

## Surface Properties After Wet Release of Microcantilever

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Received 30 October 2007; accepted 18 January 2008

Electrostatically actuated nickel microcantilever was fabricated by surface micromachining technology, using Ni (structural) and Cu (sacrificial) layers. Physical reasons influencing performance and sticking problem were analysed. Slightly increased silicon substrate surface roughness was determined by AFM for the samples after wet release. X-ray photoelectron spectra of typical samples show, that differences of surface adhesion between the samples after O<sub>2</sub> plasma treatment and after removal of Cu film and drying decreases and correlate with the copper residues and growth of the natural SiO<sub>2</sub> film.

*Keywords:* surface micromachining technology, microcantilever, wet release.

### INTRODUCTION

Microelectromechanical switches with the metallic cantilevers are used in many types of power devices because their on-resistance can be lower than that of semiconductor switches and their off-resistance and transmission frequency can be higher. Microelectromechanical switches can be found in a wide variety of electrical devices including toys, automobiles, mobile phones etc. [1 – 6].

Surface micromachining technology [7 – 12] on silicon substrate today is a basic technique for the fabrication of the microelectromechanical switches with the metallic cantilevers. This technique requires estimation of the compatibility between sacrificial and structural layers. The selection of a suitable sacrificial material depends on the structural material and particularly on the availability of an etching method that can selectively etch the sacrificial material without significantly etching the structural materials or the substrate. Polysilicon, oxide, nitride, metals, diamond, SiC or GaAs can be deposited as a structural material in combination with a selected suitable sacrificial material (polysilicon, oxide, nitride, metals, photoresist etc.).

Release of metallic cantilever (structural layer) is usually a wet process used to dissolve the sacrificial material [13 – 16]. Removal rate is usually relatively slow because the sacrificial layer is only a few microns thick and the reaction becomes quickly diffusion limited. Simply said, releasing a structure twice as wide will take 4 times more time. However if the etching lasts too long the chemical may start attacking the device structural material too. A first measure to avoid problems is to use compatible material and chemical, where the sacrificial layer is etched quickly but other material not at all. A typical example is given by the DLP (Digital Light Processing) from Texas Instrument, where the structural layer is aluminum and the

sacrificial layer is a polymer [17]. The polymer is removed with oxygen plasma, and prolonged release time will only slightly affect the metal.

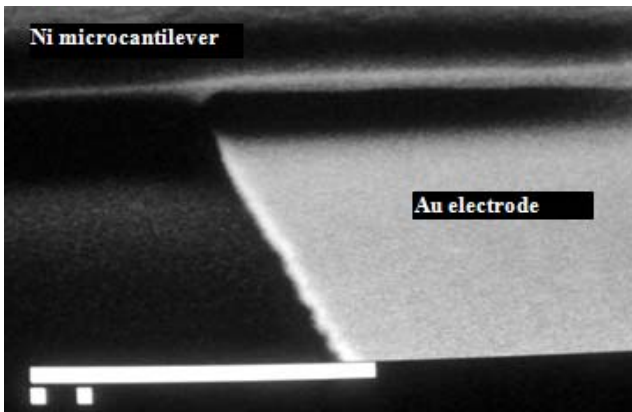
The problems with wet release continues during drying of sample. The meniscus created by the receding liquid/air interface tends to pull the structure against the substrate. This intimate contact gives rise to van der Waals force, which irretrievably pins the structural layer to the substrate at the end of the drying and device will be destroyed. This is referred as static stiction. To avoid this problem several ways are under investigation, e.g., coating the structure with non-sticking layer (e.g., fluorocarbon, hydrophobic DLC film etc.) can be applied. But first off all one should understand changes happening with the surface state to find the best ways to avoid sticking.

Our design of a microelectromechanical switch requires a nickel microcantilever, and we applied a copper layer as suitable sacrificial layer. So, the aim of this work was to investigate the influence of wet release on silicon surface properties in the system Ni (structural layer)/Cu (sacrificial layer). This investigation may help to find out the ways to decrease the stiction phenomenon during the wet release in the surface micromachining technology.

