

Surface Micromachining Process for the Integration of AlN Piezoelectric Microstructures

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Abstract—We report a novel micromachining process to fabricate AlN (Aluminum Nitride) piezoelectric microstructures for actuator applications. Piezoelectric AlN thin films can be grown with (002) preferential orientation by means of RF reactive sputtering on various substrates. For this study, AlN was deposited on doped polysilicon layers, which act as both structural, and electrode layer. A thin layer of Cr was used as top electrode and as a mask for patterning of the AlN piezoelectric layer. Silicon oxide was used as a sacrificial layer. The structures were released by a freeze drying technique. Single run deposition of AlN and Cr turns out to be necessary to ensure good adhesion of the Cr layer to the AlN thin film during the sacrificial etching process either using BHF or HF. The released beams show compressive stress due to the AlN thin film. Further process development, using Cr seed layers to reduce stress, and device testing is in progress

Keywords— Piezoelectric AlN, Anisotropic patterning, Surface micromachining

I. INTRODUCTION

Integration of AlN thin films into a surface micromachining process will find many applications in micro systems and optoelectronics devices. AlN properties such as piezoelectricity, high acoustic velocity and chemical stability at high temperatures are attractive for MEMS devices. AlN with top and bottom electrodes forms a basic configuration for piezoelectric sensors/actuators for probe techniques. Preferentially (002) oriented AlN thin films are favourable for piezoelectric device applications [1]. Many methods to grow AlN thin films on several substrates have been discussed in the literature [2-4]. RF reactive sputtering is one of the common methods used to deposit

polycrystalline AlN thin films with preferentially (002) orientation.

Patterning and compatibility are the two key factors in silicon surface micromachining processes including AlN layers. For micro device fabrication, the minimum size of a structure and the etch selectivity to the mask layer are key parameters to be concerned. Micromachining of AlN with Cr as bottom and top electrode on SOI substrates after sacrificial layer etching was discussed in the literature [5]. The authors discuss AlN suspended resonators of size (160 μm x 160 μm) etched by 0.6 % wt. TMAH solution. Etching of AlN by different dry etching and wet etching methods was reported in [6-9]. Pearton et al. extensively discussed KOH etching of AlN and its compatibility with other compounds of nitrides and has shown selective etching of AlN to form a disc type structures for laser applications [6].

In this paper, a new silicon based micromachining process for AlN thin film is developed to make microstructures with a feature size down to 2 μm for piezoelectric device testing. Sacrificial layer etching is used to release the structures in post processing steps. Anisotropic patterning of AlN using wet chemical etchant Tetra Methyl Ammonium Hydroxide (TMAH) (25%) and Phosphoric acid based solutions is discussed. Chemical inertness to other chemicals such as BHF and HNO₃ (100%) are also tested. Etch rate and selectivity (undercut of AlN below the mask layer) are measured based on the inspection of structures at different steps using a Dektak surface profiler and scanning electron microscope.

II. EXPERIMENTS

A. Sample preparation

This section focuses on the sample preparation to (1) study the etching methods of AlN grown on the silicon substrates and (2) for the fabrication of various microstructures by a surface micromachining process.

Two types of sample configuration were used in this study (figure 1). Sample 1 (a) with AlN/Si was used for etch studies with various etch chemicals whereas sample (b) with AlN/doped polysilicon/SiO₂ layers was used to study silicon based surface micromachining. Hence, the doped polysilicon layer serves two purposes: to form a structural layer and to act as a bottom electrode for device testing. A thin layer of Cr is used as a top electrode as well as a mask layer for etching AlN and polysilicon.

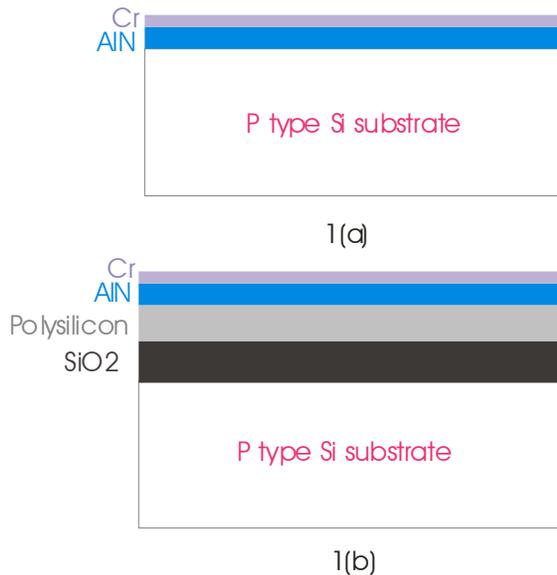


Figure.1 Layer schematic of samples prepared for wet etching experiments on Si and polysilicon layers (a) for patterning and (b) for surface micromachining

