Modification on surface roughness by combining dry and wet etching

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ASTRACT

The materials owning the rough surface have been adopted in many applications, such as MEMS devices, solar cell, DRAM, and so on. However, the modified targets on the previous methods were almost limited to silicon-type materials, and some had the limitations in the material properties. Recently, a process combining spin-on photoresist and one-step RIE was proposed to modify various materials, which can be etched by RIE. Here, a modification process, which combines spin-on photoresist, two-step RIE, and wet etching, is proposed to extend the feasible materials to be modified, because more materials can be etched by chemical solutions. Also, it is a low temperature process, and no extra mask is needed. From the experimental results, the modified surface can be used to alleviate stiction of microstructures, and a detachment length is found to be about 2.2 times longer than the cantilevers without the modified surface. Moreover, the related parameters for the anti-stiction are also developed, and the effects are compared with the previously developed one-step RIE method.

Keywords: surface modification, surface roughness, texture, RIE, stiction, detachment length.

1. INTRODUCTION

Materials owning the rough surface have been applied to dynamic random access memory (DRAM) [1], silicon solar cell [2]-[7], and sticking prevention in MEMS microstructures [8]-[12]. In 1979, Gittleman and Sichel [2] first used reactive ion etching (RIE) with fluorine based gases to texture the surface of Si wafer; the principle was based on the *in-situ* deposition of aluminum, and the rough surface was applied to silicon solar cells. Later, the similar methods [3][4] were developed to modify the surfaces of amorphous silicon and tungsten respectively, and another method was proposed by using Cl_2 gas [5] to form the pyramid-like structure on the multicrystalline silicon. In 2000, Schnell *et al* [6] modified the black silicon process [7] to texture the multicrystalline silicon for the same applications. On the other hand, textured surfaces can be used to alleviate stiction phenomenon of the suspended microstructures due to the reduction of interfacial areas between the contact solids. In the past, there were several methods proposed for the MEMS applications. Alley *et al* [8] combined thermal oxidation and RIE to texture the polysilicon layer with large grains. In 1996, Yee *et al* [9] applied the heavy doping, thermal oxidation, and two-step RIE, where honeycomb-shaped grain holes were formed on the surface, and the technique was realized in DRAM application [1] also. In SOI-MEMS applications, Matsumoto *et al* [10] performed silicon anodization process to form the hillock on the surface. Later, Fujitsuka and Sakata [11] used $HF-HNO_3-CH_3COOH$-mixed solution as the selective etching of Si substrate to prevent stiction. However, the modified targets on the above mentioned strategies were almost limited to silicon-type materials, and some had the limitations in the material properties. Recently, the process combining spin-on photoresist and one-step RIE [12] was proposed to modify various materials, but the modification process could not apply to every material, since some materials could not be etched by RIE.

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Here, a new method, which is expected to apply surface roughness modification to more materials, is proposed. The process consists of spin-on photoresist, two-step RIE process, and wet etching process. The principle is based on uneven RIE to the photoresist, which acts as the self-assembly etching mask. After performing wet etching to the underneath material by the corresponding chemical solutions for proper time, the original smooth surface will be patterned and the rough topography will be obtained. In this study, polysilicon and silicon oxide are demonstrated as the modified materials. Also, the characterization on the modified surfaces and anti-stiction effect are shown and discussed.

2. MODIFICATION PROCESS

The proposed process of surface roughness modification is schematically shown in Figure 1. First, positive photoresist (Fujifilm FH-6400) is spun on the surface of the material that is desired to be modified, so-called the target material. The resist layer is baked at 120°C for five minutes, and the thickness is above 1 μm (Figure 1(a)). Then, two-step RIE is carried out in SAMCO RIE-10N etcher. The process is used to make the photoresist form the self-assembly etching mask for the underneath target material. At the first RIE step, SF_6+O_2 gases are performed to unevenly etch the photoresist until the target material is nearly revealed (Figure 1(b)). Since the fluorine radicals may react with some materials to be the solid compounds and etch process cannot progress further, the target material should not be revealed at the current step. Then, the secondary step is achieved by RIE with O_2 only, and the target material is revealing in some spots (Figure 1(c)). After above two-step RIE process, the photoresist patterns with nano scale are created and can be used as the etching mask for the next step. Followed by wet etching with the corresponding chemical solution for proper time, the target material is patterned and the topography of the photoresist is transferred in the process. Finally, residual photoresist is removed by the H_2SO_4 and H_2O_2 mixed solution (ratio=3:1, 85°C~95°C), and the target material with pyramid-shaped asperities is revealed, as Figure 1(d) shown. Because most materials can be etched by chemical solutions with photoresist as the masking layer, roughness modification on more feasible materials can be achieved by the proposed method.

