rpm	500 rpm

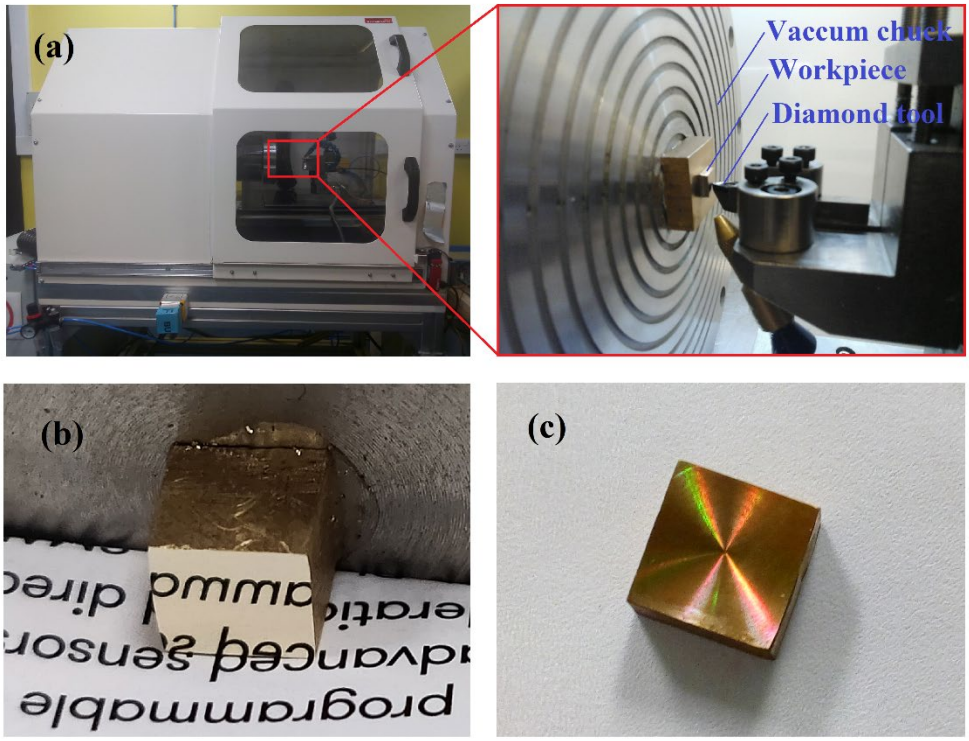

Figure 1. Fabrication of brass stamp with SPDT. (a) Machining set up. (b) Brass stamp after flat turning. (c) Brass stamp after microstructure fabrication.

The machined brass surface was inspected by an atomic force microscope (DI Dimension 3100). The AFM images and the profile of the structures are shown in Figure 2. It has been observed that some cutting chips are accumulated and remain at the edges of the grooves, which from burrs (Figure 2a-b). Some of the burrs have heights of >100 nm, which can make the inhomogeneous contact between the stamp and the substrate during the oxidation process. The fabricated parallel lines have a periodicity of 2 μm with an average height of ~380 nm (Figure 2c). The surface texture on the top of the motif is shown in Figure 2(d), where the maximum variation of the peak of the nanostructures is measured to be 13 nm.

There are many factors influencing the burr formation, such as cutting speed, tool geometry, feed rate, undeformed chip thickness, and material properties of the workpiece [14]. Li et al. found that the burr formation during diamond turning of the brass materials at the smaller grating pitch (~2 μm) mainly results from the material metallographic inhomogeneous properties (the uneven proportion of α and β

metallographic) [15]. Less burr formation occurs with the more β metallographic in the brass.

