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MATERIAL CHARACTERIZATIONS FOR MEMS VIBRATION SENSORS AND BIOSTRUCTURES APPLICATIONS

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Despite the MEMS/NEMS devices are used in many applications, a lot of new characterization and testing methods have been developed in order to improve their functionality, reliability and stability. The correct material selection criteria are essential when designing micro/nano structures. The material properties of micro components depend on the the manufacturing and processing conditions. This article presents the investigations of two materials obtained by diverse deposition techniques, for manufacturing of MEMS used as vibration sensors and in bio applications. LPCVD undoped and doped polysilicon layers with a thickness varying from 50 nm to 2 μm and a biocompatible polymeric material (SU-8) with a thickness of 10 μm and 20 μm were investigated using Atomic Force Microscope (AFM), nanoindentation techniques, X-ray Diffraction System **(**XRD) and Scanning Electron Microscopy (SEM) characterization tools. In order to demonstrate the applicability of the investigated materials two types of MEMS structures were manufactured.

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1. Introduction

The reliability tests are significant issues for the successful product of micro-electro-mechanical systems (MEMS) either as sub-components or as standalone products. Usually, micromachined components have been manufactured splitting the design and fabrication processes from packaging and reliability difficulties. Exposure of MEMS to shock environments can occur during fabrication, deployment, or operation [1-2].

Reliability depends on the compatibility of the various parts with respect to the desired functionality, and the designs and materials from the standpoint of long-term repeatability and performance accuracy [1-2]. Reliability testing provides techniques for compensation, and an understanding of the failure mechanisms in microsystems. Since MEMS/NEMS invariably are composed of movable micromachined 3D mechanical structures, the thermo-electro-mechanical interaction of components greatly affects their overall performance and reliability during their operation [1-2].

To improve the stability, the lifetime and reliability of MEMS devices, a lot of new characterization and testing methods have been developed. A fundamental understanding of surface topography properties, roughness, friction and mechanical properties of used materials at

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micro and nano scale is expected to bring improvements in the design and functionality of MEMS devices [3]. The mechanical properties of the involved materials influence the tribological performances of the surfaces of the structural parts of the micromachined devices. The materials used and failure mechanism at meso-micro scale require more investigations when designing and manufacturing micromachined device, such as resonant sensors [4-11]. Additionally, the material properties of micro components depend on the fabrication conditions.

This paper presents materials obtained by different deposition techniques, analysis and characterization used for MEMS vibration sensors and for MEMS biostructures applications. The results regarding the analyses of surface topography and mechanical properties of structural materials used for MEMS fabrication are reported. The paper presents the material processing conditions which are varied. Different test methods were applied for the identification of the surface and mechanical properties of separate types of samples.

Five types of LPCVD undoped and doped polysilicon layers with thicknesses of 50 nm, 150 nm, 180 nm and 2 μm and a biocompatible polymeric material (SU-8) with thicknesses of 10 μm and 20 μm were investigated. The films were fabricated as following: Polysilicon and SU-8 layers were deposited on $Si/SiO_2/Si_3N_4$ or Si/SiO_2 substrates. In order to analyse their properties, topography scans were carried out using an Atomic Force Microscope (AFM). Mechanical properties of the materials, such as Young's modulus, have been investigated also using the nanoindentation technique. The cristallinity of the materials and the variation of the grain sizes were measured using X-ray Diffraction System **(**XRD) and Scanning Electron Microscopes (SEM) characterization tools. In order to demonstrate the applicability of the investigated materials two types of MEMS structures were manufactured.

2. Experimental details

2.1 Materials

We have characterized two different materials in order to evaluate the processing parameters and to improve the fabrication stability of MEMS/NEMS devices.

The first studies consisted in testing of five different polysilicon runs. LPCVD undoped and doped polysilicon layers deposited at 580^{o}C and 610 ^{o}C, with four thicknesses of 50 nm, 150 nm, 180 nm and 2 μm were investigated.

The second considered material was a biocompatible polymer, SU-8 2000 which is an improved formulation of epoxy based photoresist SU-8 that has been widely used by MEMS/NEMS producers for many years. SU-8 is use for micromachining and other microelectronic applications, where a thick, chemically and thermally stable image is desired [12]. SU-8 2000 is best suited for fabrication of structures with a wide range of thickness.