B. Pre processing

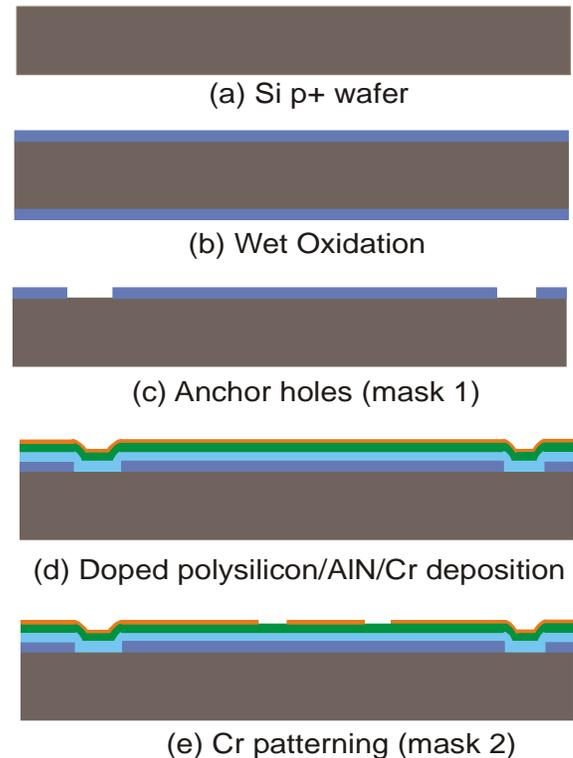
Prior to the deposition of any layer on silicon substrates, wafers were cleaned by standard HNO₃ cleaning procedures. For samples of type 1(a), prior to the deposition of AlN, the native oxide layer on the silicon substrates was removed by treating them in a 1% HF solution for a minute. Sample 1(b) was made with a stack of layers with a configuration of Cr/AlN/doped Poly Si/SiO₂/Si. The sacrificial oxide layer was grown on the silicon substrate by wet oxidation at 1150⁰ C to a thickness of 2.2μm. The polysilicon layer with thickness of about 1.8 μm was grown by Low Pressure Chemical

Vapour Deposition (LPCVD) at 590⁰ C on top of the oxide layer. Solid source boron diffusion in polysilicon was applied to obtain conduction layers.

AlN thin films with (002) preferential orientation were deposited on both the samples by RF reactive sputtering [7, 8]. A single run deposition of AlN and Cr layer was done without breaking the vacuum. The samples were checked for (002) preferential orientation using X-ray diffraction (XRD) 2θ-scan. The Cr layers were patterned by photolithography to make etch openings to expose the AlN thin films.

C. Surface micromachining process for AlN

Figure 2 shows a schematic process description for the AlN surface micromachining experiment. This process involves the sacrificial oxide layer etching to release the microstructures forming free cantilevers and suspended beams. The sample of type 1(b) was used to study this process. Highly doped silicon wafers were used as substrate to have good electrical conductivity between the polysilicon layer and the substrate. A Cr layer was used as a mask for patterning AlN. Cr is a better candidate as an electrode and a mask layer for this process when compared to Aluminum. Aluminum is attacked by AlN etchants and cannot be used in this process. Cr does not suffer from these drawbacks.



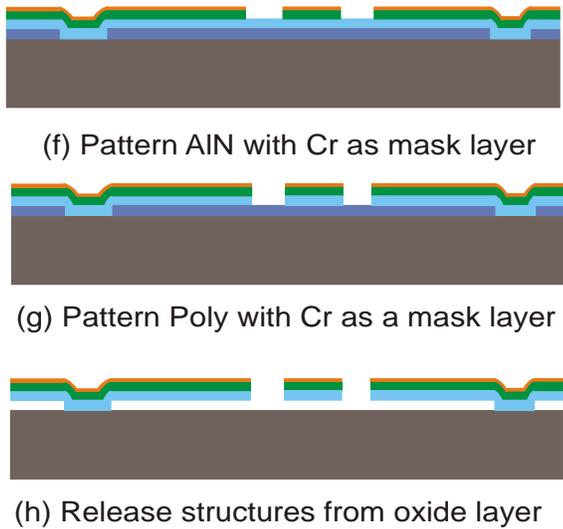


Figure 2. Schematic surface micromachining process chart for AlN thin film on doped polysilicon layer.

