# MEMS/NEMS TECHNOLOGIES AT CENTRO RICERCHE FIAT

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**Key words:** microtechnologies, nanotechnologies, MEMS, NEMS, etching

**Abstract:** The research on micro and nanotechnologies at Centro Ricerche Fiat ranges from NEMS/MEMS, to microoptics, substrates nanostructuring, smart materials, to many different technologies for sensors, actuators, displays and miniaturised energy production and storage devices.

Here we will describe two examples, SISA technology (Stress Induced Self Assembly) and its application to the fabrication of low cost MEMS IR spectrometers, and piezoelectric materials nanostructuring for adaptive photonic crystals and diffractive gratings.

# Tehnologije MEMS/NEMS v razvojnem oddelku koncerna FIAT

**Kjučne besede:** mikrotehnologije, nanotehnologije, MEMS, NEMS, jedkanje

**Izvleček:** V prispevku predstavljamo raziskave na mikro in nanotehnologijah v Raziskovalnem centru firme Fiat, ki obsegajo raziskave NEMS/MEMS, mikrooptike, nanostrukturiranja substratov, pametnih materialov in raziskave različnih tehnologij za izdelavo senzorjev, aktuatorjev, prikazovalnikov in shranjevalnikov energije in podatkov.

Nekoliko bolj podrobno predstavljamo tehnologijo SISA ( Stress Induced Self Assembly ) za izdelavo cenenih MEMS IR spektrometrov ter nanostrukturiranje piezoelektričnih materialov za izdelavo prilagodljivih optičnih kristalov in uklonskih mrežic.

## 1. Introduction

Micromachining technologies can be divided in two main classes, distinguishing **top down** and **bottom up** approaches.

The first set essentially consists of "planar" technologies, where the electronic components and MEMS are fabricated on (or in) substrates that are in the form of flat wafers. The microelectronics industry has made huge investments to develop wafer-level processes. To take advantage of these available and well experimented techniques, MEMS designers try to use the same technologies of the microelectronics industry, or variants based on the same steps. Common practice is to identify "bulk micromachining" with processes that etch deeply into the substrate, and "surface micromachining" with processes that remove sacrificial layers from beneath thin-film structures, leaving free standing mechanical structures. The structures are generally defined by film deposition, UV lithography and etching, repeated for each layer of the structure. The etching phase, both in bulk than in surface micromachining, can be wet or dry. Alternatives to photolithography, like focussed ion beam (FIB) or nanoindentation by modified atomic force microscopes, have been recently used to define detail at nanometre scale.

"Bottom up" approaches include all the so called "self assembly" techniques, and are typically referred as part of nanotechnology. Being life the most amazing example of self assembly, these techniques potentially represent the meeting point of "hard sciences", like physics and mathematics, and "life sciences", biology and medicine.

In the following figure (fig. 1) the above described classification is synthesized.

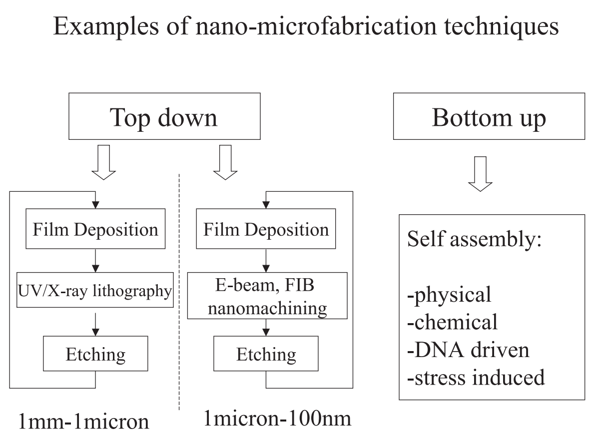



*Fig.1. Examples of nano-microfabrication techniques*

In this paper we will give few examples of bottom up techniques and their implementation at micro scale in MEMS-NEMS devices.

## 2. Stress induced 3d self assembly (SISA).

Stress Induced Self Assembly (SISA) technique is a combination of top down and bottom up approaches. In particular the **top down** approach is used to define geometries of objects at the micrometer scale by photolithography and details at the nanoscale level by focussed ion beam technology. The **bottom up** approach is used for the final "stress induced" assembly of the structures. In the following table the logic sequence of the steps defining the proposed technology are summarised (fig.2).

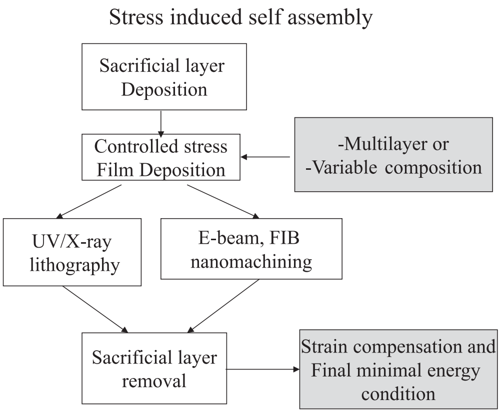



*Fig.2. Scheme of SISA technology.*

A sacrificial layer is deposited and patterned on a substrate by photolithography and etching. A second structural layer is then deposited, changing the composition of the alloy at different depths to control the stress of the film (fig.3).

