

Characterization of Electrical Fields of Buried Interdigitated Nanoscale Ti-Electrode Arrays by a Novel Atomic Force Microscopy Measurement Procedure and Their Fabrication by FIB Milling

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ABSTRACT

We present in this article the fabrication of 500nm pitch (width and distance of/between the electrodes is 500 nm) and 200nm pitch interdigitated Ti nanoelectrodes by Focused Ion Beam (FIB) chemical enhanced etching with XeF_2 on a quartz substrate. Secondary Electron Microscopy (SEM), contact mode Atom Force Microscopy (AFM) and two-point electrical measurement on the SiO_2 covered nanoelectrodes are performed to show the structure and functionality of the fabricated nanoelectrodes. The electrostatic force field of the fabricated 500nm pitch interdigitated Ti nanoelectrodes was quantitatively investigated with a novel Force Distance Curve (FDC) and AFM based method. The method is described and first measurement results are discussed.

Keywords: AFM, SFM, force distance curves, electrostatic force field, interdigitated nanoelectrodes

1 INTRODUCTION

The recent progress in fabrication methods for nanostructures enabled new applications in different fields. Interdigitated electrode arrays with sub-micrometer size gaps and width are used for instance in impedance spectroscopy of biomolecules [1], DNA biosensors [2, 3], and gas sensors [4]. The reliable measurement of the electrostatic field above these sub-micrometer interdigitated electrodes however is challenging due to the dimensions and material limitations. We present here a fabrication method to produce sub-micrometer size interdigitated electrode arrays on a nonconductive substrate and a new method to measure the electrostatic field above these electrodes with applied electrical potential difference.

2 NANO-ELECTRODE FABRICATION

2.1 Preparation and Equipment

Prior to carrying out FIB operations the substrates were prestructured by standard photolithography in order to add

bonding pads to the nanoelectrodes. First a 60nm Ti layer was sputtered (Spider-600, Pfeiffer Vacuum, D) onto a quartz wafer, followed by a positive photolithography process and a dry etch step with BCl_3 as process gas.

After FIB milling, the interdigitated nanoelectrodes have been covered by sputtering 30nm SiO_2 (Spider-600, Pfeiffer Vacuum, D) in order to realize embedded nanoelectrodes. The contact pads were protected by using a Kepton® tape, in order to keep them uncoated for subsequent bonding. The SiO_2 sputtering process was performed in presence of 10^{-4} mbar of oxygen in order to improve the electrical quality of the SiO_2 layer.

2.2 Fabrication

For the fabrication of the interdigitated nanoelectrodes we used a dual beam FIB electron microscope from FEI (Nova 600 NanoLab), which uses an electron and Ga^+ ion gun. Nanoholes and -squares with a diameter of a few hundreds of nanometers were first FIB milled into the substrate for fine adjustment of focus and stigmatism. To avoid distortions due to surface charging we grounded the Ti-film using a Cu-tape and scanned with the e-beam whilst milling the nanostructures. For good discharging the primary electron current is chosen about 20 times higher than the ion current.

The effects of redeposition that usually occur during FIB milling were counteracted by introduction of XeF_2 gas during the milling process. [5] Figure 1 and 2 display the final 500nm pitch and 200nm pitch interdigitated Ti nanoelectrodes milled in a prestructured sample prepared as described above. The electrodes have well defined edges due to the optimized FIB machining process to minimize the redeposition rate.

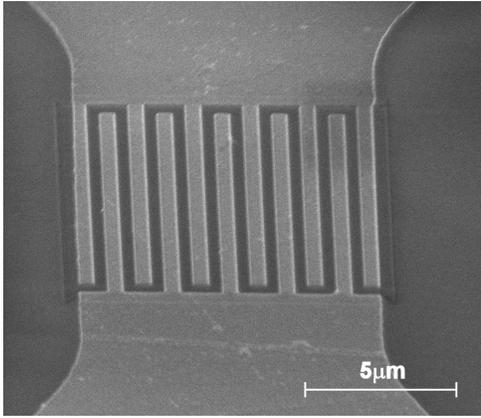


Figure 1: Tilted (52°) secondary electron microscope (SEM) image of the 500nm pitch interdigitated Ti nanoelectrodes.