Among the modification process, the recipes of two-step RIE affect the surface status of photoresist mask directly, and the etching time to pattern the target material depends on the material to be modified. Here, the polysilicon is used as the target material to demonstrate the proposed method. More details about the process parameters will be discussed in the following sections.

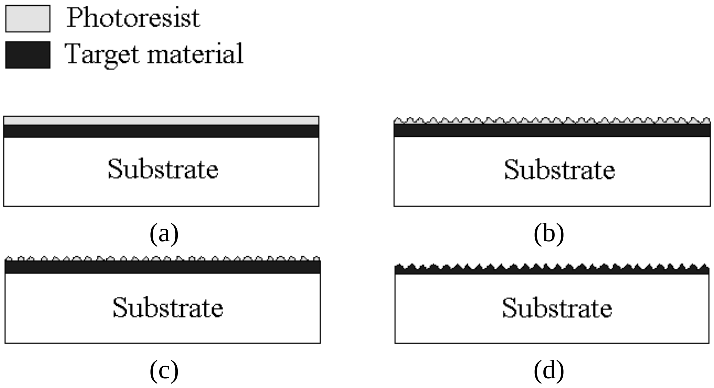


Figure 1: The proposed modification process on surface roughness: (a)spinning and baking photoresist on the target material; (b)etching the photoresist by RIE with SF_6+O_2; (c)etching the photoresist by RIE with O_2 only; (d)etching the target material with the corresponding chemical solution and removal of photoresist.

3. MODIFICATION PARAMETERS AND RESULTS

The modification process can be divided into two procedures, forming nano patterns on the photoresist mask and patterning the target material by wet etching. The parameters of the former procedure will affect nano patterns on the mask, and the second procedure will affect the surface status and the anti-stiction effects of the modified target material directly.