### EXPERIMENTAL

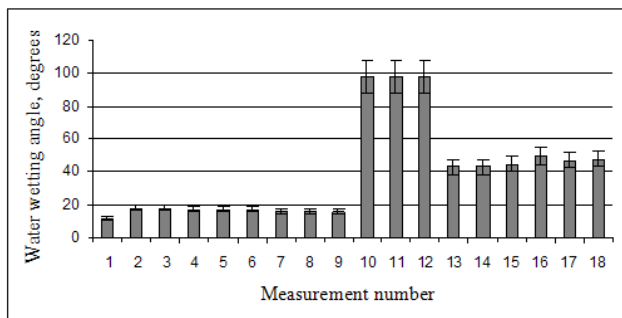
Fabrication of the microcantilever starts from lift-off lithography of the electron beam deposited thin Cr and Au ( $d = 200$  nm) films on Si <100> substrate. A thick sacrificial copper layer ( $d = 5000$  nm) is then deposited following the Cu opening lithography. After the deposition and subsequent patterning of Au ( $d = 200$  nm) layer, electrochemical deposition of a nickel film ( $d = 4000$  nm) is performed. Thick patterned SU-8 resist is used as a mold for the Ni electrochemical deposition. The final step is a wet release of the microcantilever using H<sub>2</sub>O:Cr<sub>2</sub>O<sub>3</sub>:H<sub>2</sub>SO<sub>4</sub> solution. Fig. 1 shows a gap between the Au electrode and the Ni microcantilever after the wet release of the Cu sacrificial layer.

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**Fig. 1.** SEM view of a gap between the bottom Au electrode and the Ni microcantilever after wet release of the Cu sacrificial layer. Mark size is 10  $\mu\text{m}$

The principle of microcantilever operation is based on electrostatic force between the bottom electrode and the microcantilever and the elasticity of beam. When a voltage is applied between two conductor plates, the microcantilever is pulled down due to the electrostatic force and contact closes; when a voltage is turned off, the microcantilever goes back due to the elasticity of beam.  $I-V$  curve of switch show, that after wet release of Cu film the off-contact is not perfect (it shows resistivity close to the on-contact). It means, that wet release is not completed successfully and stiction doesn't let us to fabricate the properly operating device.



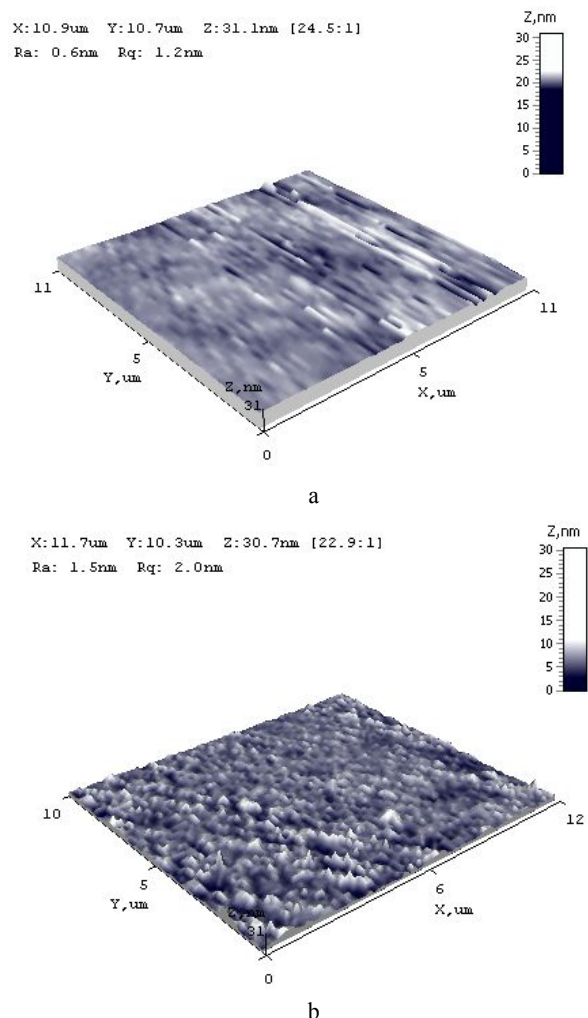
**Fig. 2.** Surface water wetting angle of silicon samples: 1 – 9: numbers of samples after  $\text{O}_2$  plasma treatment; 10 – 18: numbers of samples after removal of Cu film and drying