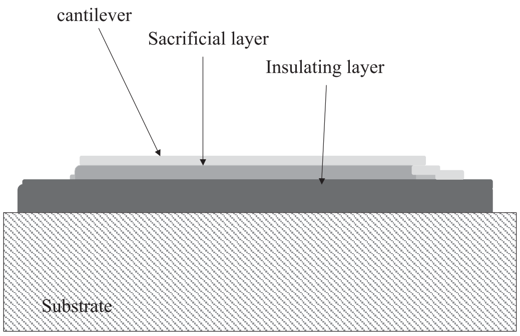



*Fig.3. Example of multilayer deposition technique for SISA process.*

With a precise control of the deposition conditions, strong internal anisotropic stress can be induced. In particular, if z is the direction perpendicular to the substrate we have:

$$\frac{\partial \sigma_{x,y}}{\partial z} \neq 0 \tag{1}$$

The structural layer is then patterned by usual photolithography and etching. A selective etching of the sacrificial layer allows the compensation of the stress by deformation of the structural layer. A proper control of both induced stress and patterning generates the desired 3D structures. A similar result can be obtained by bimorph or "multi-morph" structures.

Depending on the sign of the derivative of the stress along the z axis, different curvatures can be obtained (fig.4).

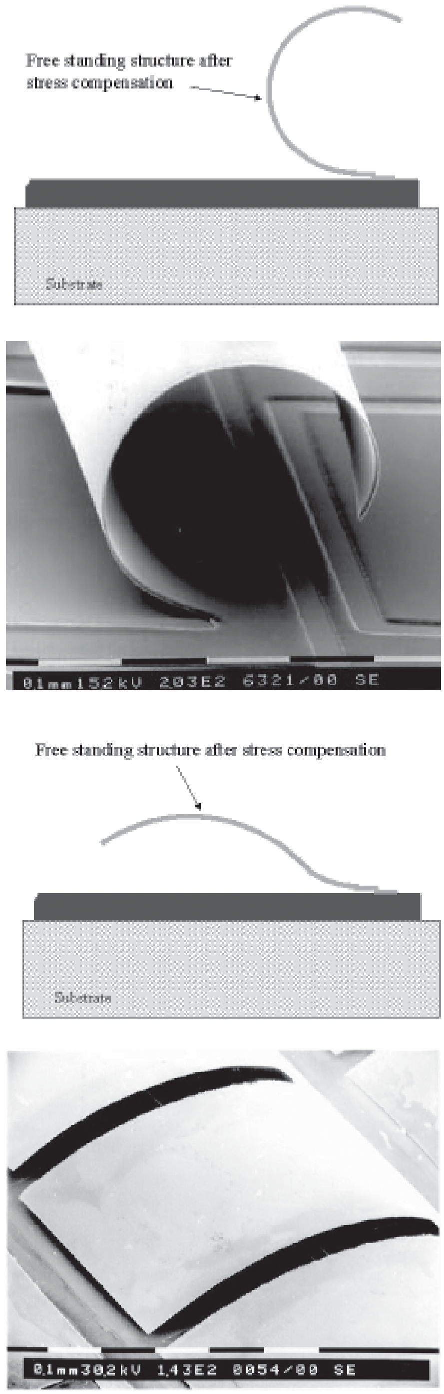



*Fig.4. Examles of free standing structures after sacrificial layer removal and stress induced compensation.*

Several components like microshutters /1/, microreservoirs /2/, microcoils have been fabricated by this self assembly technique (Fig.5 and 6).

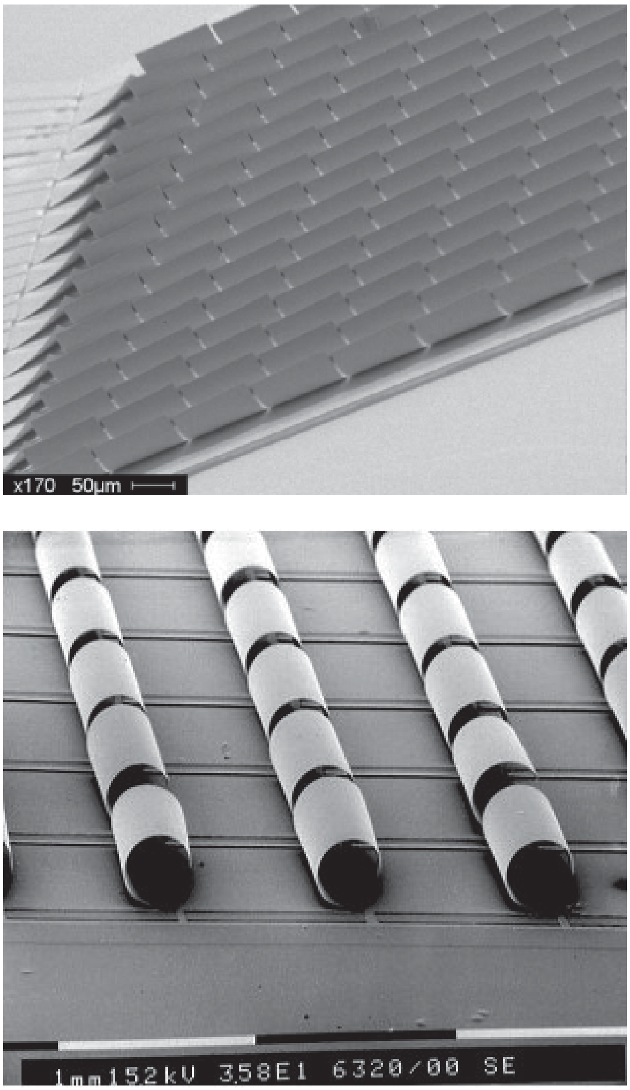



*Fig.5 Examples of stress induced microfabrication, microshutters.*

This technique is currently gaining more popularity and interesting works have been published implementing this method to fabricate complex structures. An original evolution of these concepts is the so called "origami technique", introduced by the research group in ATR in 2003 /3/. In this technique rigid parts are assembled using the flexible stressed structures as active hinges.