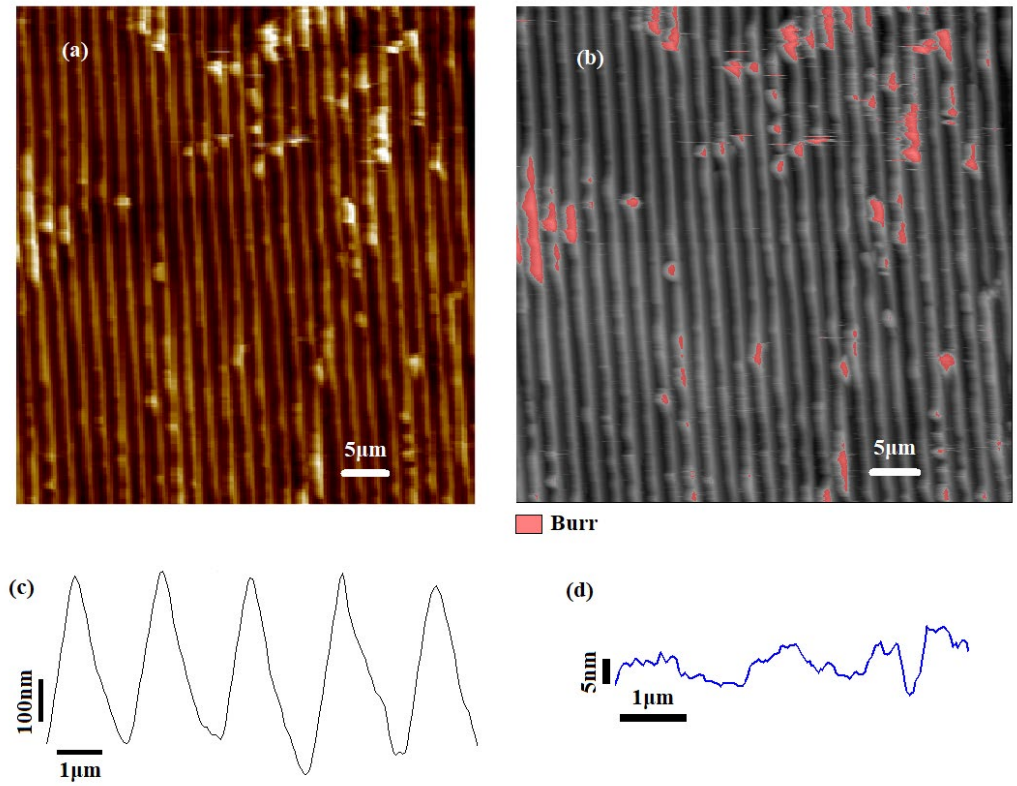

Figure 2. (a) AFM images of the fabricated brass structures. (b) Burrs are marked to visualize clearly. (c) Profile of the fabricated structures. (d) Surface topography on the top of a motif.

2.2 Preparation of the Sample

The first experiment was performed with the brass stamp on a silicon (100) substrate. But no significant oxidation occurred because of the non-uniform contact due to the high surface deviations and the burrs, as explained in the previous section. A brush layer can be effective in improving homogeneous contact between the surfaces of the stamp and the sample. By introducing a thin layer of polymer between the surfaces of the stamp and the sample, the oxidation process is improved as this polymer film is deformed under the force exerted by the stamp, adapting the separation surface to obtain a homogeneous pressure over the whole surface. The hydroxyl-terminated polystyrene (PS–OH) is a suitable polymer as a brush layer in this regard. These

polymers are used to create chemical patterns that guide the self-assembly of block co-polymers [16].

To prepare the sample, a thin film of hydroxyl-terminated polystyrene (PS–OH) of 4.5 kg/mol (Mn) molecular weight and polydispersity index of 1.09 was deposited by spin-coating and annealed on a p-type Si (100) substrate with a native oxide layer. The resistivity of the Si substrate was 4 –40 Ω-cm. The structure of the sample is shown in Figure 3.