2.2 Fabrication process of Polysilicon and SU-8 layers

Five different polysilicon runs were manufactured. In all cases, the thin films were obtained by low pressure chemical vapor deposition (LPCVD) technique, by silane (SiH_4) decomposition. During the deposition process, we have changed different parameters like deposition temperature, silane flow and deposition pressure. These factors directly influence the morphological properties of the material.

We investigated also the dependence of the material properties of the doping concentrations and annealing conditions for polysilicon films. Measurements were performed after each step. A significant impact on the morphological properties was observed.

We tested different thicknesses of polysilicon layers in order to be used as electrods and structural layers for a vibration sensor.

First polycrystalline silicon thin layers, with a thickness of 50 nm, was deposited at a temperature of 580^{o}C, 20 sccm silane flow and a pressure of 0.2 mbar on thermally oxidized silicon silicon wafers. For this run, we have used a thermally oxidized silicon substrate.

Secondly, we deposited polycrystalline silicon thin layer, keeping the same deposition parameters, less the temperature. We have increased it to 610 ^{o}C, in order to study the polysilicon grain sizes. We used the Si/SiO_2 substrate.

For the third run we have increased the thickness from 50 nm to 150 nm; we have kept the silane flow constant and decreased the temperature to 580°C and the pressure to 0.18 mbar. In this case, the substrate was $Si/SiO_2/Si_3N_4$.

The forth polysilicon thin film (180 nm thickness) was obtained at 610 °C, 20 sccm silane flow and 0.2 mbar pressure. In this case the substrate was also $Si/SiO_2/Si_3N_4$.

The last deposition was for a structural layer of 2 μm polysilicon, at 580 °C, 25 sccm silane flow and 0.267 mbar pressure, on an oxidised silicon wafer. For these parameters, we have calculated a theoretical deposition rate of 15.3 nm/min. The deposition conditions for each run are presented in Table 1.

Table 1. The deposition conditions of polysilicon for different runs.

Polysilicon films	**Films thicknesses (nm)**	**Deposition temperature (°C)**	**Silane flow (sccm)**	**Deposition pressure (mbar)**
Si/SiO_2/Poly: Run 1	1700/50	580	20	0.2
Si/SiO_2/Poly: Run 2	1700/50	610	20	0.2
$Si/SiO_2/Si_3N_4$/Poly: Run 3	440/300/150	580	20	0.18
$Si/SiO_2/Si_3N_4$/Poly: Run 4	1700/300/180	610	20	0.2
Si/SiO_2/Poly: Run 5	1700/2000	580	25	0.267

After manufacturing the polysilicon films, we doped all samples using a phosphorus source, in nitrogen atmosphere. Depending on the initial deposition temperature and layer thickness, we have chosen different temperatures and doping times (Table 2). The last step consisted in the annealing treatment, which was performed at 900 °C.

Table 2. The doping conditions of polysilicon for different runs

Polysilicon films	**Doping source**	**Doping temperature (°C)**	**Doping time (min)**
Run 1 and 2	P	1000	30
Run 3	P	875	10
Run 4	P	875	15
Run 5	P	1000	30

The other material used in experiments and analized as structural layer was SU-8, a biocompatible polymer which was used to develop MEMS based microgrippers, for biocells manipulation.

In order to obtain a structural layer of 10 μm thickness and 20 μm thickness, respectivelly, the SU-8 polymer was deposited on Si/SiO_2 substrate using spin coated process. Two layers of SU-8 2010 and SU-8 2025 were spun on to the substrate (silicon wafers). A relaxation period of 10 minute were folowed allowing for the back fill of air pockets. The uniformity of the films increased in such a way.

A hotplate with good thermal control was used during the Soft Bake step of the process. The Soft Bake procedure consist of 1 minute at the temperature of 65 °C and 2 minutes at 95°C (SU-8 2010). For SU-8 2025 the bake was 4 minutes at 95°C. The wafers were then cooled for 30 minutes, returning to ambient temperature.

The SU-8 is then exposed to UV using a mask in order to test the quality of the vertical walls, via a 360 nm optical filter.

After exposure, the layers were prepared with the Post Exposure Bake procedure, which consists of a 3 minute temperature ramp from room temperature to 65 °C, 1 minute at this temperature and 3 minute at a temperature of 95 °C. For SU-8 2025 were used 4 minutes (baked) at 95°C.