Photolithography and etching are generally used to obtain the patterns in the stressed films but during the study and development of a new device the implementation of FIB (focussed ion beam) technology in a sort of "rapid nanoprototyping" can be very useful. A structural layer with controlled internal stress is deposited on a sacrificial layer. FIB technology is then used to define the shape of the film (cutting with the ion beam with tens of nanometers level precision) and to deposit additional patterns to compensate the internal stress in defined regions. After release by etching of the sacrificial layer the desired self assembled structure is obtained. An example of this technique is given in the next figure (fig. 7), where a rectangle with two lateral circles have been defined. On the circles an additional "doughnut" platinum layer has been deposited. After the etching of the sacrificial layer the rectangular part will roll up in a cylindrical shape while the rigid circles will form the basis of the cylinder.

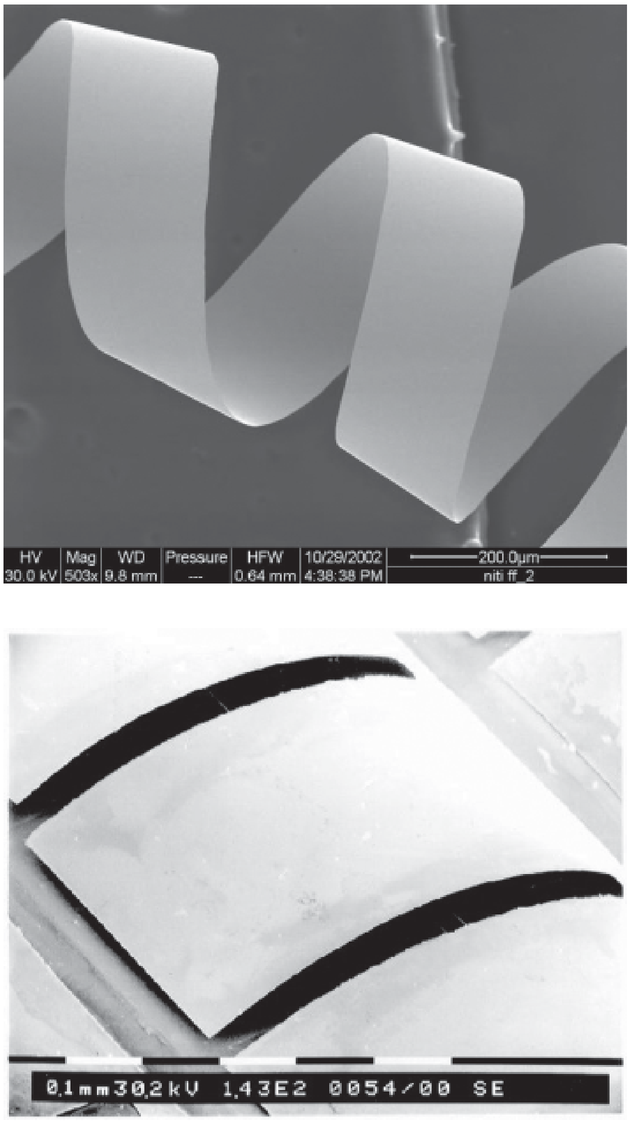



*Fig.6. Examples of stress induced microfabrication, microcoils and flexible elements for electrostatic actuators*

Parts of the stressed layer can be covered by a selective Pt layer to obtain, after sacrificial layer removal, a flat part of the free standing film, as reported in fig. 8.

The technology is expected to reduce the development time of nano and microstructures allowing the preparation of few test samples without the need of mask design and fabrication, multiple steps of photoresist deposition, baking, exposure, development and dry or wet etching of the