The stiction phenomenon is determined by the surface properties of the sticking surfaces. To investigate silicon surface state after the wet release of Cu film (in comparison with silicon surface state after usual chemical and plasma cleaning procedure), we have prepared two types of samples: 1) Si  $\langle 100 \rangle$  samples with the deposited and removed Cu film following standart deionized water cleaning; 2) usually cleaned Si  $\langle 100 \rangle$  samples using chemical and  $\text{O}_2$  plasma treatment. The surface of the samples was investigated using measurement of the surface water wetting angle, atomic force microscope (model NT-206: maximum scan field area: up to  $30 \times 30$  microns; measurement matrix up to  $512 \times 512$  points and more; maximum range of measured heights: 4 microns; lateral resolution: 2 nm, vertical resolution: 0.1 nm–0.2 nm) and scanning electron microscope (SEM, Jeol JSM-IC25S, accelerating voltage 25 kV). The chemical structure of

samples was determined using the X-ray Photoelectron Spectrometer XSAM800 Kratos Analytical (information can be obtained from up to the 10 nm depth, angle between the X-ray beam and sample can be changed in  $30^\circ$ – $90^\circ$  range – it enables nondestructive profiling of the surface; deeper profiling can be performed using ion beam etching; dual X-ray anode (Al/Mg) is used; X-ray energy –  $\text{MgK}_\alpha = 1253.6$  eV,  $\text{AlK}_\alpha = 1486.6$  eV).

## RESULTS AND DISCUSSIONS

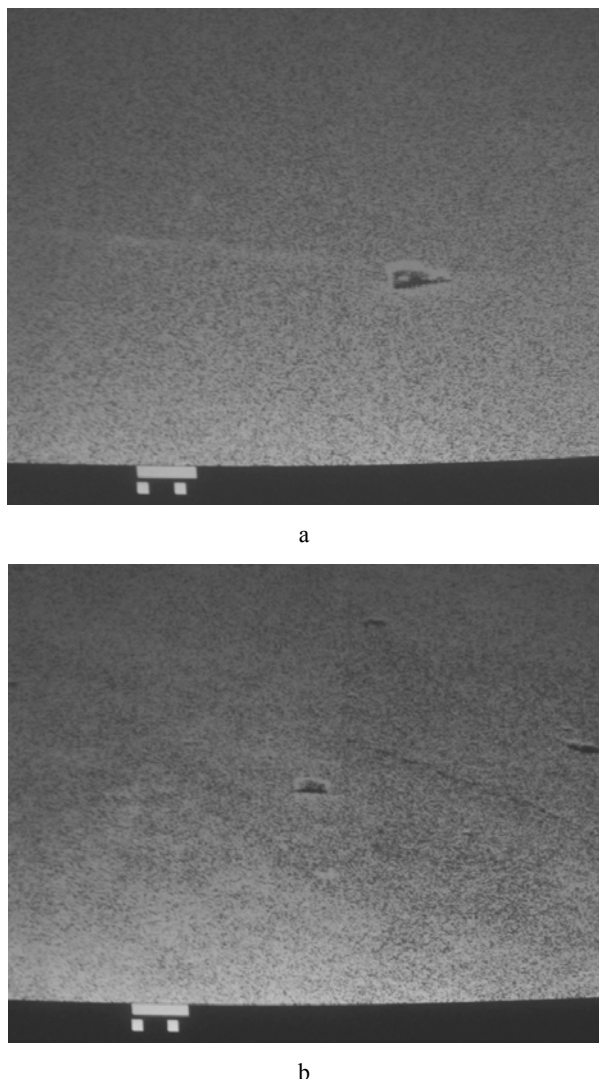
Surface water wetting angle is one of the basic parameters allowing to determine the surface free energy value. The surface is named hydrophilic, if the angle is less than 40 degrees, and it is named hydrophobic, if the angle is more than 90 degrees. The measurements were done using a drop (about 10 ml) of deionized water (A type,  $\rho = 18$   $\text{M}\Omega\text{-cm}$ ). Fig. 2 shows the measurement results of the surface water wetting angle for two types of the samples.



