Nanolab System For Nanoelectronics And Sensors

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ABSTRACT

Nanostructures are interesting for the increasing field of possible applications required by the scaling down of technologies and, in particular, by electronics. The idea is to use nanogap base devices for molecular electronics and sensors where the detection of molecules having comparable dimensions becomes possible and so on. Nanogaps support measurements requested by authors; as a consequence it is necessary to find a method able to produce nanogap small and in a reproducible way. There are different methods to obtain the nanogaps: cutting by FIB, Mechanical Break Junction, Lithography; the Electromigration Induced Break Junction (EIBJ) had been shown a simple process based on cheap technology controlled by an electronic circuit. EIBJ technique is used to make a nanogap from a gold probe by generating a voltage waveform with a feedback control. The Nanolab is the platform in which the basic structures are fabricated becoming the starting point for applications such as biosensors and nanodevices. The NanoLab can be exploited in order to manage general nanostructures called NanoLabElements (NLEs).

Keywords: nanogap, nanostructures, electromigration, feedback control

1 INTRODUCTION

Molecular electronics [1] are really one of the most important candidate to replace standart CMOS technologies; in fact sooner or later scaling down of these technologies will reach limits: the impossibility of achieving geometries under a critical dimension as a consequence of the lithographic limits [2]. Molecular electronics are able to reduce costs and energy; the conduction in molecular devices [3], [4] is associated to a low voltage applied and low currents, so reducing the required power. In this work it will be described the realization of the chip containing the structures in which the nanogaps will be produced, the process methodology, the customized feedback controlled system, for managing fabrication, and the molecular binding.

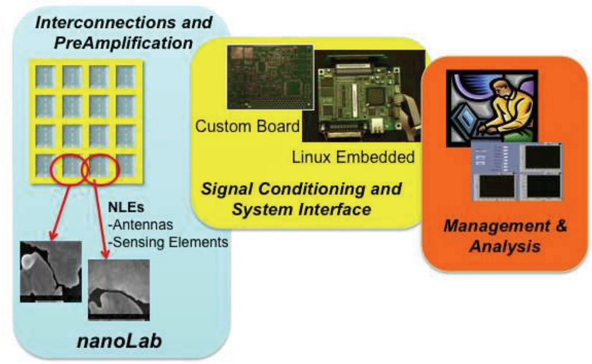

Figure 1: NanoLab structure composition: in the chip there are eight NLEs for experiments realization, its high level operations are made by a Linux Embedded system connected to a control station for user interfaces.

2 NANOLAB

The NanoLab structure [5], [6] is made by an elements array that allows many experiments for fabricating nanoelectrodes and for measuring molecular devices. It has composed of three parts (Fig.1):

- NanoLab is the core of the system which contains areas devoted to host the realization of custom nanostructures called NanoLab Elements (NLEs), in this particular case the gold probes in which nanogaps will be built.
- Signal Conditioning and Interfaces is for input/-output generation thanks to a custom board developed for the specific experiment and for signal conditioning interfaces. The board is connected to a Linux Embedded system useful for high level operations, implementation of control algorithms and interfacing the system to the Control Station.
- Control Station contains user interfaces and is interconnected to the Embedded System by wireless, to remotely controll the experiments.

This system allows to control the construction of the nano/micro devices (nanogaps) and to manage the experiments that will use them as the molecular binding.

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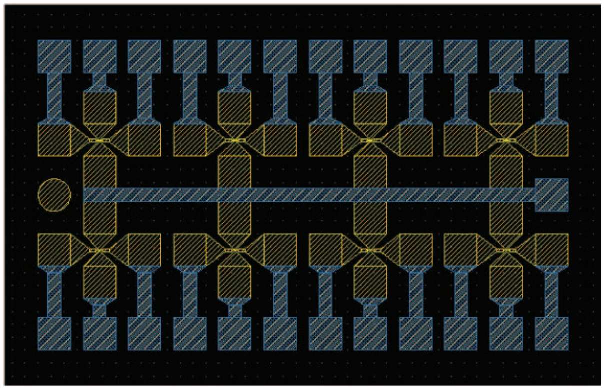

Figure 2: The projected chip mask containing eight nanostructures for experiments realized in gold (yellow parts) with alluminum connections to the pads (light blue parts) for bonding.