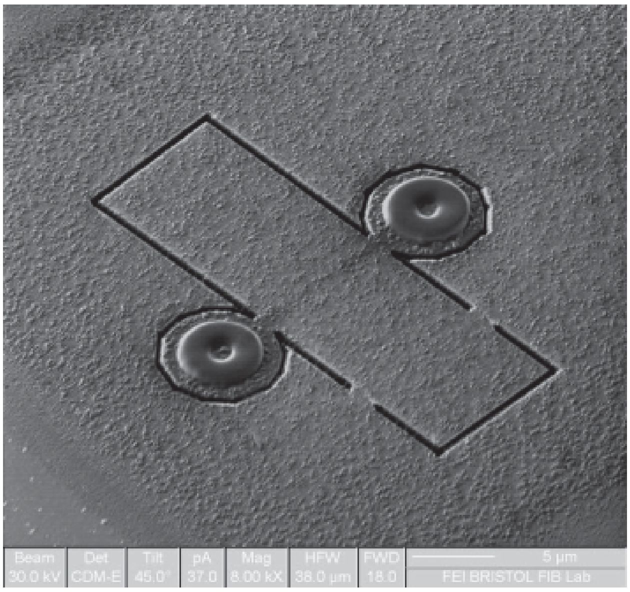


*Fig.7. Example of FIB micromachining*

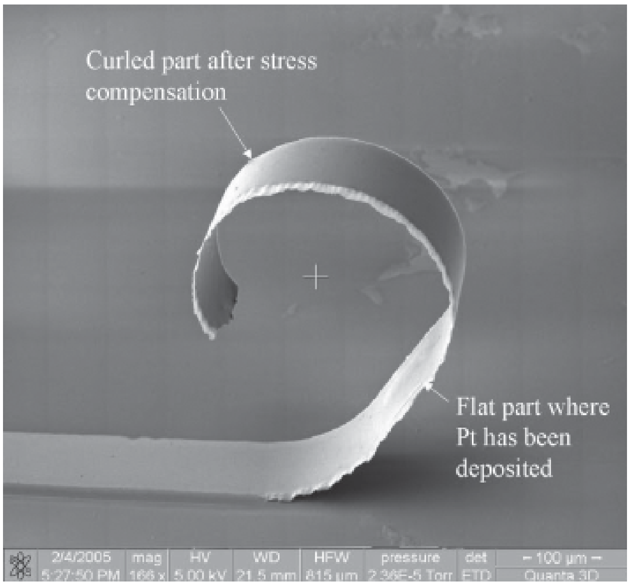



*Fig. 8. Stress control by FIB additional layers deposition.*

microstructures, deposition of additional layers, and again lithography steps. For successful configurations only the masks for batch processing are generated.

The main application explored by the authors implementing stress induced self assembling techniques is related to electrostatically driven microstructures, used both for optical modulation than for miniaturised actuators. Many technological issues have been addressed to combine suitable materials and processes for electrostatic actuation, self assembly, etching compatibility, optical and mechanical features. In the following an example of application of SISA technology to IR and optical modulators will be given.

## 2.1 Exhaust gases emission control

Microsystem technologies can have a strong impact on the evolution of the engine control strategies. The availability of low cost, miniaturised sensor arrays for the detection of the main physical and chemical parameters will enable a higher degree of control of the combustion conditions and of the engine operation in general.

Due to the concern of health, environment and climate the limits for emissions of nitrogen oxides (NOx), hydrocarbon (HC), carbon monoxide (CO), particulate matter (PM), and the greenhouse gas carbon dioxide (CO2) from vehicles equipped with combustion engines are continuously lowered. In the year 2008 the European limits for NOx and PM for diesel engines will be 0.08 g/km and 0.005 g/km, respectively (Euro V). The US limits will be similar (TIER 2-BIN 5).

The continuous evolution towards low ecologic impact cars can be enabled by a more precise and continuous control of the emissions.

Infra red spectroscopy is normally used to characterise the engine behaviour but the available gas spectrometers are currently not suitable for on board application, being bulky and expensive. A novel concept of spectrometer based on SISA fabrication is here presented.

## 2.2 Microshutter based spectrometer.

The structure of the MEMS device implemented in the spectrometer is shown in fig.9. It is based on an optically transparent substrate, e.g., glass or sapphire. The substrate is coated with two optically transparent layers, first an electrically conductive layer and then an insulating layer.

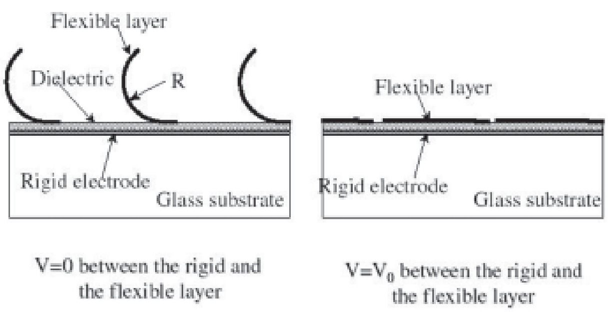



*Fig. 9. Structure and working principle of electrostatic microshutters.*

Pixels have a “digital” response that is freely programmable by the user, e.g., in a Hadamard sequence. A choice of substrate materials is available, including glass and sapphire, which in turn allows utilisation over a wide wavelength range including the visible, near infrared, and mid infrared ranges. The device can be used in both transmittive and reflective optical architectures. Hermetic packaging is not required, instead, dust-proofing is sufficient. A light beam is directed on the gas sample under study; the transmitted light beam is divided in its components: a narrow part of the dispersed beam is selected by mean of a linear array of micromechanical shutters. The intensity of the transmitted beam is than detected by a single sensor (fig.10).