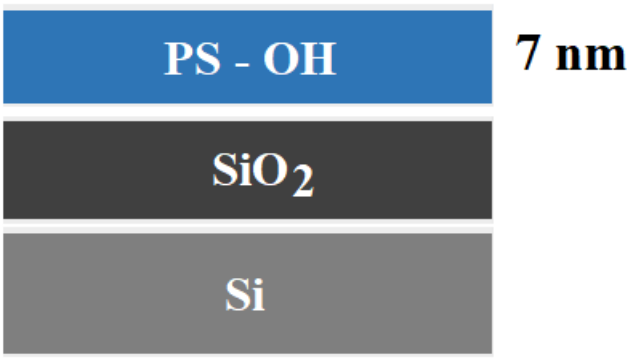

Figure 3. Schematic of the Si substrate with PS-OH brush layer

2.3 Oxidation process and results

The oxidation process was performed with a nanoelectrode lithography oxidation instrument, which is kept inside an enclosure. The sample was placed on a metal base, which could be moved in the x and y directions. The stamp was held above the sample on a platform which could be moved vertically (z direction). The motions of these three directions allows the correct positioning of the sample relative to the stamp. The sample and the stamp were connected to the positive terminal and the negative terminal of a voltage source, respectively. To maintain a certain humidity condition, bubbling nitrogen air was supplied inside the enclosure. A hygrometer was used to measure the relative humidity. The force exerted on the sample was measured by a weighing scale placed under the sample base. After the stamp was gently placed in contact with the silicon substrate, a bias voltage of 65 V DC was applied between them for 5 minutes under a relative humidity of 80%. After the oxidation process, an RIE etching was performed in a commercial system (Oxford 80 plus RIE). A SF_6 (10 sccm) and O_2 (5

sccm) plasma of 15 W and 6.25 × 10^{-2} m Torr pressure etched 12 nm of silicon in 32 seconds.

Figure 4 (a – c) shows the optical images of the silicon motifs at different areas of the substrate after the RIE etch. It can be seen that the motifs have been transferred to the surface of silicon with some irregularities and inhomogeneities in their shapes such as broken lines (line interception), nano bridges between lines and double lines. When comparing the images of the brass stamp (Figure 2), it is observed that the transferred patterns are similar, and the irregularities are due to the presence of the burrs on the stamp. Again, some areas of silicon substrate could not be oxidized (Figure 5). This is happened due to the existence of some burrs with considerable height variation on the stamp, which makes the misalignment between both surfaces of the stamp and the sample within these areas during the oxidation process. Figure 4(d) shows the profile of the section marked in Figure 4(c). It is observed that the height of these silicon motifs is about 12 nm with a full width at half maximum of 700 nm and 2 µm apart. In this case, the aspect ratio of the motifs is achieved as 0.017.