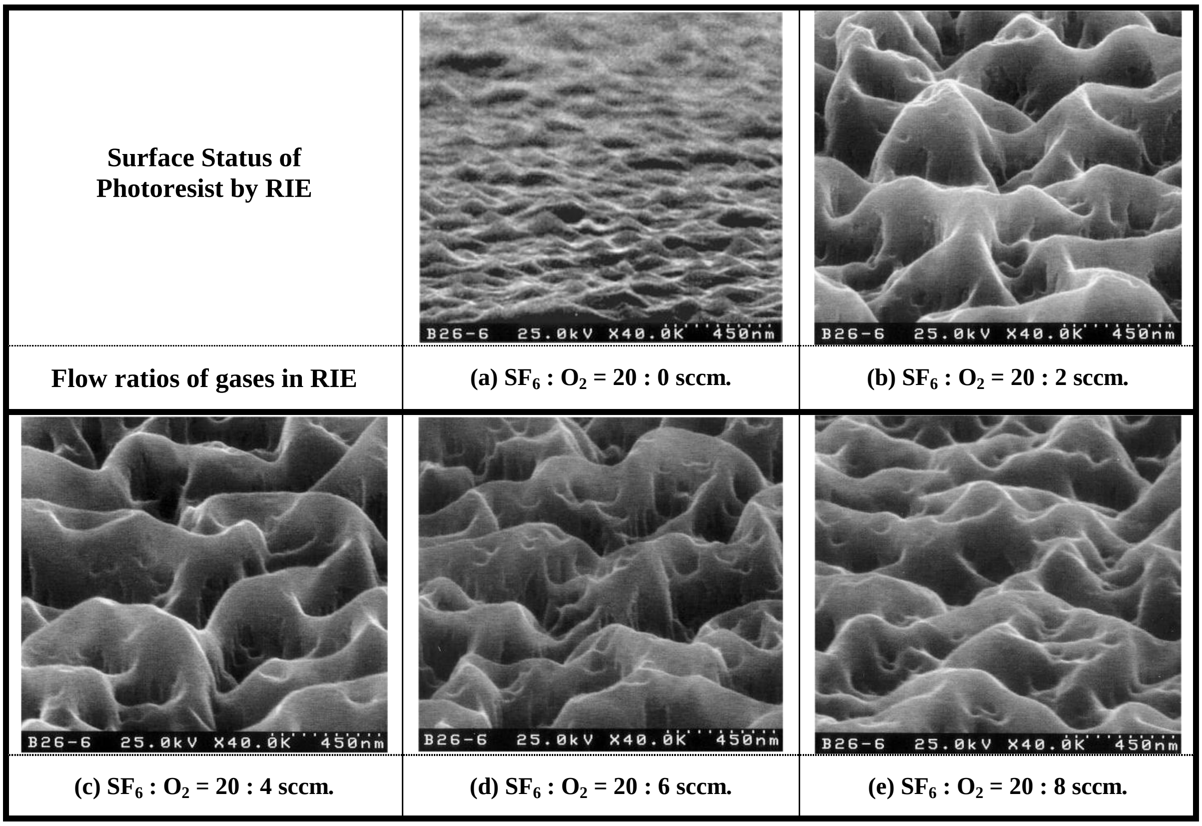
3.1 Surface modification on photoresist

The surface status of photoresist is determined by the two-step RIE process. The photoresist surface suffering etch from the first RIE step is shown in Figure 2. The working time, working pressure, RF power, and the SF_6 flow rate are fixed at 4 minutes, 20 mTorr, 100 W, and 20 sccm, respectively, and the experiments focus on the influences of the different O_2 flow rates. According to the SEM observations, it can be found that the O_2 gas plays a critical role in the modification of photoresist. Adding O_2 in SF_6 makes the topography of photoresist ragged, and using the pure SF_6 shows relatively even surface. Also, the O_2 flow rate affects the topography of photoresist surface. The surfaces by RIE with 2 sccm and 4 sccm have similar topography. Both O_2 flow rates are acceptable, because photoresist surfaces have evident ripples. The recipes of SF_6 and O_2 respectively equal to 20 sccm and 4 sccm under different RIE etching time have been tested in detail. Consequently, the parameters in the first RIE step with SF_6/O_2 = 20/4 sccm and etching time = 4’10” are selected.

The secondary RIE step with O_2 only is used to reveal the target material underneath the photoresist layer. The same parameters except SF_6 in the first step are performed with various etching time of pure O_2 RIE. It is found that 15” etching time is sufficient to satisfy the requirements; however, the time longer than 30” attenuates the surface roughness. Figure 3 shows the surface status of photoresist after the second RIE. The target material underneath the photoresist shown in Figure 2 needs to be revealed at the present step. The self-assembly nano patterns on photoresist will be served as the etching mask for the later wet etching procedure. Surface modification on the target material can be achieved after transferring the photoresist patterns by wet etching for proper time.

3.2 Surface modification on the target material

After forming the nano patterns on the photoresist mask, wet etching is applied to modify the target material surface by the proper chemical solution. Here, polysilicon is chosen as the target material, and the solution composed of $HNO_3:NH_4F:H_2O$ (mixing ratio= 64:3:33, etching rate=0.2 μm/min.) is the corresponding etchant in the wet etching. The proper etching time can be determined from the anti-stiction effect. Figure 4 shows the surface status of polysilicon for various etching time. The experimental results indicate that the photoresist nano-patterns have been transferred into the polysilicon layer after 30” wet etching. When etching time reaches 30” or 45”, both surfaces show asperities with many sharp apexes. However, longer wet etching time seems to reduce the rugged situation, as shown in Figure 4(d), where the etching time is 90”. The surface roughness/contour measurement instrument (Kosaka Laboratory Ltd., ET-4000) is used to calibrate the surface status of the modified surface, and the results of surface profiles, mean roughness (*Ra*), and ten-point height of irregularities (*Rz*) are listed in Figure 5. When etching time is equal to 30” or 45”, both surfaces exhibit the relative high density of sharp apexes. Furthermore, longer time decreases the density of sharp apexes, and only makes deeper etching depths.



(a) $SF_6 : O_2 = 20 : 0$ sccm.

(b) $SF_6 : O_2 = 20 : 2$ sccm.

(c) $SF_6 : O_2 = 20 : 4$ sccm.

(d) $SF_6 : O_2 = 20 : 6$ sccm.

(e) $SF_6 : O_2 = 20 : 8$ sccm.

Figure 2: The SEM images of photoresist surface by RIE with different flow ratios of SF_6 and O_2 gases for four minutes (pictures at 40k magnification).