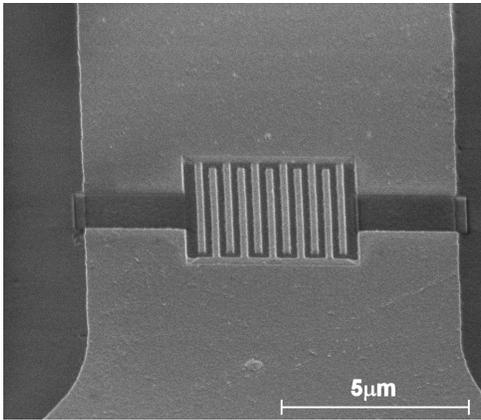


Figure 2: Tilted (52°) SEM view of the 200nm pitch interdigitated Ti nanoelectrodes.

This process for the 500nm pitch interdigitated electrodes has been carried out using the following parameters: number of passes $p = 800$, dwell time $t_d = 1\mu s$, Magnification $M = 3500$, Ion current $I_1 = 10pA$, acceleration voltage $U_1 = 30kV$, no I-beam spot overlap, XeF_2 precursor temperature $T_p = 28^\circ C$. The theoretical Ion-Beam diameter for these conditions is 12nm. The parameters for the e-beam used for charge neutralization were: electron current $I_e = 210pA$ and acceleration voltage $U_e = 2kV$.

For more details of the XeF_2 enhanced FIB milling process and for the fabrication of 50nm pitch interdigitated Ti nanoelectrodes please consult [5].

2.3 Electrical Characterization

The electrical properties of the embedded interdigitated nanoelectrodes were determined from a Voltage – Current diagram taken with a precision semiconductor parameter analyzer (Hewlett Packard 4156A). A DC voltage sweep of -35 to +35 volts with a rate of 15V/s revealed a maximum

measured current of $6 \cdot 10^{-12}$ ampere. This corresponds to a minimum resistance of about $5.8T\Omega$.

3 E-FIELD CHARACTERIZATION

3.1 Force Distance Curve Basics

Almost every AFM allows today performing force distance curves (FDC's). FDC's are widely used their application range covers chemical, biological [6], physical [7], and material applications [8]. For a FDC the deflection of the cantilever as function of its distance from the surface is monitored. The obtained FDC curve is linked to the attraction forces and adhesion forces that determine the interfacial interaction forces between bodies.

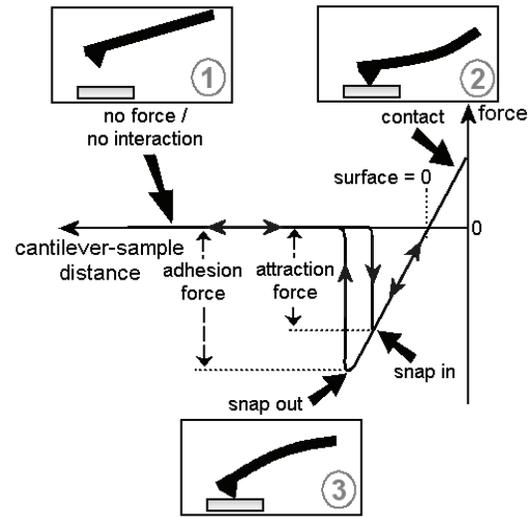


Figure 3: Diagram of a simplified force distance curve.

A simplified FDC is schematically presented in Figure 3. The elastic deformations of the cantilever at different positions depend on the cantilever spring constant and the acting forces on the cantilever. They are schematically shown in the sketches 1 to 3. A detailed description of the FDC measurement procedure and its properties can be found elsewhere. [9]

3.2 Setup

The experimental set-up consists of an AFM (Topometrix, Explorer) with linearised x,y,z piezoelectric actuators installed in a environment regulated glove box. The temperature, humidity and pressure are monitored with a Rotronic HygoPalm3 with calibrated HygroClip SC05. A power supply (HP E3631A, Hewlett Packard, USA) was used for applying voltages between +25V to -25V DC to the interdigitated and aluminum wire bonded electrodes. AFM, Rotronic HygoPalm3, and power supply are controlled and coordinated by a visual basic program

running on a PC. Prior to use, the glove box is flushed at least for 12 hours with pure dry nitrogen at 20 l/min.

For the measurement of the 500nm pitch interdigitated Ti nanoelectrodes a Pt/Ir coated Si-cantilever was used that has a nominal spring constant (k_N) of 1 to 5N/m and a nominal resonance frequency (f_N) of 60 to 100kHz, the tip of the cantilever is 10 to 15 μ m long with a side angle of 22,5° and a tip radius between 20 to 25nm (Nano World, Neuchatel, CH).