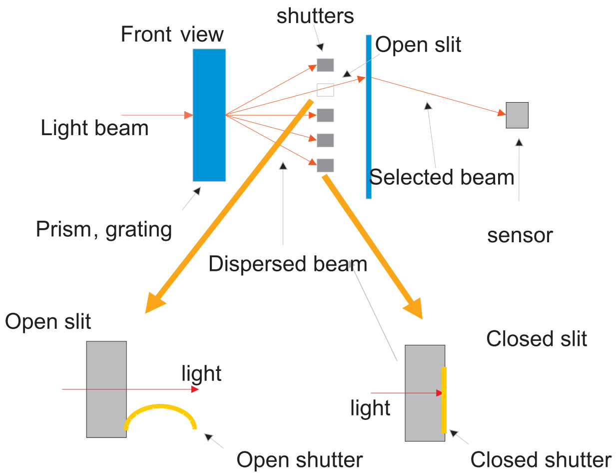



*Fig.10. Concept of the Microshutter based single sensor spectrometer.*

A single detector instead of a detectors array will improve the performances and will simplify the production process. Fig. 11 presents the packaged 25-element MOEMS shutter array and the key parts inside the spectrometer prototype.

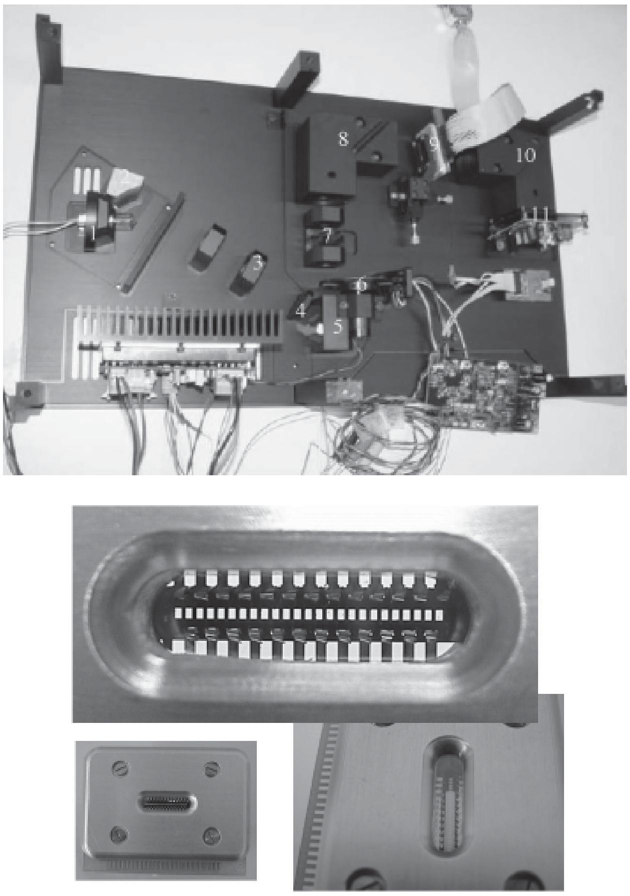


*Fig.11. The spectrometer prototype system.*

It is composed by a light source (1), a mirror (2), a band pass filter (3), an iris (4), a second mirror (5), a chopper (6), generating both the needed pulsed light beam and a clock at 525 Hz for system synchronisation, the housing for the material to be analysed (7), in particular a cuvette, the first grating (8), the microshutter device (9), the second grating (10) and the single element PbS sensor (11).

The main benefit expected from this technique is the low price level of the shutter array together with the single element detector, which will enable cost-effective spectrometers to be made. The implementation of fibre and/or integrated optics architectures will enable the miniaturisation of the system. To test the performances of the spectrometer we measured spectra of different liquids. We report here the case of urea that is of interest for some biomedical applications.

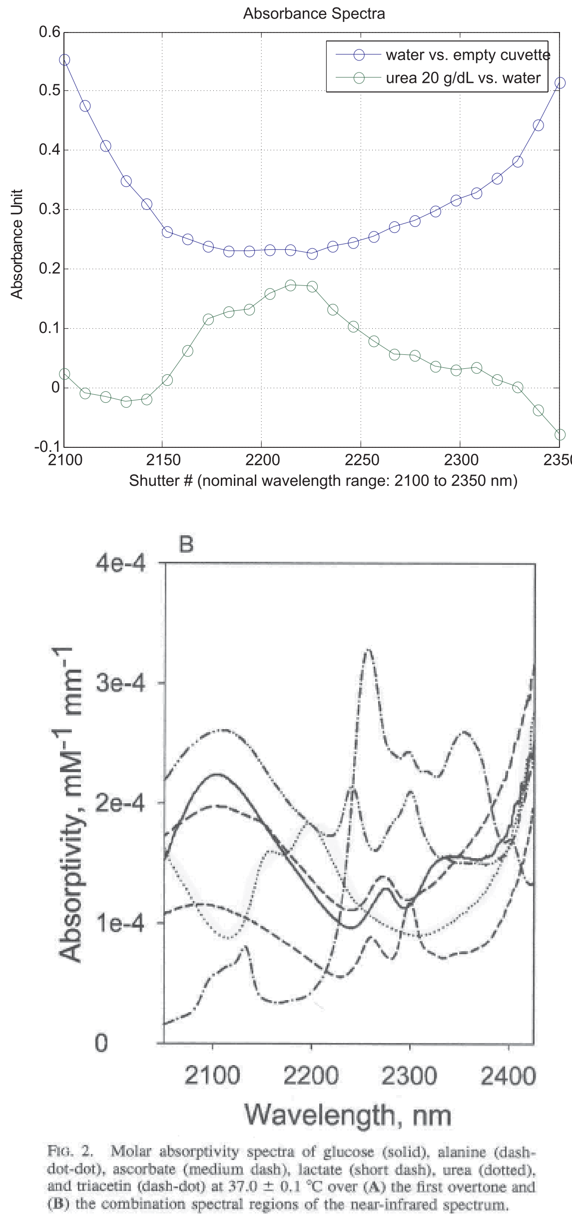



*Fig.12A,B Comparison of spectrum from microshutter based spectrometer and /4/*

The absorbance spectrum of 0.5 mm water measured with the prototype has the expected shape. Very convincing is also the analysis of the high-concentration urea absorbance spectrum measured using the prototype (green trace in Fig.12 A), which can be compared to the spectrum measured on highly accurate FT-IR type spectrometer shown in Fig.12 B.