III. RESULTS AND DISCUSSIONS

Texture of the films was identified by X-ray diffraction technique with Cu-K α radiation at 40KV, 30mA tube settings. Figures 3(a) and 3(b) show the X-ray diffraction (XRD) results for an orientation measurement of AlN thin films. AlN thin films grown on doped polysilicon layers showed the presence of predominant (002) oriented grains of AlN (FWHM = 2.7 deg). AlN grown on silicon substrates showed the presence of (100) oriented AlN grains apart from dominant (002) oriented grains (FWHM = 4-6 deg).

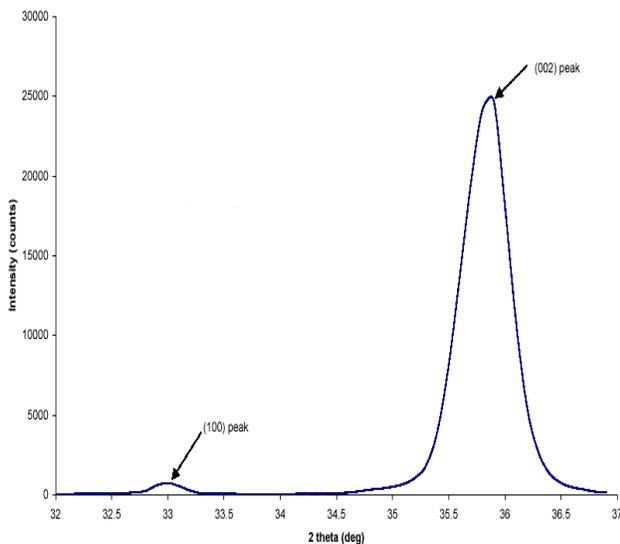


Figure.3 (a). Predominantly (002) oriented AlN thin film on doped polysilicon layer (FWHM = 2.7^o).

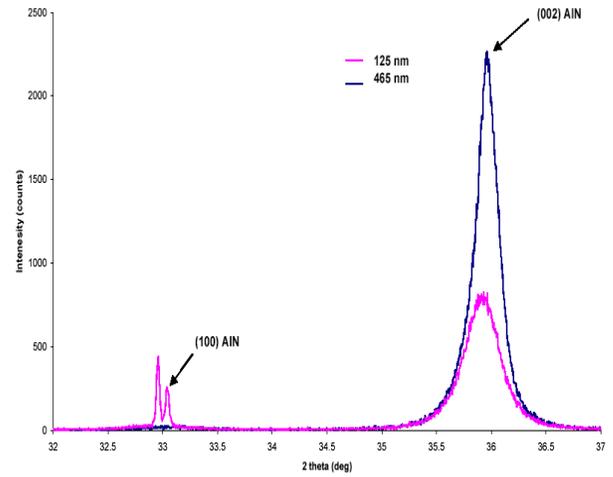
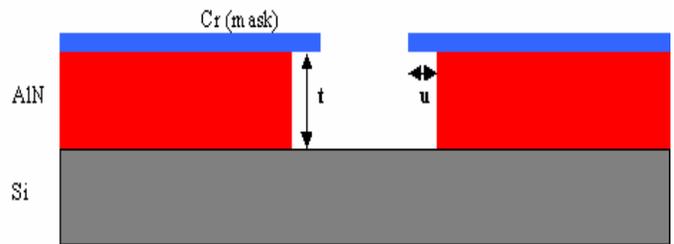


Figure.3 (b). AlN thin film on Si (100) substrates with different thickness.

As shown in figure 3(b), AlN shows the intensity peaks of (100) oriented grains when the thickness of the film is 125nm and its intensity decreases when the thickness increases to 455nm. It shows that when the substrate temperature increases the mobility of (100) oriented grains on the surface and enhances the growth in (002) orientation.