The resulting pattern is developed in a proper solvent for 4-5 minutes with a strong agitation of the wafer. A hard bake or final cure step is added to ensure that SU-8 2000 properties do not change in actual use. The wafers were baked at 175 °C for 15 minutes to cure the polymer.

2.3 Characterization

The surface morphology of the polysilicon layers was characterized by a commercial Atomic Force Microscope (Ntegra, NT-MDT Company). AFM measurements were carried out in semi-contact AFM mode with HA-NC etalon probes manufactured by NT-MDT. The scanned areas were 5 µm x 5 µm with 1024 x 1024 sampling points for different location on the same sample (Fig. 1 and 2).

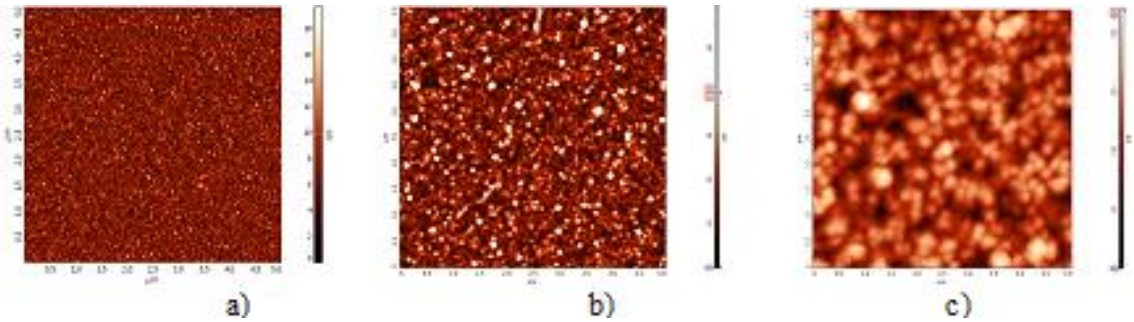


Fig. 1. AFM 2D images for polysilicon layer of samples processed at 580ºC with a thickness of: a) 50 nm; b) 150 nm; c) 2 µm. (scaned size 5 x 5 µm)

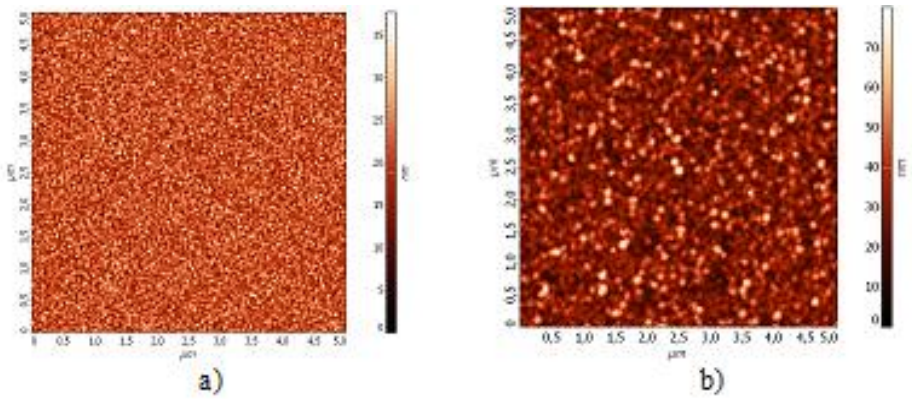


Fig. 2. AFM 2D images for polysilicon of samples processed at 610ºC: a) 50 nm; b) 180 nm. (scaned size 5 x 5 µm)

Corresponding characterization of the surface topography were performed using AFM. We analized the grain size for each sample. The grains zise increase while the thickness or/and the deposition temperature is increasing (Table 3) .

The root mean square for the doped polysilicon processed at 580 ºC with 2 µm thickness is Sq = 25-26 nm [11], while for the undoped polysilicon processed at 580 ºC with 50 nm thickness is Sq = 2-3 nm.

Table 3 Root Mean Square (Sq) values obtained using AFM for polysilicon and grain sizes

Thickness	Undoped 50 nm (580 ºC)	Undoped 50 nm (610 ºC)	Doped 150 nm (580 ºC)	Doped 180 nm (610 ºC)	Doped 2 µm (580 ºC)
Sq	2.07 nm	3.4 nm	6.7 nm	10.6 nm	25 nm
Average grain size	19 nm	32 nm	59 nm	83 nm	216 nm

The material obtained in different runs were measured and characterized using SEM (Fig. 3). We investigated the thickness of samples for each type of deposited polysilicon and the results were: 50 nm, 157 nm, 182 nm and 2 µm. For the SU-8 layers we obtained from SEM evaluation a thikness of 8.48 µm and 19.5 µm, respectivelly.