The double absorbance peak feature near 2200 nm shown in Fig.12B can clearly be seen also in Fig.12A. The water displacement effect, which was computed out in fig.12B but not in fig.12A, is responsible for the differences in the absorbance slopes near the edges of the plotted wavelength range, but this is expected and inconsequential here.

We calculated the noise performance is close to the limit set by the PbS photodetector. The prototype, given only 1 second of integration time, can resolve urea concentration changes as small as about 4.7 $(mg/dL)_{RMS}$.

## 3. Piezoelectric nanostructures.

As an example of smart materials nanostructuring and integration in MEMS devices we will shortly describe the concept and proposed fabrication of an adaptive piezoelectric photonic crystal.

Photonic crystal is a periodic dielectric material. The dielectric permeability of photonic crystal is given by

$$\varepsilon\left(\mathbf{x}+n\mathbf{a}_1+m\mathbf{a}_2+l\mathbf{a}_3\right)=\varepsilon\left(\mathbf{x}\right) \qquad (2)$$

where $\mathbf{a}_1$, $\mathbf{a}_2$, $\mathbf{a}_3$ are the lattice vectors defining the directions of periodicity. Photonic crystals can totally reflect the radiation of certain wavelengths. The so called "band gap" depends on the structure and geometry of a photonic crystal. The computing of the band gap is reduced to the solution of Maxwell's equations describing the propagation of electromagnetic waves in a periodic dielectric material. A modulation of the geometry and/or dielectric permeability would enable the fabrication of novel optical modulators and adaptive waveguides. Nanostructured piezoelectric materials can be used for that purpose. We started from the fabrication of ordered templates by self assembly techniques, in particular anodic porous alumina (APA) and artificial opals. The templates are then impregnated by solutions of PZT and reticulated by thermal treatments.

### 3.1. APA

APA is an example of 2D photonic crystal. To form the structure a high purity aluminium foil is put in contact with the anode of a electrochemical cell; the anodization process is carried out with acid electrolyte. An oxide layer begins to grow on the surface of aluminium, after some minutes pores form on the surface and the mechanical stress of the oxide force the hexagonal distribution of the pores: at this point the walls of the pore grow with constant velocity and the bottom layer thickness of the pore remains constant. Therefore it is possible to obtain thick membranes of alumina with straight channels from one side to the other with one open end; the barrier layer can be removed with a chemical etching to obtain micro channels both sides opened. Many tests have been made to determinate the main parameters of the process that affects sample geometry: time of anodization, current density, type and concentration of electrolyte, temperature of the environment. The inter-pore distance depends nearly on the anodic potential, so structures with different dimension but same geometry can be obtained as shown in figure 13.

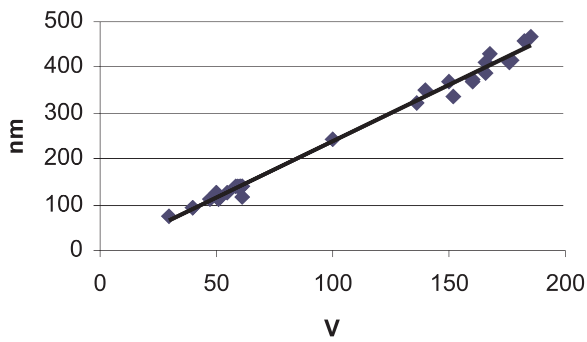



*Fig.13. Interpore distance depending on electrolytes and voltage*

Pores diameter can be increased with widening process in acid solution that penetrates in the pores and consumes pores walls. Surface and section views are reported in fig.14.

The growth rate of alumina depends on current density as well and varies from 5 to 25 μm/h, that permits to obtain alumina membranes with the desired thickness.

Porous anodic alumina is a nanostructured material that can be produced with a fast and low-cost process although the lattice can present many defects and irregularity.

### 3.2 Artificial Opals

Opal-like structures are 3D photonic crystals and are produced by CRF by two different methodologies: the first utilising Layer by Layer (LBL) process, the second by sedimentation in a centrifuge.

The LBL assembly is based on the alternating adsorption of oppositely charged species, such as positively and negatively charged polyelectrolyte pairs or polyelectrolytes and nanoparticles. Multilayer ultrathin films can be developed with "molecular architecture" design with precise control of thickness and molecular composition. It can be effectively applied to the coating of both macroscopically flat and non-planar (e.g. colloidal particles) surfaces. Opals nanospheres syntesized in water/Ethanol solvent are negatively charged: if coupled with appropriate positive polyelectrolyte the spheres can be deposited in a nanostructured film.