**Fig. 3.** Atomic force microscope measurement of typical samples: a – after  $\text{O}_2$  plasma treatment; b – after removal of Cu film and drying

Measurement show, that the surface of silicon samples after  $\text{O}_2$  plasma treatment is much more hydrophilic than that of silicon samples after removal of Cu film and drying. It means, the Cu etchant (based on sulphur acid) influences

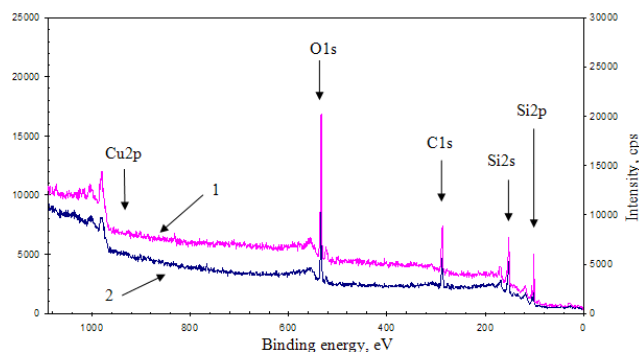
silicon surface bonds (natural silicon dioxide film, etc.) and some stable products of reactions could be expected. Despite the visual view (the surface of both samples seems quite clean) the residual products still can exist.



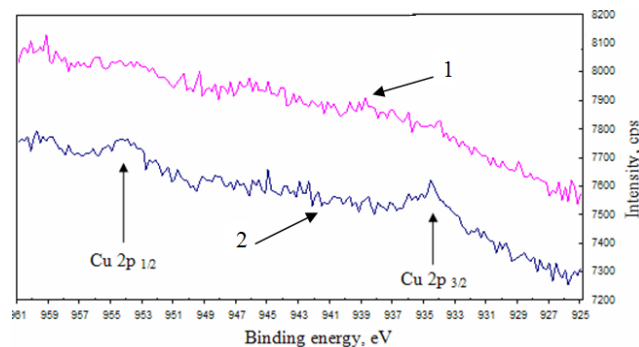
**Fig. 4.** SEM view of typical samples: a – after O<sub>2</sub> plasma treatment; b – after removal of Cu film and drying. Mark size is 10 μm

Atomic force microscope measurements of typical samples after O<sub>2</sub> plasma treatment and after removal of Cu film and drying show that the surface after the O<sub>2</sub> plasma treatment is a bit more flat than that of the sample after removal of Cu film and drying (Fig. 3). This could be a reason of differences of water wetting angle between the samples. However the roughness (*R<sub>a</sub>*) of both surfaces differs only slightly: from 0.6 nm to 2 nm, so both surfaces are very flat from the point of view of visual estimation. SEM photograph in Fig. 4 shows, that the typical sample after removal of Cu film and drying (b) is slightly more rough versus the sample after O<sub>2</sub> plasma treatment (a). The increased microscopic roughness could be determined both by residues of chemical reactions or by residues of organic and inorganic polutions. The aim of X-ray photoelectron spectroscopy analysis was to find out, what is possible cause of the surface state changes after removal of Cu film and drying.