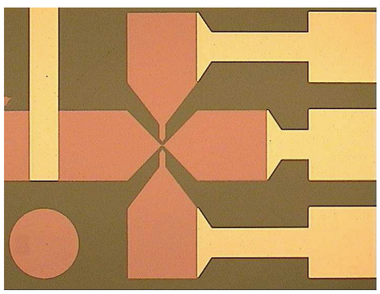

Figure 3: A structure of the realized chip (note that the gold is in red and the alluminum in yellow).

The NanoLab can be inserted in a controlled environment to realize all the experiments without manipulations, avoiding risk of contaminations.

3 EXPERIMENTAL

The chip is based on eight structures each of them contains one probe for electromigration process and two more elettrodes usefull for other experiments where molecules are gated. Electrodeposition with Crono-Potentiometry (ECP), Electrochemical Etching (EE) and Field Emission Electromigration(FEE) [7], [8] can be also used to optimize the electrodes geometries and gate structures. Moreover with this chip can be done experiments of molecular bonding by insering in the realized nanogaps a solution made by buffer and conductive molecules. In figure 2 is showed the chip mask and in figure 3 there is a zoom of a structure of the realized chip.

3.1 Silicon Chip Realization

The process for building probes is based on the evaporation of a gold layer on a self-assembly MPTMS monolayer that serves as adhesion layer between gold

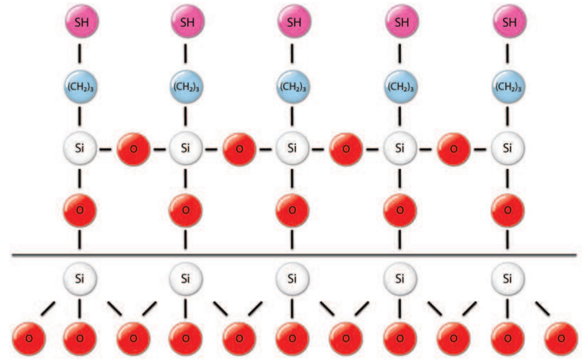

Figure 4: The self assembly MPTMS adhesion monolayer on the SiO_2 surface

and the silicon dioxide of the wafer. The fabrication of the chip ends with the evaporation of 200 nm of Al [9] for allowing the bonding of the chip's pads to a PCB. The realization of the chip starts from a n-type silicon wafers (B doped) on which are executed the following steps:

- Surface oxidation: (in a oxidation oven, Tempress) at 383 K for 4 h 30 min to grow 200 nm of SiO_2.
- Wafer cleaning: by piranha solution (3:1 H_2SO_4, H_2O_2) for 10 min than the wafer is rinsed with deionized water and dried with N_2.
- Surface activation in plasma of oxygen: this treatment is to create a surplus of oxygen atoms on the surface of silicon dioxide (applied power of 300 W for 30 s, oxygen flow 300 sccm).
- Hydroxylation: the wafer is immersed for 10 min in a 1:1 solution of H_2O_2 and HCl at 358 K. After this step the wafer surface exposes -OH groups.
- MPTMS deposition: in a vacuum chamber (p = 0.1 Torr) the surface of the wafer is exposed to vapors that create a monolayer of MPTMS on the surface of gold. This molecule has been chosen cause it is able to form strong bond with gold atoms, to realize a self assembly monolayer on the SiO_2 (Fig.4) and cause it is not conductive (the only layer we want to electromigrate is the gold one, so it is necessary to avoid any possible alternative path).
- Au deposition: a layer of 25 nm of gold is evaporated at 300 K and a pressure of $2 * 10^6$ $Torr$ (E-beam Evaporator ULVAC EBX 14-D).
- Photolithography: deposition of Image Reversal Resist (AZ5214E of Microchemicals) and soft bake. The resist is than exposed using a UV wavelength of 365 nm and using a specifically realized mask.