The following standard cyclic procedure was employed,

(i) dipping of the substrate into a solution of positive polyelectrolyte

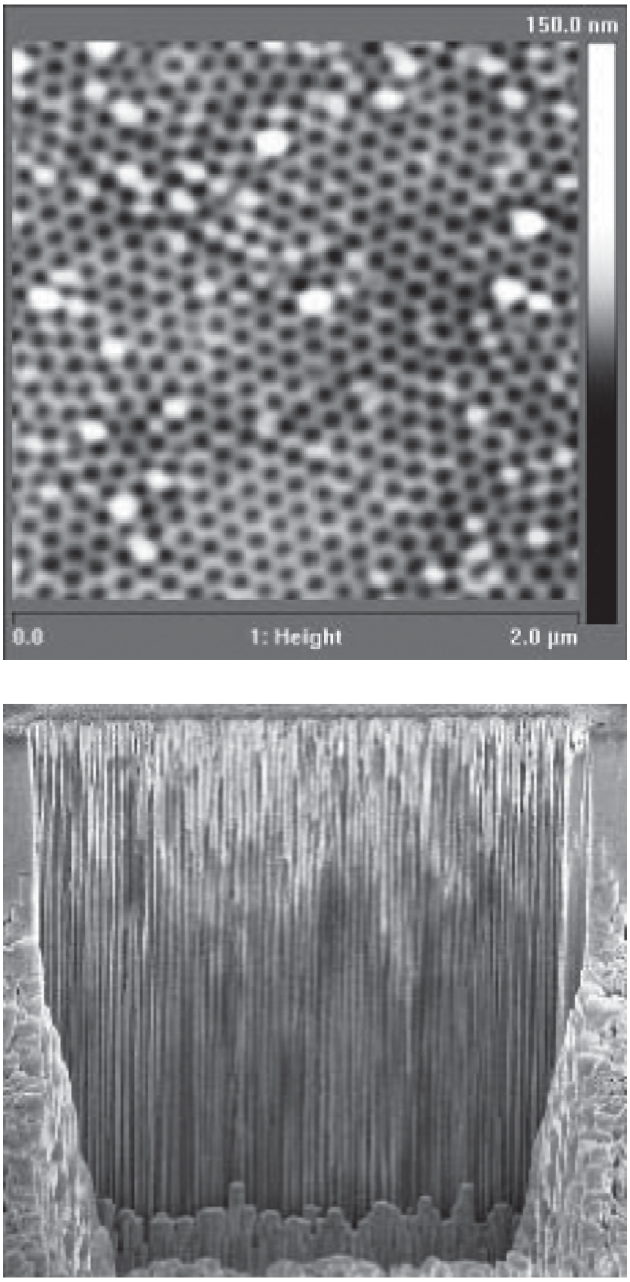



*Fig. 14. AFM image of alumina surface and FIB image of the section of alumina surface*

(ii) rinsing with water

(iii) dipping into the aqueous dispersion of opals nanospheres and rinsing with water again

The process can be cycled to obtain a multilayer film of the needed thickness. The slides with deposited opals are impregnated with PZT solution (provided by Josef Stefan Institute) by drop, dip, and spin coating. The sol infiltrates the interstitials, leaving a flat layer above the spheres (see fig. 15). After the impregnation a thermal treatment in oven is performed.

Alternatively the opals can be fabricated by sedimentation in a centrifuge. In order to have a very ordered opals film, substrates 0.5 x 0.5 cm are inserted in a flat centrifuge tube. A very short volume of opals solution is placed into the tube and centrifuged. The liquid surnatant is separated and the glass with the film is thermal treated at 400°C. The slides are characterised by AFM and FIB techniques.

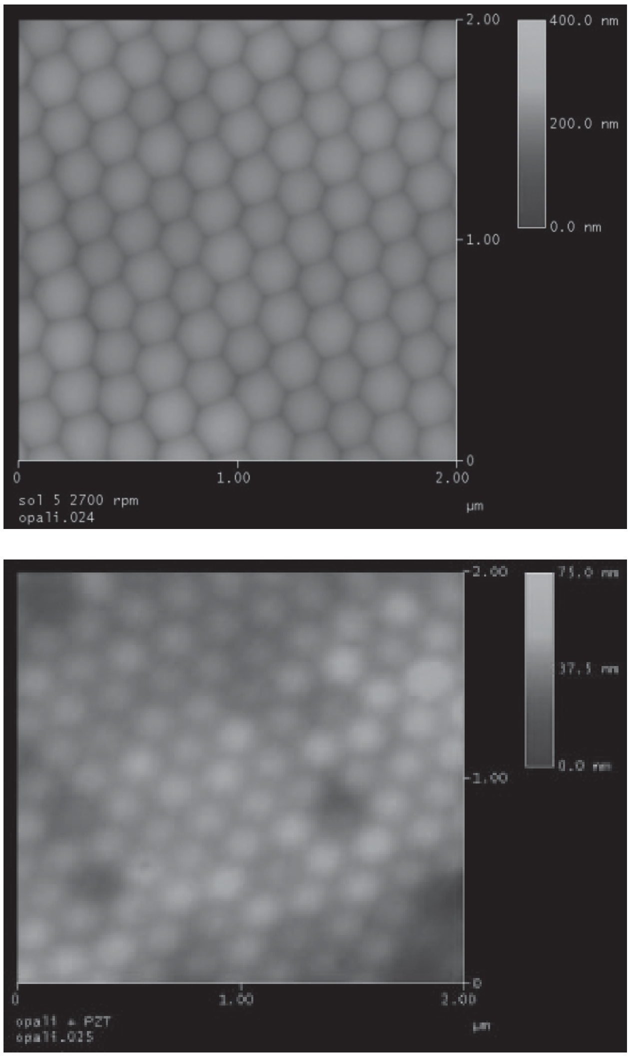



*Fig.15. Opals before and after impregnation.*

Experiments on the electrooptical characterisation are currently in progress.