A. Anisotropic wet patterning

Samples of type 1(a) treated with different wet chemical etch solutions to study patterning of AlN thins film and accompanying etch profiles were studied using scanning electron microscopy (SEM). The etch rate was measured by depth profiling using a Dektak surface scan profiler. The term anisotropy ratio is defined with a schematic picture (exaggerated view) as shown below.



$$\text{Anisotropy ratio, } R_A = t_{\text{AlN}} / u_{\text{AlN}} \quad (1)$$

Where, t_{AlN} is the thickness of AlN layer and u_{AlN} is the undercut distance of AlN under the etch mask. The anisotropy ratio was calculated for different etch solutions and the results are tabulated (table 1).

Serial No.	Etchant	Temperature ($^{\circ}$ C)	Etch rate (nm/min)	Anisotropy ratio $R_A = t_{AIN} / u_{AIN}$
1	Al etch	21	--	--
2	Al etch	55	< 1	412
3	Al etch	75	3	72
4	Al etch	95	5	137
5	H ₃ PO ₄ (85%)	120	20	13
6	TMAH (25%)	21	24	43
7	BHF	21	<0.3	1433
8	HNO ₃ (100%)	21	--	∞

Table 1. Summary of different wet chemical etching of AlN.

Standard Al etch with a composition of phosphoric acid (80%), acetic acid (16%) and nitric acid (4%) was used to etch AlN thin film. AlN etching started at a temperature above 55⁰ C. The etch rate was 3 nm/min at 75⁰ C and increased to 5 nm/min at 95⁰ C. Anisotropic etch profiles were obtained shown in figure 4 for the sample etched at 75⁰ C. Also, small whiskers were seen on the surface of the silicon wafer. These whiskers formation can be explained by the way AlN does grow initially. XRD results shown in figure 3(a) and 3(b) confirm the presence of a few atomic layers of AlN grains with a mixed orientation of (100) and (002) in the initial layer. Zhaung et.al reported a very slow etching of (100) oriented planes of AlN single crystals using KOH based etchants [9]. Hence, (100) oriented face of AlN forming whiskers because of slow etching when compared to (002) faces of hcp-AlN. The anisotropy ratio of AlN was found to be 72 using equation 1.

Aqueous phosphoric acid (85%) was also used to pattern AlN at 120⁰ C and the etch rate was found to be 20 nm/min. SEM pictures as shown in figure 5 revealed no whiskers on the surface of the wafer. It is clear that high temperature etching with phosphoric acid makes the surface of the wafer free from whiskers. The anisotropy ratio was found to be 13. Etching of AlN using TMAH (25%) solution at room temperature showed a selective anisotropic etching of AlN on silicon wafer as shown in figure 6. There were no whiskers on the surface of silicon and the etch rate was about 24 nm/min which is higher than phosphoric acid based etchants. The calculated anisotropy ratio was 43. AlN layer showed a

very high resistance to etch using BHF solution. AlN was found to be stable against nitric acid (100%) where the sample was immersed in it for about 4 hours. No etching of the AlN layer was found. The results are summarized as shown in table 1.

B. AlN in surface micromachining

Consider sample type 2 for AlN surface micromachining. AlN was patterned using Al etch solution at 95⁰ C. The pattern of Cr/AlN was transferred directly to doped polysilicon by directional etching of polysilicon structural layer using the Bosch process [10] that facilitates etch openings for sacrificial layer. As a last step, microstructures were released by isotropic etching of SiO₂ using BHF followed by a freeze-drying procedure. The released structures were inspected by scanning electron microscopy (SEM).

SEM pictures in figures 7, 8 and 9 show the fabricated microstructures of Cr/AlN stack on polysilicon layers. Whiskers on the surface of polysilicon left by the etch step of AlN, act as a mask for polysilicon directional etching using the Bosch process. As a result, needle/pillar like structures were formed on the silicon substrate as shown in figure 7. Freed clamped beams and freestanding cantilever beams are shown in figures 8 and 9 respectively. Poor adhesion of Cr on AlN causes peeling off of the Cr layer during sacrificial BHF etch was avoided by a single run sputtering of Cr after AlN deposition. The freestanding beams showed compressive stress.

IV. CONCLUSIONS

In this paper, preliminary experiments on anisotropic etching/patterning of AlN thin films on silicon substrates were studied using Al etch, Iso-phosphoric acid (85%) and TMAH (25%). An anisotropic etch behaviour of AlN thin films were found using a Cr mask. A novel AlN micromachining process on silicon based surface micromachining was established. We demonstrated the wet chemical patterning of AlN microstructures less than 2 μm size on polysilicon structural layer. Cr mask layers showed high etch resistance to AlN thin film patterning and polysilicon directional etching using the Bosch process. Single run sputter deposition of Cr/AlN layers ensures good adhesion of Cr on AlN. The Cr mask layer can be integrated as an electrode for device applications by patterning before sacrificial layer etching. Future research that include the study of surface micromachined AlN thin films on doped polysilicon layers for piezoelectric device applications.