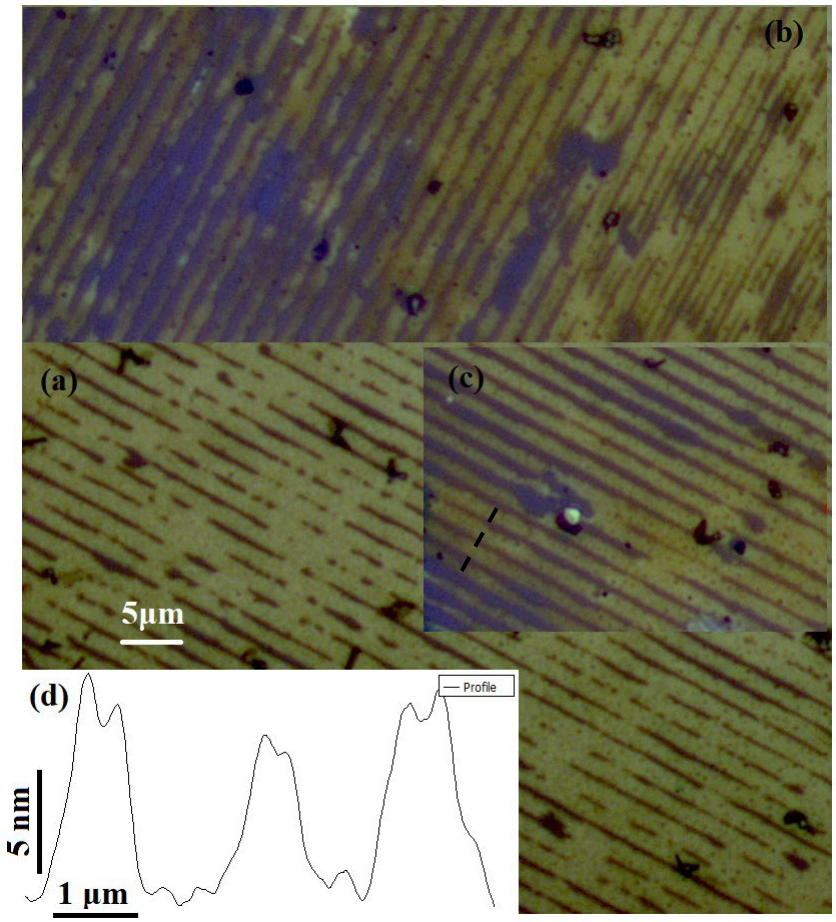

Figure 4. (a)-(c) Optical image of the motifs at different areas of the substrate after the RIE etch process. (d) Profile of the silicon lines manufactured.

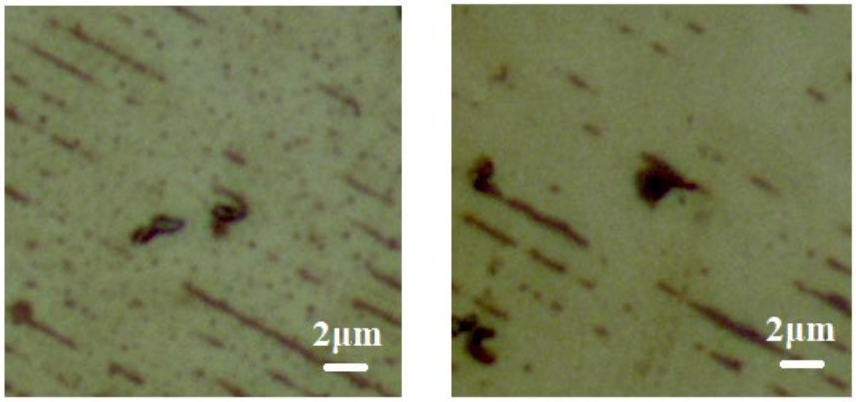

Figure 5. Unoxidized areas on the substrate due to burrs.

3. Discussions

The stamp with a low elastic modulus (for example, PDMS of ~2 MPa) makes the stamp susceptible to deform, buckle, or motif collapse [17]. Therefore, patterns with smaller features require more rigid mechanical properties of the stamp. Brass stamp with the high elastic modulus (~100 GPa) is advantageous in this regard. However, high reproducibility can be achieved with the brass stamp because of its breaking strength. No significant degradation in the stamp surface is observed after more than 10 operations. This property is beneficial in manufacturing industries as it increases the lifetime of the stamp. Another advantage of the brass stamp is the ability to achieve patterns with a high aspect ratio that is desired in nanolithography applications.

On the other hand, the most significant problem in the brass stamp is the burrs formation during the SPDT process, which causes pattern irregularity and non-oxidation area. The deburring techniques can be used to improve the surface quality, but these techniques are too specific, complicated, and high cost [18]. Alternative solutions would be to use other machining/lithography processes for the stamp fabrication that are cable to limit the burrs formation (for example, EBL, FIB).

4. Conclusions

The fabrication of patterns on the brass surface was achieved with the SPDT machine. Some burrs formation occurred during the machining process, which can be reduced by using deburring techniques or other fabrication techniques. The burrs make the brass stamp difficult to achieve the pattern replication as they make an inhomogeneous contact between the stamp and the substrate. The oxidation process has been improved by introducing a thin polymer film (PS-OH) of approximately 7 nm on top of the silicon surface. The local deformation of this thin film balances the pressure exerted by the stamp on the sample. Consequently, the homogeneous contact so as the pattern transfer has been improved substantially. However, there were some irregularities in the pattern due to the presence of the burrs on the stamp. Besides, the brass stamps show no degradation after many uses, which makes them advantageous to use in the NEL process. Therefore, the issues of the burr formation, higher surface deviations, and non-uniformity must be resolved in making these stamps applicable to the NEL process.

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Data statement

All data underpinning this publication are openly available from the University of Strathclyde Knowledge Base at https://doi.org/.....

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