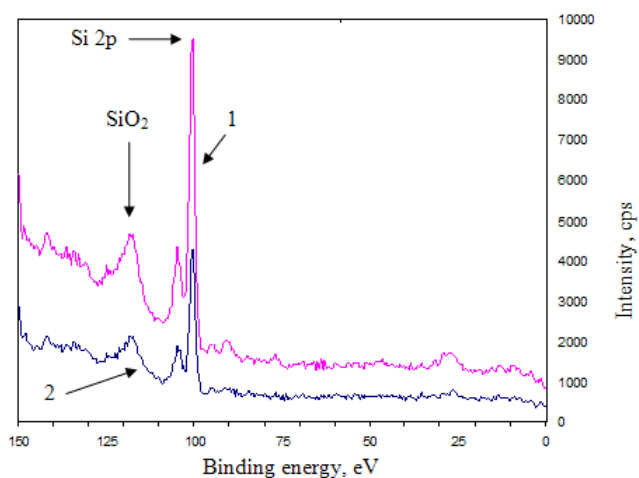
Fig. 5 shows X-ray photoelectron spectra of both typical samples. The same chemical elements (silicon, carbon, oxygen) dominate on both analyzed surfaces and just a very small intensity of copper peak can be detected in spectra of the sample after removal of Cu film and drying. The detailed analysis of the copper spectra (Fig. 6) shows small peaks of copper peaks in the sample after removal of Cu film and drying. Due to low intensity we can't establish chemical bonding of the copper, but one can suggest that the copper as well as chemically bonded copper exist on the surface of that sample determining differences of the surface free energy between the samples after O<sub>2</sub> plasma treatment and after removal of Cu film and drying.



**Fig. 5.** X-ray photoelectron spectra of typical samples: 1 – after O<sub>2</sub> plasma treatment; 2 – after removal of Cu film and drying



**Fig. 6.** The detailed X-ray photoelectron spectra of typical samples for copper lines: 1 – after O<sub>2</sub> plasma treatment; 2 – after removal of Cu film and drying



**Fig. 7.** XPS silicon spectra of typical samples: 1 – after O<sub>2</sub> plasma treatment; 2 – after removal of Cu film and drying

Fig. 7 show the silicon spectra of both typical samples. One can suggest as well, that differences of surface adhesion between the samples after O<sub>2</sub> plasma treatment and after removal of Cu film and drying can be defined by the differences of natural SiO<sub>2</sub> quantity due to the etching residues. The detailed data of the chemical analysis are given in Table 1 and Table 2.

**Table 1.** Data of the chemical analysis for the sample after O<sub>2</sub> plasma treatment

Element, peak	Peak width, (eV)	Peak area	Quantification factor	Atomic conc., %
O1s	2.38	48782.00	0.66	38.91
C1s	2.53	11505.00	0.25	24.22
Si2p	1.70	18880.00	0.27	36.81

**Table 2.** Data of the chemical analysis for the sample after removal of Cu film and drying

Element peak	Peak width, (eV)	Peak area	Quantification factor	Atomic conc., %
Cu2p	2.62	899.00	6.30	0.08
O1s	2.34	37996.00	0.66	32.05
C1s	2.32	15997.00	0.25	35.63
Si2p	1.72	15636.00	0.27	32.24

After removal of Cu film and drying, a small amount of Cu can be found on the silicon surface (Table 2). So we can conclude, that differences of surface adhesion between the samples after O<sub>2</sub> plasma treatment and after removal of Cu film and drying can be determined by copper residues (residual copper can be bonded with other chemical elements) or by difference of natural SiO<sub>2</sub> quantity due to the copper bonding that decreases growth of the natural SiO<sub>2</sub> film.

## CONCLUSIONS

Electrostatically actuated nickel microcantilever was fabricated by surface micromachining technology, using Ni (structural) and Cu (sacrificial) layers. Measurement show, that the surface of silicon samples after O<sub>2</sub> plasma treatment is much more hydrophilic than that of silicon samples after removal of Cu film and drying. Slightly increased roughness was determined by AFM for samples after wet release of Cu film and drying. X-ray photoelectron spectra of typical samples show, that differences of surface adhesion between the samples after O<sub>2</sub> plasma treatment and after removal of Cu film and drying can be determined by copper residues or by differences of natural SiO<sub>2</sub> quantity due to this residues decreasing growth of the natural SiO<sub>2</sub> film.