REFERENCES

1. R. S. Naik, R. Reif, J. J. Lutsky C. G. Sodini, *Journal of Electrochemical Society*, **146** (2), pages 691-696, 1996.
2. Sung-Ui Hong, Gee-Pyeong Han, Mun-Cheol Paek, Kyung-Ik Cho, Soon-Gil Yoonb, *Electrochemical and Solid-State Letters*, **5** (7), pages G54-G56, 2002.
3. Bing-Hwai Hwang, Chi-Shan Chen, Hong-Yang Lu, Tzu-Chien Hsu, *Materials Science and Engineering A*, **325**, pages 380–388, 2002.
4. K. Dovidenko, S. Oktyabrsky, J. Narayan, M. Razeghi, *Journal of Applied Physics*, **79** (5), pages 2439-2445, 1996.
5. Hyun Ho Kim, Byeong Kwon Ju, Yun Hi Lee, Si Hyung Lee, Jeon Kook Lee, Soo Won Kim, *Sensors and Actuators A*, **89**, pages 255-258, 2001.
6. J. R. Mileham, S. J. Pearton, C. R. Abernathy, J. D. Mackenzie, R. J. Shul, S. P. Kilcoyne, *Journal of Vacuum Science and Technology A*, **14** (3), pages 836-839, 1996.
7. Saravanan. S, Erwin Berenschot, Meint de Boer, Gijs Krijnen, Miko Elwenspoek, *Proceedings of MME'03*, 2003.
8. Saravanan. S, Meint de. Boer, G. Krijnen, M. Elwenspoek, *Proceedings of SAFE*, 2003.
9. D. Zhuang, J. H. Edgar, Lianghong Liu, B. Liu, L. Walker, *MRS Internet Journal Nitride Semiconductor Research*, **7** (4), pages 1-6, 2002.
10. F. Laermer and D. Schilp, *patent US5501893*, Robert Bosch GmbH.

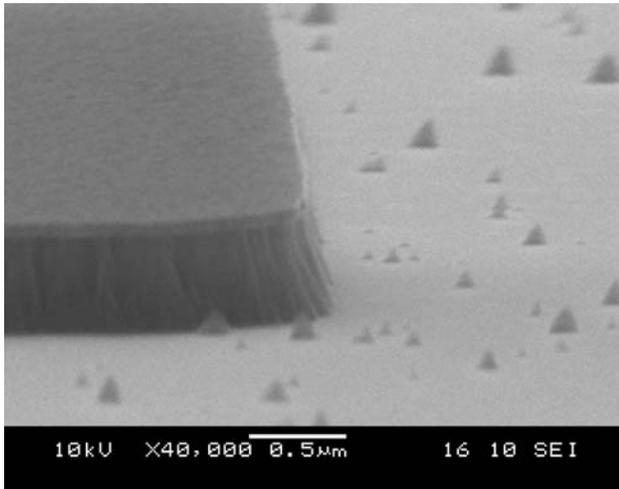


Figure. 4. Anisotropic etching of (002) oriented AlN grains with whiskers residue at the surface

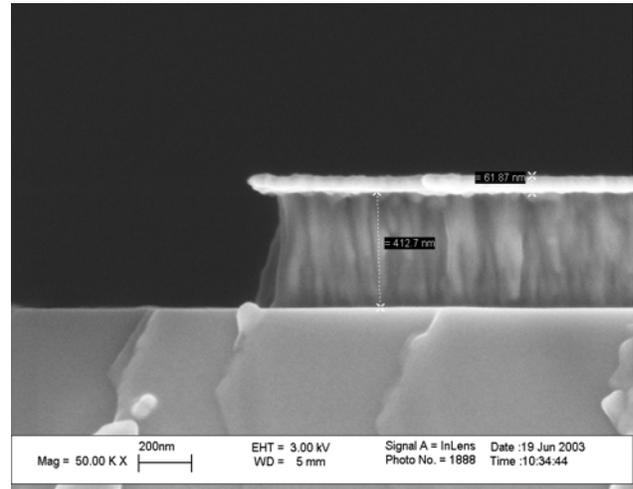


Figure. 5. Smooth vertical pattern of AlN, etched using Iso-phosphoric acid at higher temperature.