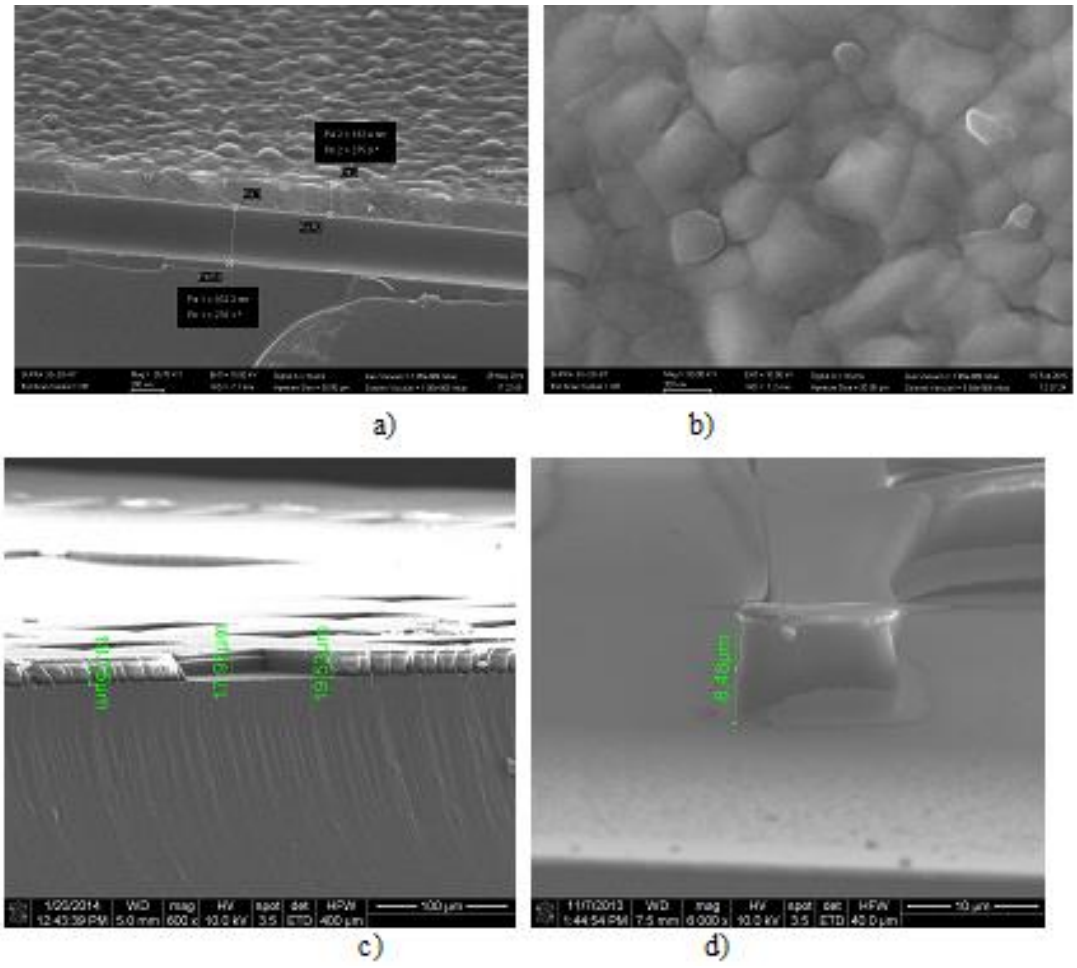
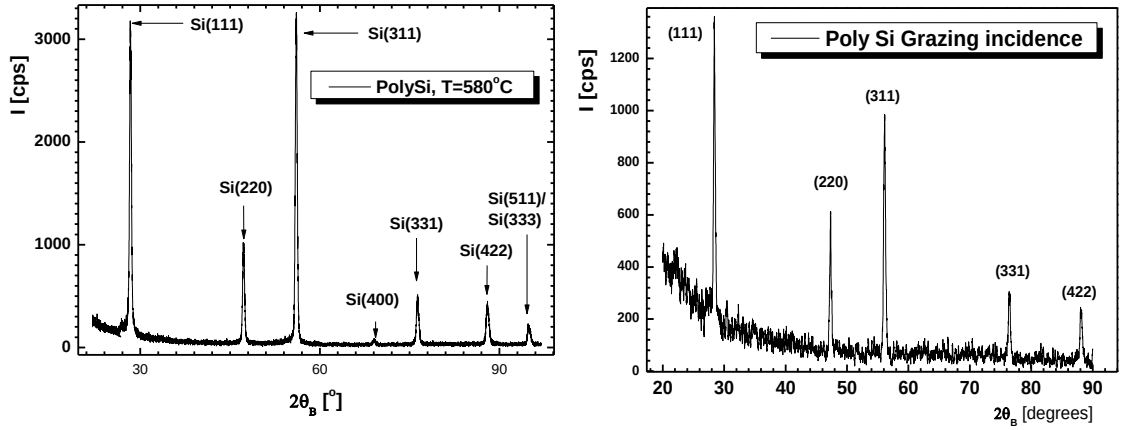