After the wafer is subjected to a reversal bake and a new UV exposure without a mask. The resist is developed using a 4:1 solution of H_2O and developer basic AZ351B (of Microchemicals).

- Au etching: in a solution of Potassium iodide (KI) 4GR Iodine (I_2) 1 g water 80 ml.
- Resist strip: using Acetone and isopropanol and drying of the wafer with nitrogen.
- Photolithography: deposition of positive resist (HPR 504 of Fujifilm). The resist is subjected to a soft bake. UV exposure occurs with a wavelength of 365 nm and using a mask made specifically for aluminum. The resist is using a 1:1 solution of H_2O and developer basic AZ (of Microchemicals).
- Al deposition: a layer of 200 nm of Al is evaporated at 300 K and a pressure of $2 * 10^6$ $Torr$ (E-beam Evaporator ULVAC EBX 14-D).
- Al lift-off: it occurs in acetone in ultrasonic bath, followed by rinsing in isopropanol alcohol.

After these fabrication phases chips are cut and separated from each others and are individually attached to a PCB. Through bonding the connection between the chip and the PCB are obtained and in this way its possible to perform experiments linking the NanoLab chip with the interface electronics for electromigration control.

4 RESULTS AND DISCUSSIONS

4.1 Nanogap Fabrication

The electromigration in gold wires [9], [10] is generated inducing a current density of about 10^8 A/cm^2 for this reason a controlled waveform must be applied for giving to the probes the necessary current avoiding risk of melting or some other effects that can destroy the probes due to excessive current and thermal runaway.

Voltage (current) can be applied with different waveforms (Fig.5):

- *Step waveform*: a step by step voltage;
- *Square waveform*: a square waveform of a constant amplitude;
- *Square/Step waveform*: a step waveform with a square added on;

A correct control of the voltage waveform can lead to a nanogap under 5 nm, a large number of experiments stands out that the *Square/Step waveform* is the best in term of number and yield more than 80 percent of positive results. In fact *Step waveform* produces small size nanogaps, around 5-6 nm, but the rupture fronts are very jagged, the *Square waveform* is more powerfull and creates larger gaps, around 10 nm, but breaking fronts are more regular and more suitable for molecular binding. A reasonable cause could be due to the fact that it is a midway between the two basic waves and embodies the positive characteristics of both. In figure 6 is showed a 3,5 nm nangap that is the best one we have obtained serving the *Square/Step waveform*, the breaking trend of the specimen is shown in the chart (Fig. 7). During the nanogap realization the software control reads current, calculates the resistance, and generates an increasing step wave up when the resistance does not increase of a predefined percentage value, otherwise the feedback control system is activated. Moreover the algorithm removes the bias voltage and restarts up when it is clear that the increase of the resistance is due to electromigration effect.

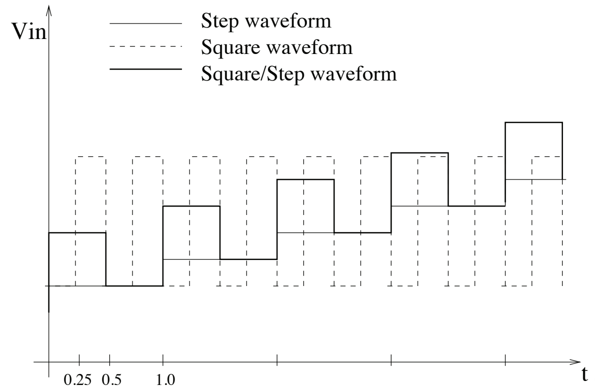

Figure 5: The trends of the waveforms used for nanogaps realization. The voltage value of the Step is of 2 mV and that of the Square is of 5 mV