## Acknowledgments

This research was supported by the Lithuanian State Science and Study foundation.

## REFERENCES

1. **Ostaševičius, V., Tamulevičius, S., Grigaliūnas, V., Kopustinskis, V., Palevičius, A., Gudonytė, A., Daukševičius, R.** Design and Fabrication of a Microelectromechanical Switch *Materials Science (Medžiagotyra)* 10 (4) 2004: pp. 313 – 316.
2. **Senturia, S. D.** Microsystem Design. Kluwer Academic Publishers, Norwell, MA, 2001.
3. Fundamentals of microfabrication. M. Madou, 2nd ed. CRC Press, Boca Raton, 2002.
4. **Wood, R., et all.** MEMS Microrelays *Mechatronics* 8 1998: pp. 535 – 547.
5. **Judy, J. W.** Microelectromechanical Systems (MEMS): Fabrication, Design and Applications *Smart Materials and Structures* 10 2000: pp. 1115 – 1134.
6. **Yao, Z. J., Chen, S., Eshelman, S., Denniston, D., Goldsmith, C.** Micromachined Low-loss Microwave Switches *J. Microelectromech. Syst.* 8 1999: pp. 129 – 134.
7. **Toshiyoshi, H., Fujita, H.** Electrostatic Microtorsion Mirrors for an Optical Switch Matrix *J. Microelectromech. Syst.* 5 1996: pp. 231 – 237.
8. **Osterberg, P. M., Senturia, S. D.** M-test: A Test Chip for MEMS Material Property Measurement Using Electrostatically Actuated Test Structures *J. Microelectromech. Syst.* 6 1997: pp. 107 – 117.
9. **Jensen, B. D., de Boer, M. P., Masters, N. D., Bitsie, E., La Van, D. A.** Interferometry of Actuated Microcantilevers to Determine Materials Properties and Test Structure Nonidealities in MEMS *J. Microelectromech. Syst.* 10 2001: pp. 336 – 346.
10. Engineers' Relay Handbook. National Association of Relay Manufacturers, Milwaukee, WI, USA, 1996: p. 2-1.
11. **Schlaak, H., Arndt, F., Hanke, M., Schimkat, J.** Silicon-microrelay with Electrostatic Moving Wedge Actuator. New Functions and Miniaturisation by Micromechanics *Micro System Technologies 96* Potsdam, Germany, 1996: pp. 463 – 468.
12. **Drake, I., Jerman, H., Lutze, B., Stuber, M.** An Electrostatically Actuated Micro-relay. Tech. Digest. *8th Int. Conf. Solid-State Sensors and Actuators (Transducers '95/Eurosensors IX)* Stockholm, Sweden 25 – 29 June, 1995: pp. 380 – 383.
13. **Simon, I., Salk, S., Kim, C.-J.** Mercury-contact Switching with Gapclosing Microcantilever *Proc. SPL E* Vol. 2882. 15 Oct., 1996. Austin, TX, USA, 1996: pp. 204 – 209.
14. **Taylor, W. P., Allen, M. G.** Integrated Magnetic Microrelays: Normally Open, Normally Closed, and Multi-pole Devices *9th Int. Conf. Solid-State Sensors and Actuators (Transducers '97)* Chicago, USA 16 – 19 June, 1997: pp. 1119 – 1152.
15. **Nagaoka, S.** Compact Latching Type Single-mode Fiber Switches Fabricated by a Fiber Micromachining Technique and Their Practical Applications *IEEE J. of Select. Topics in Quant. Electron.* 5 (1) 1999: pp. 36 – 45.
16. **Comtois, J., Bright, V., Phipps, M.** Thermal Microactuators for Surface Micromachining Processes *In Proceeding SPIE 2642* 1995: pp. 10 – 21.
17. A (Not so) Short Introduction to Microelectromechanical Systems. F. Chollet, HB. Liu. Version 2.0, 2006.