Diamond Replicas on Silicon Substrate

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Abstract

In this work we evaluated quantitatively the fidelity of the diamond replicas, concerning to the dimensions and morphology of the silicon molds. The micrometric and nanometric characterization of the diamond replicas and the silicon substrates was done by atomic force microscopy.

Keywords: Diamond, replicas, silicon

1. Introduction

Most of the microelectromechanical systems (MEMS) devices have been based on silicon up to now. This is due to the technological know-how accumulated on the manipulation, machining and manufacturing of silicon. However, the silicon properties are poor, especially when compared with diamond, concerning to the wear resistance, chemical inertness, thermal conductivity, etc. Therefore, fabrication of diamond microstructures is a promising field for MEMS [1-5].

In this work we evaluate quantitatively the fidelity of the diamond replicas, concerning to the dimensions and morphology of the silicon molds. The basic technique used to obtain the replicas can be summarized as: in the first step, the silicon mold is fabricated using well established process technology, in the second step a diamond film is grown on it and, finally, the silicon substrate is etched out. In this way we obtain a diamond replica of a silicon mold, as the final device [6-10]. The diamond surface formed in contact with the silicon substrates will be named "back" of the film and the upward side of the diamond film will be called "top" of the film.

2. Material and Methods

The silicon mode preparation was made using a conventional microfabrication method (see the sequence in fig. 1). Initially the silicon wafer was cleaned and an aluminum thin film was deposited on it (fig. 1a). A photoresist layer was then deposited on the aluminum film (fig. 1b) and, using a desired mask, the wafer was exposed to ultraviolet light. The photoresist was then developed (fig. 1c) and, using wet etching, we removed selectively part of the aluminum film (fig. 1d). Following, a plasma etching was used to remove selectively part of the silicon wafer (fig. 1e). The aluminum film and the photoresist were then completely wet etched (fig. 1f). At that time we have already the microstructure on the silicon wafer. Note that, the aluminum film was used to avoid the silicon etching in the masked region, since the photoresist is also etched by the plasma etching.

The next step was to seed diamond microparticles (with 1 μm in size) on the silicon to improve the diamond nucleation. Following a diamond film was deposited using a microwave plasma assisted chemical vapor deposition system (fig. 1g). A description of the CVD equipment can be seen elsewhere [11, 12]. The following growth parameters were fixed: 300 sccm hydrogen flow rate, 3 sccm methane flow rate (1-vol% methane in hydrogen), 80 torr chamber pressure, 820° C substrate temperature, with a nominal 850 W microwave power and the growth time was 24 h.

Finally, the silicon substrate, used as a mold, was wet etched, with a solution composed of a mixture of hydrofluoric acid, nitric acid, and acetic acid at 23°C. The $HF:HNO_3:HC_2H_3O_2$ volume ratio was 2:1:1. The acetic acid was added to prevent violent reaction that can fracture the diamond film during the substrate etching (fig. 1h).

In this way a diamond replica was obtained, reproducing the microstructure of the silicon surface.

An Atomic Force Microscope, the NANOSCOPE IIIA from Digital Instruments, performed the micrometric and nanometric characterization of the diamond replicas and the substrates. A silicon nitride tip was used, with a highest measurable angle of about 65°. The radius of the tip used for the AFM measurements was about 50 nm, what mean that approximately 20 nm of the tip was in contact with the sample. Considering that the highest magnification used was (32 x 32) μm, the smallest pixel used for the images was 63 nm, then it is not necessary to take into account the convolution effect of the tip shape and surface profile.

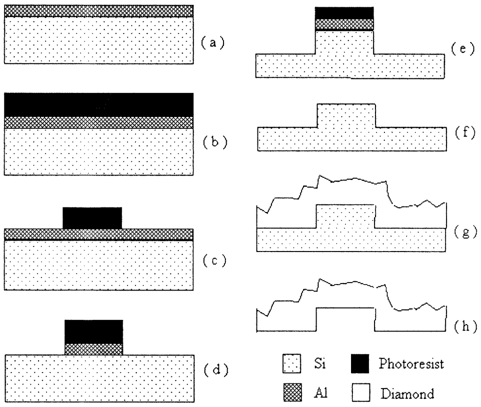

Figure 1: Sequence used in the microfabrication on the silicon surface.

We have imaged five different regions of the silicon mold with scan size between 32 μm and 64μm and, after the replica preparation, we have imaged exactly the same regions on the back of the diamond replica. Then, the corresponding images mold/replica were compared quantitatively.

3. Results and Discussions

In fig. 2 is presented a panoramic picture of the silicon mold. The registered area is (8.3 x 5.3) mm^2. The dark region is the etched area and the bright one corresponds to the original silicon, or no etched area.

A significant parameter to determine the fidelity of the replicas [13] is the surface roughness ω, which is defined by

$$\omega = \sqrt{\frac{\sum_{i}(Z_i - Z_{ave})^2}{N}}$$

where Z_{ave} is the average height and N is the number of points considered in the sample surface. It is basically the standard deviation (rms) of the heights (Z_i) for the N considered points of the surface: That is, the roughness ω gives the uncertainty of the measured heights on a given surface.