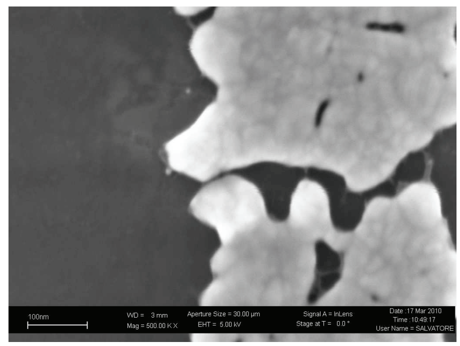

Figure 6: FESEM image of the best obtained nanogap having a dimension of 3,5 nm

4.2 Molecular Binding

A first series of experiments for Oligothiophene I-V measurements has been done by mean Keithley's 6517B Electrometer. The Metal-Molecular-Metal junction had been obtained by inserting in a nanogap a buffer solution of THF and Oligothiophene and checking the molecules

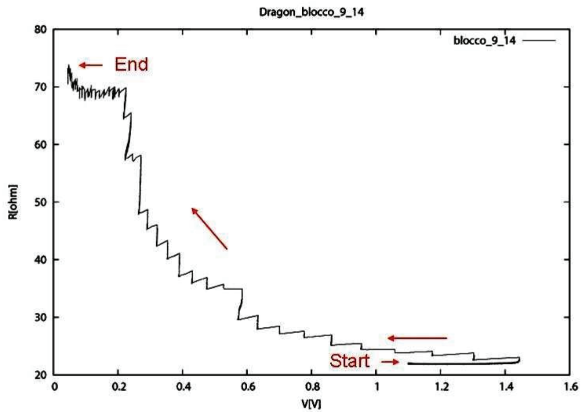

Figure 7: A V-R graphic obtained during a controlled electromigration breaking in which are shown the numerous feedback activation up to the realization of the nanogap

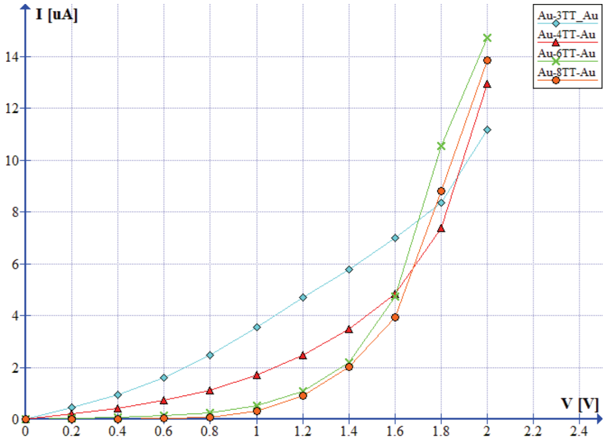

Figure 8: I-V characteristic of the Au-nTT-Au structure with thiophene rings number (n) higher than 2

binding by impedance measurements. First obtained results showed that a bonding between the molecule and the two metal parts occurred, even if the resistances are not completely in accordance with physical simulations. In figure 8 the I-V curves for different length of the Oligothiophene molecules (number of thiophene rings) are presented; showing the high level of current that can be obtained also for long chains: this is mainly due to the internal hopping mechanism of conduction that does not display an exponential degradation of the conductance with the chain length. This behaviour is particulary usefull when the nanogap dimensions can vary of few nanometers with accettable current level. Measurement were initially performed on THF applying 2 V and the resistance value was about 1 GΩ; than a second series of measurements has been done inserting also Oligothiophene repeating the measures with an input voltage starting from 0 to 2 V. The result point out that the measures are deeply affected by the metal molecules interfaces so a greater care must be spent to improve these interfaces.

5 CONCLUSIONS

The NanoLab has been realised, starting from the chip that allows a large number of experiments thanks to its particular structures: NanoLabElements. By serving the NanoLab for experiments management were fabricated nanogaps, and the best obtained results is of a 3,5 nm gap. Moreover the chip of the NanoLab, thanks to its shape allows to put solutions in nanostructures (NLEs), on this basis first tests on molecular bonding have been performed and the results are promitting. Now it is required a huge work to reduce the gaps dimension and improve the molecular conduction. Finally the authors would like to say thanks to Professors Luisa Schenetti, Adele Mucci of the Modena University for Oligothiophene synthesis and to the thesis of Fabrizio Maggiulli and to Dr. Davide Bardella, it is also with their support that this work has been realized.

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