Fig. 3. SEM images: a) Doped polysilicon layer with a thickness of 182 nm (thickness measurement); b) Detail for the 2 µm polysilicon layer deposited at 580ºC at 200 nm scale; c) SU-8 layer of 20 µm thickness; d) SU-8 layer of 10 µm thickness.

Difraction spectra XRD measurements were performed for the 2 µm polysilicon layers processed at 580 ^{o}C (Fig. 4). The cristallinity of the undoped polysilicon sample 580 ^{o}C is more than 98 % while for the doped polysilicon is 100%. The crystallite size for undoped polysilicon at 580^{o}C is 20.96 nm, while for the doped polysilicon is of 24.21 nm. A strain of 0.138 % is observed from XRD measurements for the doped layer.



a) b)

Fig. 4. Difraction spectra XRD measurements for: a) undoped 2 μm polysilicon sample deposited at 580 °C; b) doped with phosphorus of 2 μm polysilicon sample at 580 °C

Using the nanoindentation techniques, the Young's modulus values have been measured for the 2 μm undoped and doped polysilicon layers obtained at 580 ºC and also for the SU-8 layers.

Nanoindentation tests have been carried out using a Nanoindenter G200 (Agilent Technologies) equipped with a Berkovich indenter with a nominal tip radius of 20 nm. Continuous stiffness measurement (CSM) tests were performed in order to obtain and evaluate the Young's modulus as a function of depth. The influence of the testing parameters, especially the strain rate, on the measurements was checked and the parameters were optimized. 20 indentations at a strain rate of 0.01 s^{-1} have been carried out on each specimen. In order to evaluate and minimize the influence of surface morphology on the measurements, the range of indentation depths was much greater than the characteristic size of surface roughness of the film specimens [11].

The averaged modulus values have been calculated. The Young's modulus stabilizing after approximately 400 nm at a value close to 130 GPa for the undoped polysilicon [11] and close to the 143.8 GPa for the doped polysilicon layer (Fig. 5).

The various factors affecting this behavior and underlying the difference between the film specimens are the surface roughness, the internal stress of the films, the possible phase transformations occurring under high pressure conditions in the load range used in the tests and not ultimately the potential preferential orientations in the grains forming the films [9, 11].

The Young's modulus measurements for the SU-8 layer were evaluated faster after approximately 50 nm of depth at a value close to 5.5 GPa (Fig. 6).

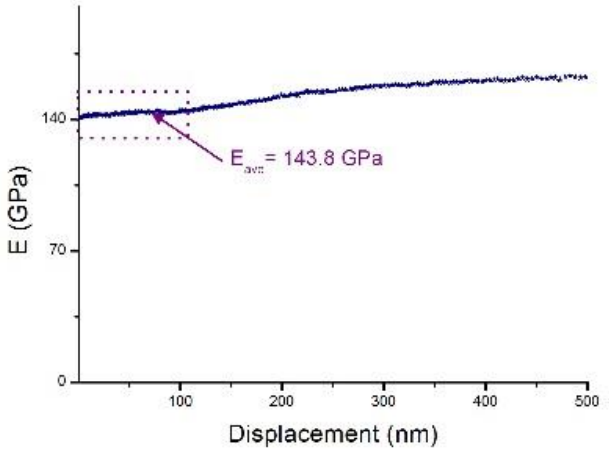


Fig. 5. Young's Modulus vs. depth for doped polysilicon films deposited by LPCVD technique at 580ºC