## 3.3 Adaptive diffractive gratings

A different approach based on adaptive diffractive gratings has been investigated too. A binary grating has been fabricated on a flexible, compliant material and integrated with a piezo tube (supplied by Ferroperm). The coupling of the grating with the piezo actuator allows the modulation of the height of the grating profile. Being the efficiency diffraction dependent on that height, the idea is to actuate the compliant grating to change the diffracted light in different orders.

The experimental setup to verify the feasibility of the device is composed by a laser source, the adaptive diffractive grating and an optical sensor measuring the intensity of a part of the diffracted light (fig.16)

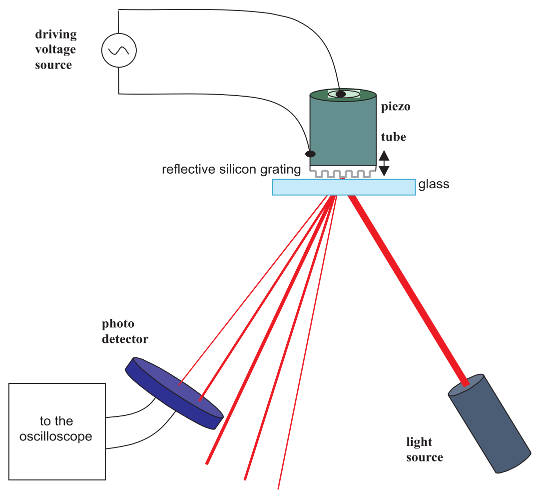



*Fig.16A*

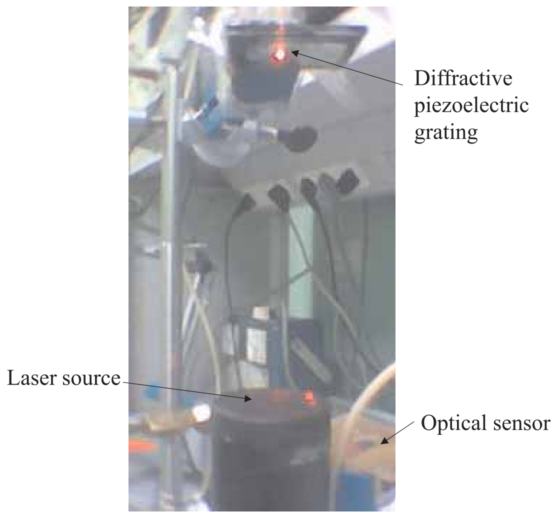



*Fig. 16B*

When the element is not actuated there is no AC signal on the sensor (fig.17A) while when the element is actuated the modulation is displayed.(fig.17B)

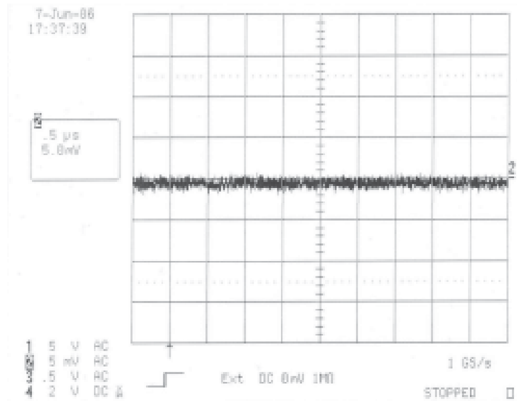
As expected the light modulation follows the frequency of the piezoelectric actuator.

## 4. Conclusion

Several automotive applications of MEMS have been proposed in the last years. Sensing seems to be the most promising field for the implementation of MEMS-NEMS technologies in new components with novel or improved functions. In particular a new application of Microshutter Technology to exhaust gases control has been presented.

A novel MEMS optical modulator based on adaptive nanostructured metamaterials has been addressed. It is an example on how the implementation of nanoscale techniques in MEMS devices can be a promising way to effectively exploit the great potentialities offered by nanotechnology.

A



B

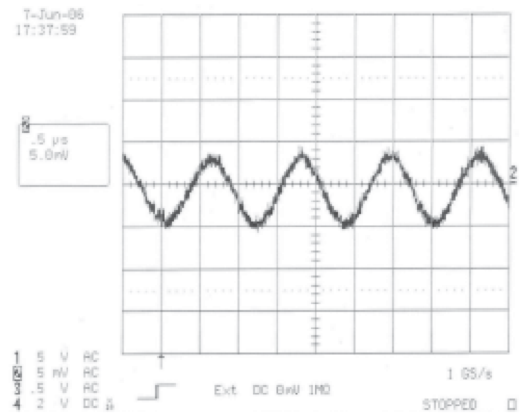


*Fig.17A Piezoelectric not actuated – no signal on the photodetector and B Piezoelectric actuated @ 860 kHz (60 Vpp) – modulated light on photodetector*

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