Fabrication of Multifunctional Atomic Force Microscopy-Scanning Electrochemical Microscopy Probe Arrays

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Abstract/Introduction

Combined atomic force scanning electrochemical microscopy (AFM-SECM) probes integrated with micro/nano-electrodes enable simultaneous collection of electrochemical information along with high resolution topological imaging [1-3]. Thereby, an innovative technique for correlating surface chemical activity and topography during a single sample surface scan is provided. In the present research, a batch fabrication process at the wafer level was developed for incorporating an array of four AFM-SECM cantilevers with recessed electrodes. The electrode was located at a defined distance from the tip apex enabling separation of correlated topographical and electrochemical information. The bifunctionality of the individual probes is demonstrated by AFM imaging and electrochemical characterization of the integrated electrodes.

The presented development was at the forefront of combined scanning probe technology providing sub-microelectrode integrated AFM probe arrays for the first time.

Fabrication

Multifunctional cantilever arrays were fabricated from a 4″ silicon-on-insulator (SOI) wafer consisting of a device layer of 10 μm silicon (Si), a stop etch layer of 1 μm silicon dioxide (SiO_2) and a handle layer of 500 μm Si. This procedure is conducted using a batch process involving the development of over three hundred microchips on a single wafer. Each microchip contains four gold (Au) pad openings and four cantilevers of various heights and widths.

First, the device silicon layer AFM tips were fabricated using an isotropic reactive ion etching (RIE) process. The AFM tip surface was insulated by a 500 nm plasma enhanced chemical vapor deposition (PECVD) silicon nitride (SiN) layer. An etch mask for backside Si etch was patterned by successive 6 μm PECVD SiO_2 layer deposition and wet etching. The electrode layer is then patterned using lift-off process of 100Å titanium and 2000Å gold deposited using an e-beam evaporator. Another 500 nm PECVD SiN layer insulated the metal layer. Four gold pads were exposed by an RIE process for electrical connection to individual electrodes for electrochemical measurements. Each cantilever profile was defined by etching the SiN and the silicon device layers with RIE and inductively coupled plasma (ICP) etching processes respectively. The backside of the microchip was then etched through ICP. Each cantilever chip was then removed from the host wafer and focused ion beam (FIB) technology was used to open each tip electrode and shape the AFM tip [1].

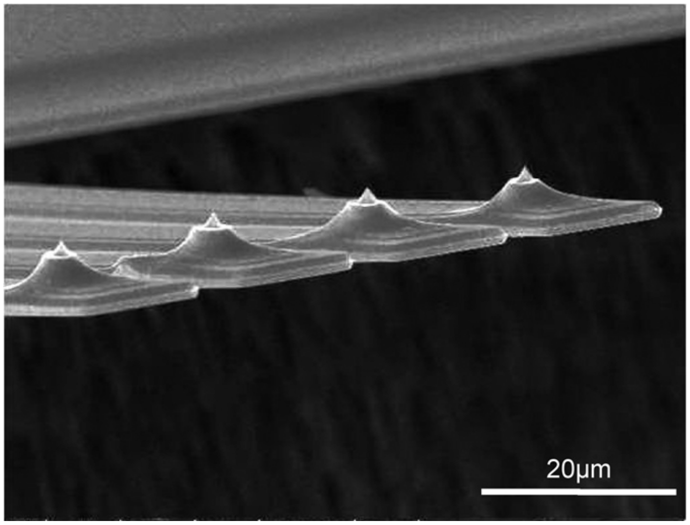

Figure 1: SEM image of a fabricated AFM-SECM probe array.

Results and Conclusions

An AFM-SECM probe array is shown in Figure 1. Each cantilever chip contains four separate cantilevers with 20 μm or 16 μm spacing between each tip apex. The imaging quality of the cantilever was tested.

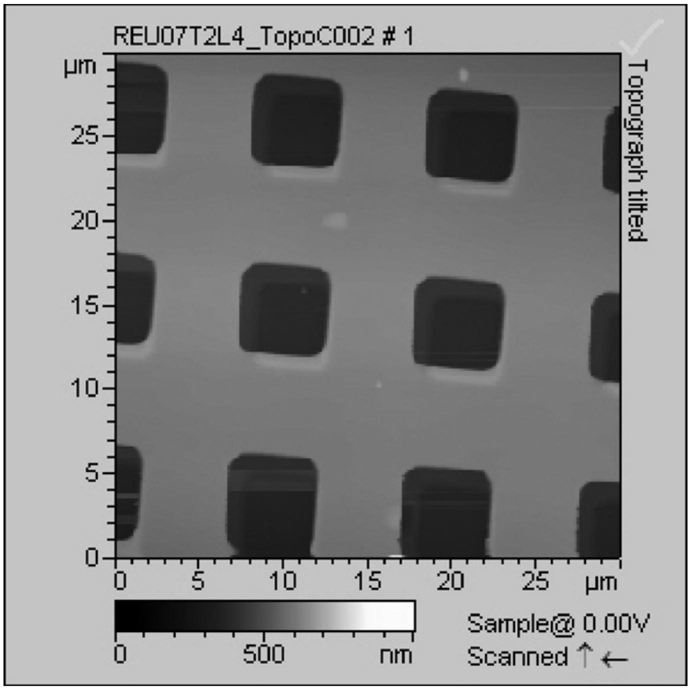

Figure 2: AFM of a test grid recorded with a single cantilever from the array.