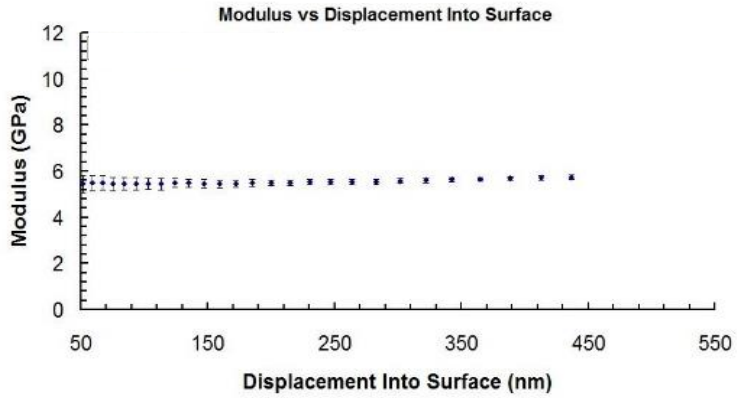


Fig. 6. Young's Modulus vs. depth for SU-8 layer

3. Results and discussion

The two different materials were investigated with the aim to be used for manufacturing of vibrational sensors, clamped beams, and micromanipulators for biostructures applications, like micro grippers. In order to obtain a reliable vibrational structure is necessary to know the uncertanties which influence the response of the final device.

The first experimental measurements started with thin films of polysilicon, deposited at different temperatures. The roughness, crystallite size, cristallinity and mechanical properties were investigated for a better understanding of the behaviour of materials used for MEMS/NEMS fabrication (in our case used as electrods of a vibrational sensor).

For 50 nm polysilicon thin film, doped using a phosphorus source, discontinuity were observed. Optical and AFM investigations have shown that the treatment at 1000^{o}C for 30 minutes have damaged the thin polysilicon film deposited at 580 ^{o}C, making it discontinuous (Fig. 7).

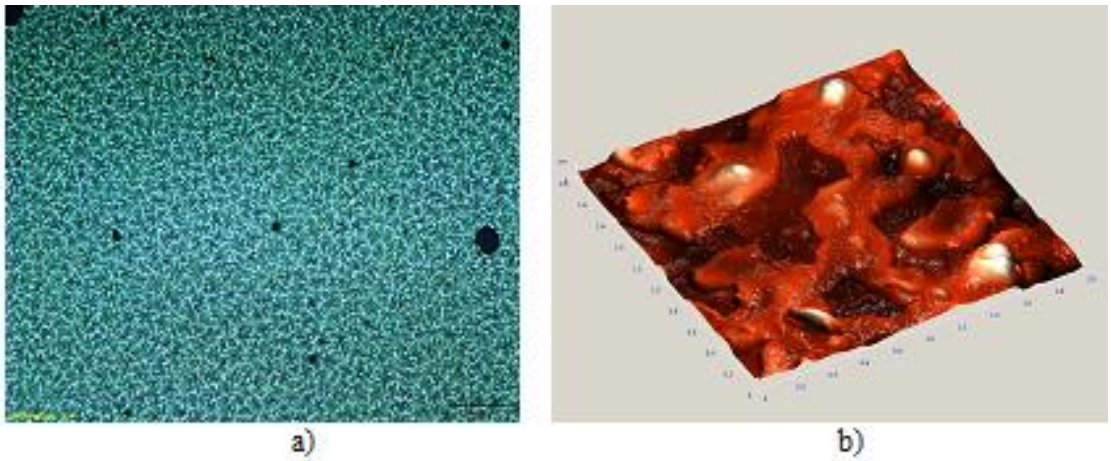


Fig. 7. Images of the 50 nm polysilicon film deposited at 580^{o}C, after phosphourus doping process (T= 1000^{o}C, 30 minutes): a) Optical image; b) AFM 3D image

The results regading the doping time and the temperature used for the 50 nm thin film help us for the next doping process settings. The temperature and the time of the doping process were decresed for the next thin films (Table 2). The run of thin films with 150 and 180 nm thickness show a good behaviour after doping treatment, observing no discontinuity.

Also the roughness is necessary to be also controlled. A higher roughness in not desired due to the influence in stiction of the 3D structural part and the electrods.