For automatic FDC measurement, data acquisition and treatment, programs were written in different programming languages (Visual Basic, Delphi and Matlab).

3.3 Calibration

Calibration of the measuring set-up is a key issue for obtaining reliable quantitative data. Therefore the hardware lineariser for the piezoelectric actuators was calibrated with calibration standards before each measurements series. The real spring constant k_R of the cantilever was determined by measuring the z-piezo traveling distances after contact on two different substrates, a rigid diamond sample (Z_{plate}) and a stainless steel spring sheet (Z_{sheet}) (Young's modulus of $E_{sheet} = 200$ MPa; width $b = 186\mu$ m; thickness $h = 20\mu$ m; free hanging length $L = 2.959$ mm), and using equation (1) and (2). Applying this method we obtained $k_R = 1.23$ N/m as real spring constant. [10]

$$k_R = \frac{k_{sheet} (Z_{sheet} - Z_{plate})}{Z_{plate}} \quad (1)$$

$$k_{sheet} = \frac{E_{sheet} h^3 b}{4L^3} \quad (2)$$

In addition, the system calibration factor (SC) is determined before and after each measuring series. SC connects the z-piezo movement to the cantilever deflection and the output of the photodiode, which registers the deflection of the laser beam. This is done by monitoring the photodiode signal (P_S) with known decrease in z after contact with a rigid surface. The average measured SC was 0.179nA/nm.

3.4 Measurement Procedure and Data Treatment

The interdigitated electrode sample was positioned under the AFM in the glove box and connected via feed through connectors to the power supply. The AFM cantilever was properly aligned in the x-y axis to the electrodes and the system calibration factor SC was measured.

For the FDC - AFM measurements, the nitrogen flux is reduced to 4 l/min. After fine realignment of the cantilever,

remeasuring SC and determination of the coordinates of the to be measured area, the automated process of acquisition of force distance curves (FDC) is started.

At each point in the measured area two FDC's were taken successively with the grounded cantilever, one with grounded electrodes and one with a freely chosen applied voltage of +20V and -20V on the interdigitated electrodes, respectively. Every force distance curve (FDC) was treated individually. The approach and the retraction part were separated and the measured AFM photodiode signal (P_S) (nA) that is proportional to the cantilever deflection values, were divided by SC and multiplied with k_R to get the deflection force in nN. The offsets were removed and the distance values (x-axis) were corrected for the cantilever deflection to get the real distance of the cantilever tip from the surface.

The electrostatic force acting due to the applied electrical potential is bigger than the other attraction forces and can therefore be obtained by subtraction of the approach curve of the cantilever onto the electrodes with applied voltage from the all grounded approach curve.

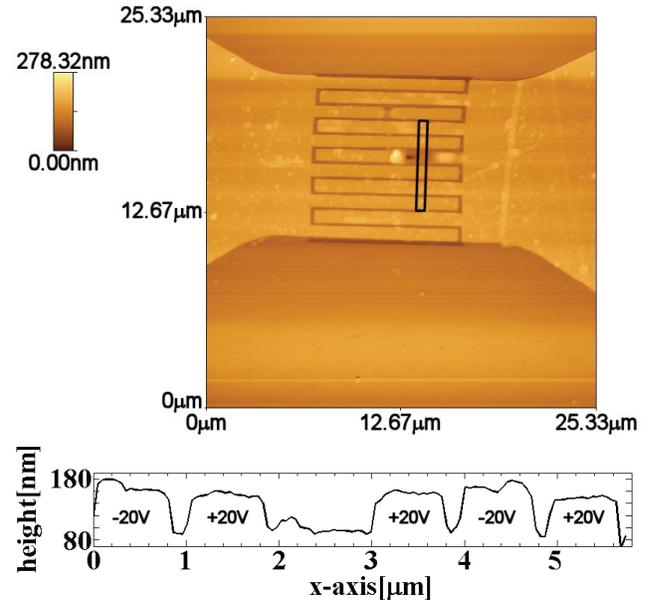


Figure 4: Top graph: AFM scan of the measured 500 nm pitch interdigitated Ti nanoelectrodes. The black rectangle shows the position of the contact AFM line scan that is shown in the bottom graph.

3.5 Measurement Results and Discussion

Figure 4 shows an AFM image and a line scan of the measured 500 nm pitch interdigitated Ti nanoelectrodes. It shows two visible defects within the nanoelectrodes. Figure 5 shows a cross-section through the e-field resulting measured force field perpendicular to the electrode plane of the same sample. This cut was obtained along the same direction as the AFM line scan in Figure 4.