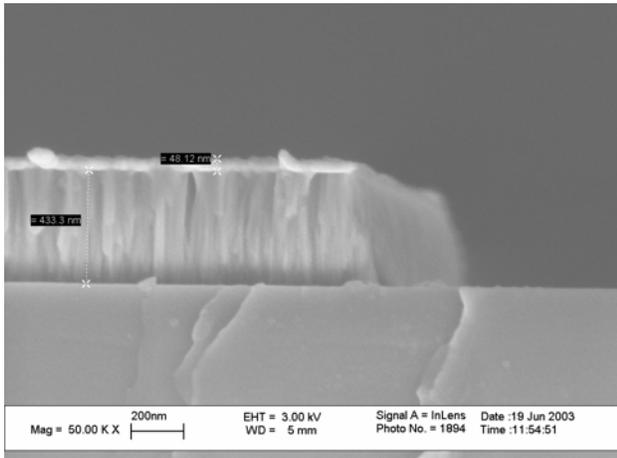


Figure. 6. Selective etching of AlN using TMAH (25%) with Cr mask layer at room temperature.

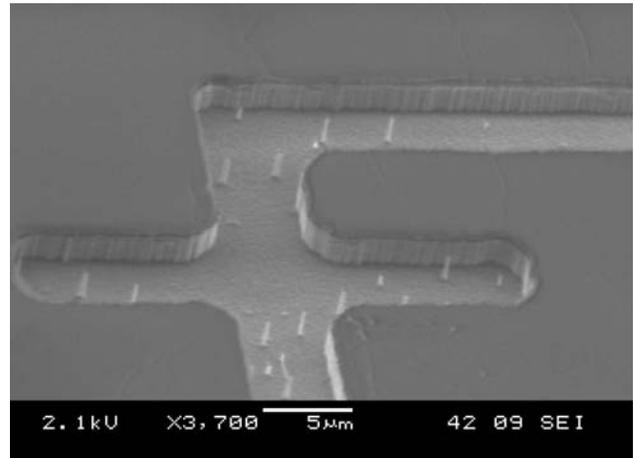


Figure. 7. Whiskers of AlN act as a mask and its pattern is transferred to polysilicon in Bosch process.

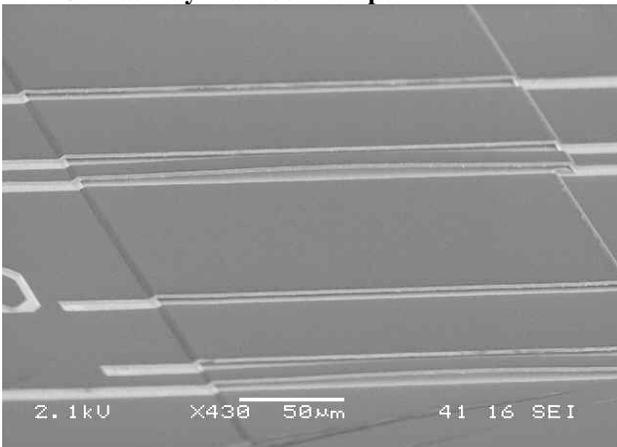


Figure. 8. Suspended clamped beams of AlN on doped polysilicon structural layer.

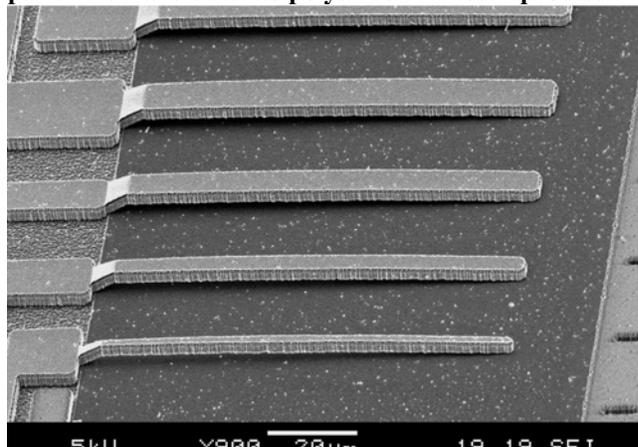


Figure. 9. Microfabricated cantilever beams after freeze-drying procedure.