One major requirement for the MEMS/NEMS devices in bio-medical applications is the good biocompatibility of the materials used. The SU-8 polymer can be used as structural material for a variety of structures in medicine applications [13].

In order to demonstrate the applicability of the investigated materials two kind of manufactured MEMS structures are presented. A fabricated vibrational cantilever using the deposited polysilicon layers of 157 nm and 2 μm and a polymeric micro gripper [14] proposed to be used for manipulation and bio applications are shown in Figure 8.

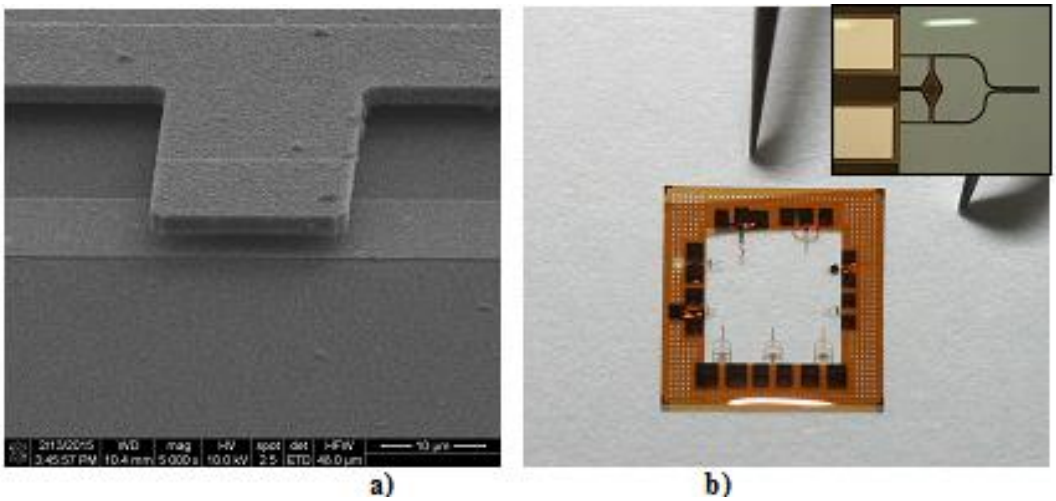


Fig. 8. a) SEM image of a fabricated and released polysilicon micro cantilever; b) Image of a chip with free standing SU-8 and Gold micro gripper structures

4. Conclusions

A good knowledge of the properties of the materials used for fabrication of MEMS/NEMS components is expected to bring improvements in the design and functionality of manufactured devices. The material properties are important in order to performe an accurate design and to obtain precise simulation results.

In this paper we investigated two materials obtained by different deposition techniques, we performed analysis and characterizations used for MEMS vibration sensors and for MEMS biostructures applications. The results regarding the analyses of surface topography and mechanical properties of structural materials used for MEMS fabrication are reported.

Five types of LPCVD undoped and doped polysilicon layers and a biocompatible polymeric material (SU-8) were investigated. In order to measure their properties a topography scan were carried out using an Atomic Force Microscope (AFM). Mechanical properties of the materials such as Young's modulus have been investigated also using the nanoindenter technique. The cristallinity of the materials and the variation of the grain sizes were measured using X-ray Diffraction System **(**XRD) and Scanning Electron Microscopes (SEM) characterization tools.

Young's modulus values for the polysilicon and the SU-8 materials were obtained. For the undoped polysilicon a Young's modulus of 130 GPa were obtained, and for the doped polysilicon a Young's modulus of 143.8 GPa were measured. For the SU-8 layer we obtained a Young's Modulus of 5.5 GPa. These values help us in the numerical simulations necessary to be performed for study of the structures behaviour before manufacturing.

Furthermore, the investigations helped us to choose the 2 μm polysilicon layers deposited at 580^{o}C to be used as structural material for vibrational beams manufacturing. The roughness of these layers is lower than for other deposition temperatures. For the electrode, the 150 nm film was the best choise due to the lower values of the roughness, obtained for 580^{o}C. A higher roughness in not desired due to its negative influence in stiction. The crystallinity of the material is

also important. If we raise the deposition temperature the cristallinity growth, but the roughness increase also.

The characterizations and tests performed contribute to a better design and experimental manufacture of the two proposed MEMS test structures.

Acknowledgments

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