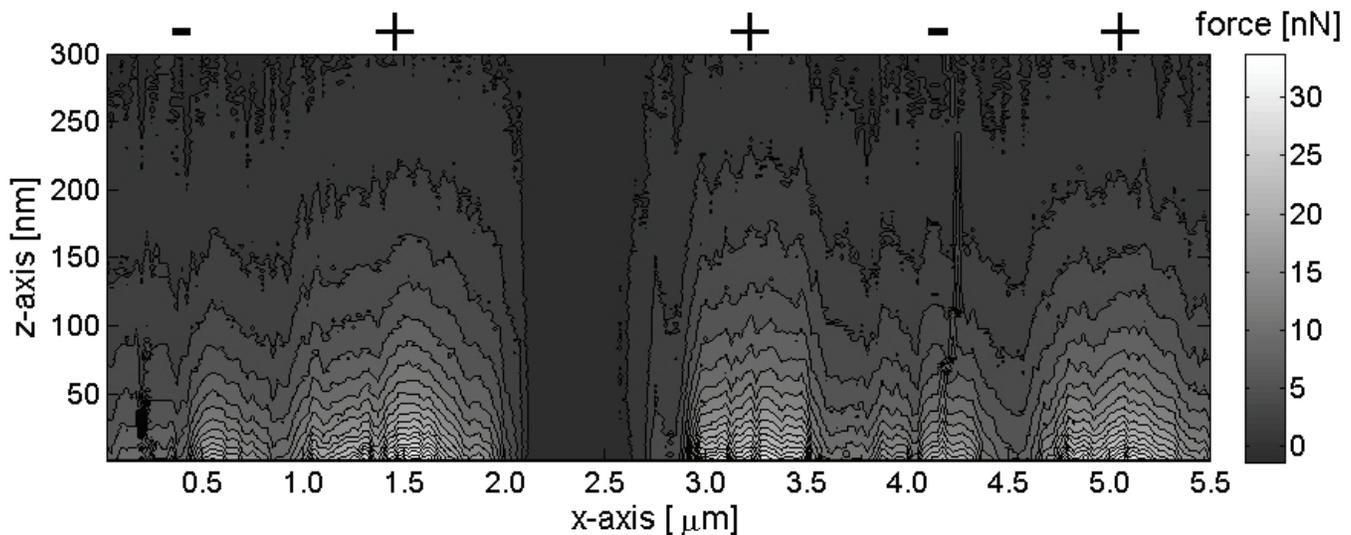


Figure 5: Cross-section of the measured resulting electrostatic force field perpendicular to the 500nm pitch interdigitated Ti nanoelectrodes with +20V and -20V DC applied, respectively.

The positive and negative applied voltage results in slight different force fields, probably due to a slight deviation from zero of the grounded potential of the cantilever. The maximum force on the cantilever shortly before touching the sample is on the electrode with positive potential about 30nN and on the electrode with negative potential about 20nN. The same value of 20nN is reached in a distance of about 20nm above the positive electrode. The electrostatic force on the cantilever above all electrodes becomes immeasurably small in a distance of about 250 to 300nm. This makes us believe that the electrostatic field attracts only the final part of the 10 to 15 μ m high pyramidal cantilever tip and not the cantilever beam. Comparing Figure 5 and 6, both the electrode defect and dimensions are clearly visible. We observe only a squeezing of the x-axis of the measured force field by about 250nm in comparison with the x-axis of the AFM line scan. This corresponds to a displacement mismatch of about 1nm per AFM tip movement in the x-axis, which is small with respect to a total acquisition time of 4h.

Effort to precise the position of the scan area and the numerical simulation and its comparison with the measurements are presently under investigation. [11]

4 CONCLUSIONS

We presented the fabrication of 500nm pitch and 200nm pitch interdigitated Ti nanoelectrodes milled in a prestructured Ti-quartz sample by FIB chemical enhanced etching with XeF₂. A two-point electrical measurement on the SiO₂ covered 500nm pitch interdigitated Ti nanoelectrodes show that they are well electrically insulated. The electrostatic force field of nanoelectrodes

was measured with a novel FDC and AFM based method. It showed quantitatively the forces acting on the cantilever tip at distances smaller than 300nm with an applied potential difference of 40V between the interdigitated electrodes.

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