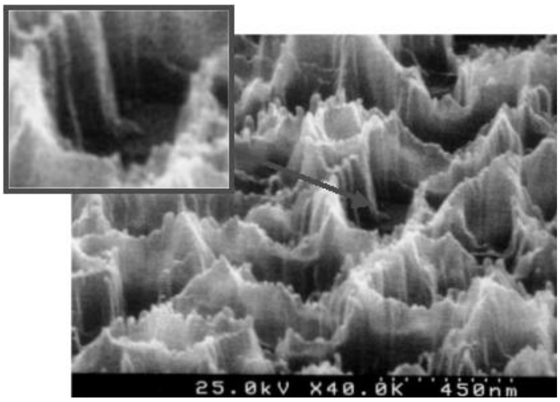
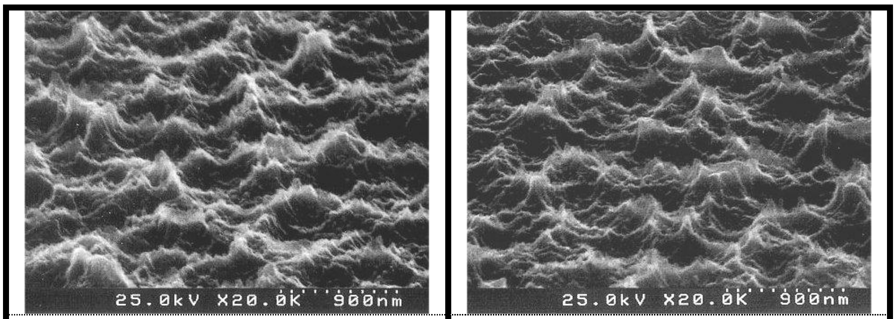
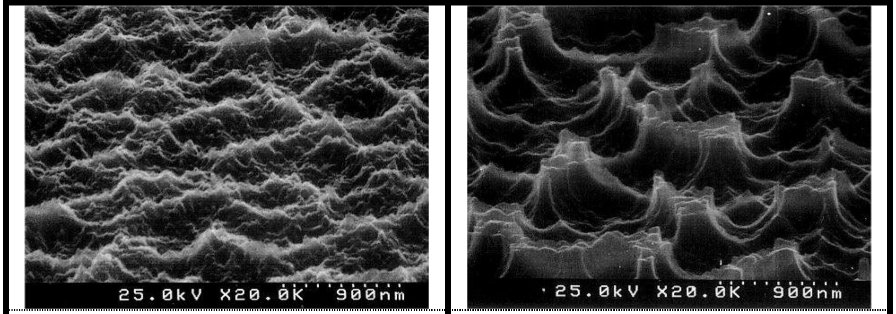


Figure 3: The self-assembly photoresist mask with nano-scale etching holes formed by the second RIE process (the SEM picture at 40k magnification), and the enlargement of an etching hole shown in the top left corner.



(a) Wet etching for 30”

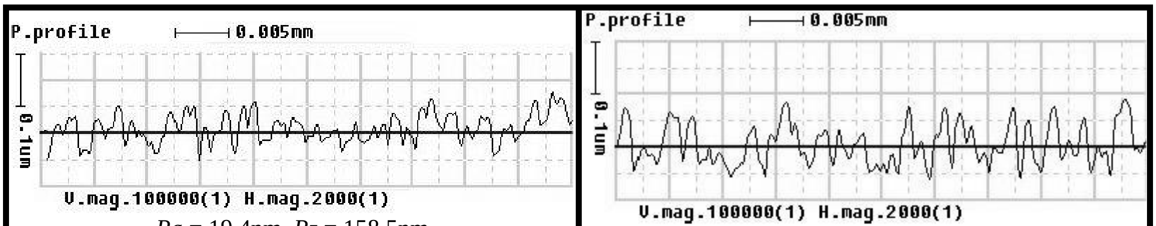
(b) Wet etching for 45”



(c) Wet etching for 60”

(d) Wet etching for 90”

Figure 4. The SEM on surface of polysilicon with different etching time in HNO_3:NH_4F:H_2O etchant.