We measured, by AFM, the roughness of the silicon mold surface and the roughness of the diamond replica, on its back. The roughness rms of the silicon mold, on a region etched was 0.7 nm and on a region no etched was 0.3nm. On the back of the diamond replicas we got about the same roughness rms of 10 nm for both regions, where the silicon mold was etched and no etched.

With these results, we can conclude that, for the deposition parameters and substrate pretreatment used here, we do not expect to reproduce the silicon mold with precision better than 10 nm.

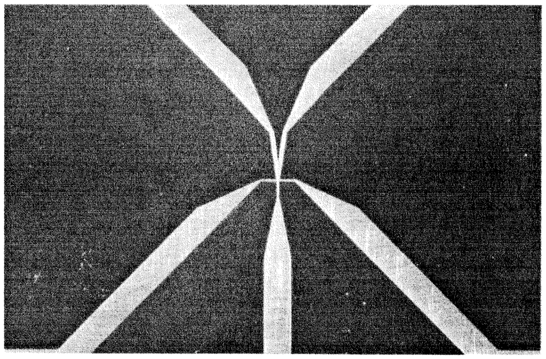

Figure 2: Picture of the silicon mold with a total area of (8.3 x 5.3) mm^2. The dark region is the etched area.

Fig. 3a shows an AFM image of the silicon substrate with (64 x 64) μm^2 and a z range of 400 nm. Fig. 3b shows an image of the corresponding region on the back of the diamond replica with (64 x 64) μm^2 and a z range of 600 nm. Note that the diamond replica image was inverted ($z \rightarrow -z$), so, in this way, it is easier to compare them. It is important to observe that the replica image presents the surface that was in contact with the silicon surface shown in Figure 3a.

In the AFM images of the silicon mold, the measured step was between 150 and 200 nm.

In fig.3, on the edges of the no etched regions, it is possible to observe a smaller step with about 2 μm width and height between 30 and 50 nm.

So, in this work we measured two steps in different scales range. The larger step in a scale of hundred of nm and the smaller step, giving information about the replica reproduction for a reduced scale.

For the larger scale, the shift of the reproduction was between 3 % and 23 %. For the smaller scale, the measured shift was between 3 % and 38%.

As expected, for the scale between 30 and 50 nm (the smaller one), the shift was larger due to the rms roughness, around 10 nm. But for the scale between 150 and 200 nm, the shift was also high, which cannot be due to the roughness of the back of the diamond replica.

A possible explanation for the observed high shift, between the height of the silicon mold and the diamond replica, is the stress of the diamond film. Probably, when the silicon substrate is etched out, the relaxation of the diamond film deforms, changing the original local profile of the replica.

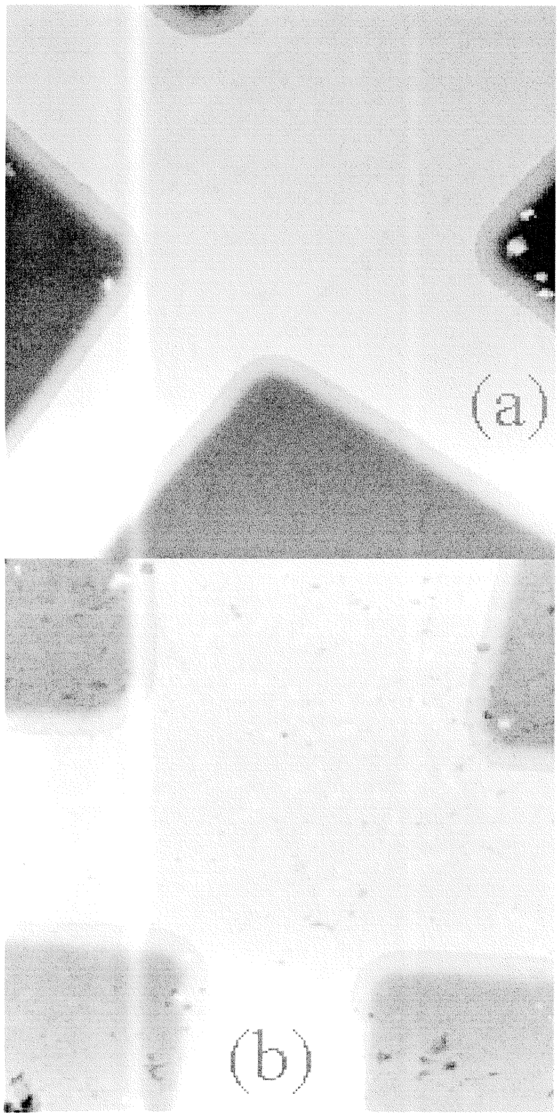

Figure 3: AFM images of the silicon substrate (a) with (64 x 64) μm^2 and a z range of 400 nm and of the corresponding region on the back of the diamond replica (b) with (64 x 64) μm^2 and a z range of 600 nm.

4. Conclusions

It was appropriate to analyze the microstructure reproducibility of silicon in diamond replicas using the AFM technique. In order to perform this analysis we have measured the roughness and vertical distances on the silicon mold and on the back of the diamond replica.

It is known that the fidelity of diamond replicas on silicon substrate depends on the substrate preparation [13], since it affects the roughness of the replica. In this paper , we suggest that the stress of the diamond film can also affect the fidelity of the diamond replica. So, it should be interesting to study diamond replicas changing the substrate preparation and also the deposition parameters, trying to control de diamond film stress.

Acknowledgments

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