The AFM laser was focused on one of the cantilevers and scanned across a model sample; the AFM image is shown in Figure 2. The change in color along the lower edge of each etched region is related to the spot size of the used laser, which results in an overlapping readout of two neighboring cantilevers.

The electrochemical functionality of the cantilever array was then tested by cyclic voltammetry (CV) in 0.1 mol/L $K_4Fe(CN)_6$ in 0.5 mol/L KCL in a potential range from 0V-0.6V. CVs were recorded before and after focused ion beam (FIB) milling (Figure 3). As shown in the voltammograms, the current level before tip milling is in the low pA range, which indicates good insulation. The same test was then conducted after tip milling. The measured steady state current of 1.68 nA is in good agreement with the expected theoretical value for the given electrode size.

Finally the free vibration of the cantilever in air was recorded and the spring constant of the cantilevers was calculated to be 0.95-1.2 N/m which is comparable to that of commercial cantilevers. The frequency response of the cantilevers in air was also conducted using a mean-square displacement which verified that the AFM-SECM cantilevers have a resonant frequency of 69.1 kHz, also comparable to commercial cantilevers [1].

In conclusion, the first known AFM-SECM integrated probe array was successfully fabricated using microfabrication techniques suitable for production on a wafer level. The gold microelectrodes were integrated recessed from the tip apex allowing simultaneous topographical and electrochemical imaging. The AFM-SECM cantilever performance as AFM cantilevers was verified and is comparable to that of commercial cantilevers and electrochemical functionality was demonstrated by cyclic voltammetry.

Future Work

Simultaneous topographical and electrochemical imaging will be demonstrated with one of the four cantilevers of the array. Parallel readouts from each cantilever are anticipated in the future with a modified AFM set-up. Reducing the size of the integrated microelectrode will result in improved resolution for electrochemical imaging. Ultimately, individual modifications of each electrode of the cantilever array with biosensing layers or pH sensing layer will allow multiple parameter readouts during AFM imaging.

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References

[1] C. Kranz, G. Friedbacher, B. Mizaikoff, A. Lugstein, J. Smoliner, E. Bertagnolli, "Integrating an Ultramicroelectrode in an AFM Cantilever: Combined Technology for Enhanced Information"; Anal. Chem., 73, 2491, (2001).

[2] A. Lugstein, E. Bertagnolli, C. Kranz, A. Kueng, B. Mizaikoff, "Integrating micro- and nanoelectrodes into AFM cantilevers using FIB techniques"; Appl. Phys. Lett., 81, 349 (2002).

[3] H. Shin, P. Hesketh, B. Mizaikoff, C. Kranz, "Batch Fabrication of AFM Probes with Recessed Integrated Ring Microelectrodes at a Wafer Level"; Anal. Chem, 79, 4769-4777, 2007.

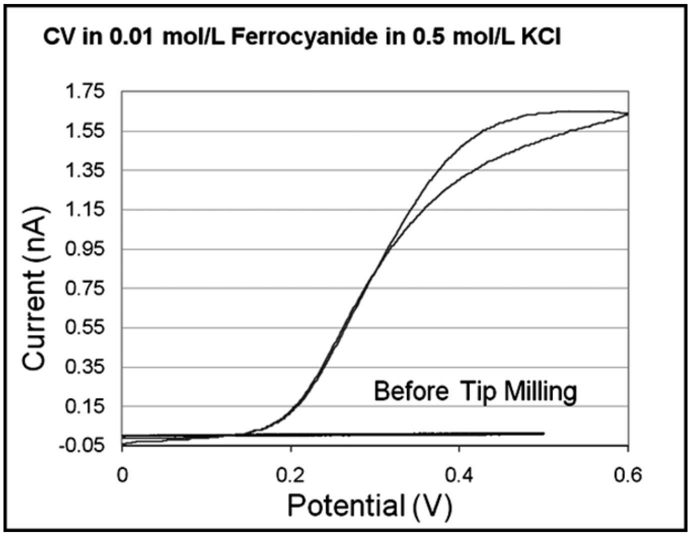

Figure 3: CV recorded at the integrated electrode of the cantilever.