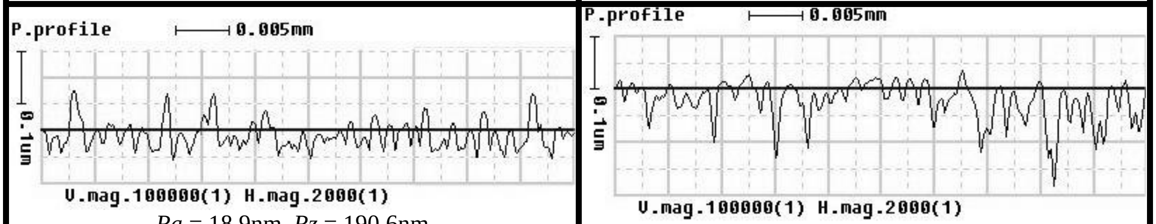


Ra = 19.4nm, *Rz* = 158.5nm

Ra = 30.4nm, *Rz* = 184.7nm

(a) Wet etching for 30”

(b) Wet etching for 45”



Ra = 18.9nm, *Rz* = 190.6nm

Ra = 24.4nm, *Rz* = 235.2nm

(c) Wet etching for 60”

(d) Wet etching for 90”

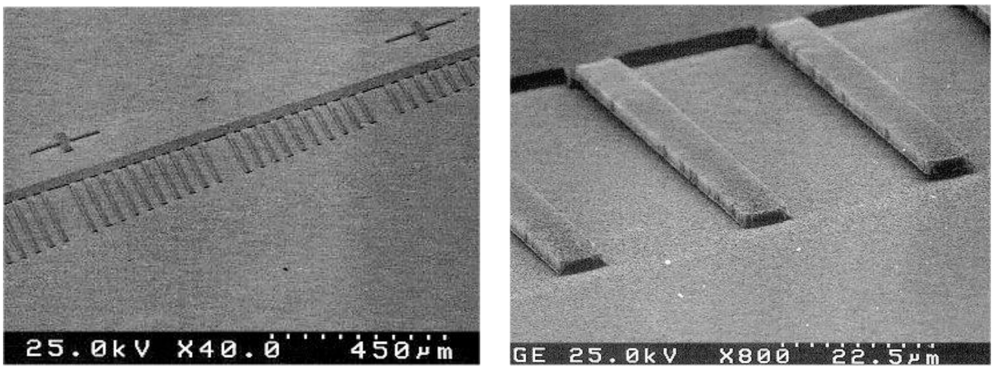
Figure 5. The surface profiles, mean roughness (*Ra*), and ten-point height of irregularities (*Rz*) of modified polysilicon with different etching time in HNO_3:NH_4F:H_2O mixed etchant.

4. ANTI-STICTION CHARACTERIZATION

Since a rough surface can alleviate stiction between the contact solids, detachment length technique [13], i.e. different lengths of cantilevers above the target materials, is performed here to verify the anti-stiction effects. In experiments, the cantilever array made of polysilicon with various lengths is fabricated above the modified surface of polysilicon. The width, thickness of the cantilevers, and height above the substrate are 10 μm, 2 μm, and 1.8 μm, respectively. The cantilever lengths range from 50 μm to 1500 μm in every 10 μm.

Referring to the fabrication process of cantilevers, first a 0.5 μm thick silicon nitride and 0.6 μm thick polysilicon are deposited on a (100) silicon wafer. This polysilicon layer is used as the target material, and the surface modification process with different modification time are performed. Later, an 1.8 μm thick oxide layer is deposited as the sacrificial layer. The regions of anchors are defined by mask #1. Then, another polysilicon layer with 2 μm thickness is deposited as the structure layer of cantilevers, followed by annealing at 1100°C for 2 hours in N_2 ambient. Cantilever beams with various lengths are patterned by mask #2 and etched by RIE. Finally, the cantilevers are released by pure HF solution for ten minutes, rinsed by IPA for one hour, and dried by hot plate at 120°C for one hour. Figure 6(a) shows the fabrication result of cantilever array with various lengths for the test of anti-stiction effect. The detachment lengths are determined by the SEM observation, as Figure 6(b) shown.

Figure 7 shows the detachment length distribution with polysilicon as the target material under different wet etching time; the upper and lower bounds are plotted in the figure also. Without modification on the surface roughness, i.e. wet etching time equal to 0”, the average detachment length is found to be about 123 μm. When etching time is equal to 45”, the maximum detachment length occurs and it also owns the minimum variation, where the average detachment length is about 270 μm. However, the detachment lengths decrease gradually with wet etching time longer than 45”, for example, the detachment length becomes 218 μm at 90” wet etching time. From the surface status of polysilicon shown in Figure 4 and 5, both surfaces at 30” and 45” show asperities with the largest density of sharp apexes, which indicate that they have less contact areas between two solids, which are consistent with the results of the detachment lengths. Therefore, etching time 30”~45” is found to be the satisfied etching time for the anti-stiction application. Also, according to the known etching rate of 0.2 μm/min, the distance of lateral etching is about 0.1 μm~0.15 μm at this period.



(a) (b)

Figure 6. (a)The fabrication result of cantilever array with various lengths for the test of anti-stiction effects; (b)Sticking and un-sticking cantilevers above the modified surface by the SEM observation.

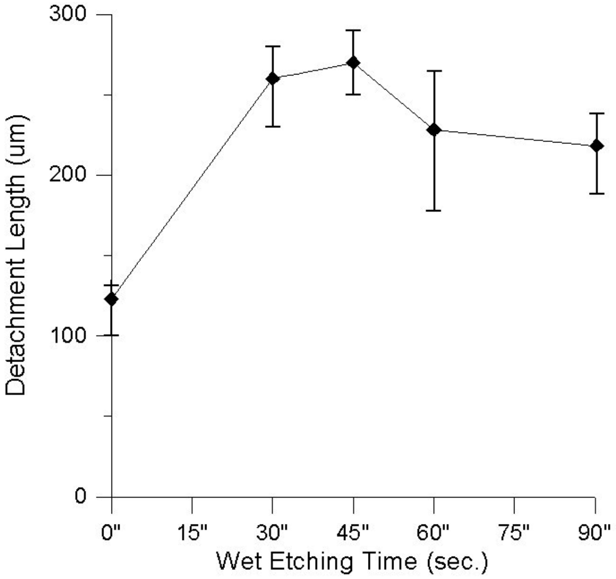


Figure 7. The detachment length distribution with polysilicon as the modified substrate for various etching time.

5. DISCUSSIONS

The process parameters in surface roughness modification are listed in Table 1, because the current process combining the self-assembly photoresist patterns and wet etching of the target material, the surface modification is expected to be useful for more materials as long as they can be patterned by wet etching, even not by RIE. Comparing to the method presented previously by Lee and Hsu [12], the current approach has the better anti-stiction effect, but larger variations in detachment length. The phenomenon implies that the surface modification by the current method may have less contact area between contact solids but the uniformity is not as good as the one-step RIE process. Figure 8 shows that another material, thermal oxide, is modified successfully on the surface by the process described in Table 1.

Table 1. The summary of the modification process on surface roughness for the anti-stiction application

STEP	PROCESS
1. Photoresist Spin	1 µm Fujifilm FH-6400 spun and baked at 120°C for 5min.
2. Surface Modification on Photoresist	**1st RIE step:** SF_6:O_2=20:4 sccm. at 20 mTorr and RF = 100W for 4’10” **2nd RIE step:** O_2= 4 sccm at 20 mTorr and RF = 100W for 15”
3. Surface Modification on Target Material	Wet etching by the proper etchant (later etching distance= 0.1~0.15µm)
4. Removal of residual resist	H_2SO_4:H_2O_2 = 3:1 with hot bath for 15 min.

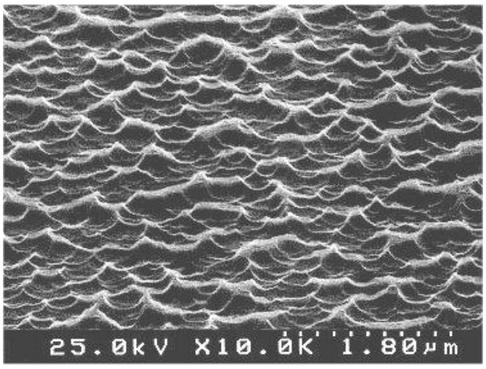


Figure 8. Successfully modified thermal oxide surface by the process described in Table 1.

6. CONCLUSION

A surface roughness modification process combining spin-on photoresist, two-step RIE, and wet etching, is proposed here. Since lots of materials can be patterned by chemical solution, it is possible to use the proposed method on more diverse materials in modification of surface roughness. Beside, the low-temperature process does not change the original fabrication process of micro devices and no extra mask is needed. The proposed process has been demonstrated to modify the surface of polysilicon and silicon oxide. The parameters for the anti-stiction application are also developed. The experimental results show that the detachment length after modification process is about 2.2 times longer than the cantilevers without the modified surface.

7. ACKNOWLEDGEMENTS

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