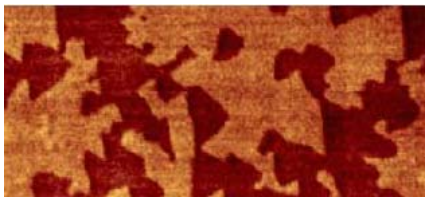

Nanostructured Interfaces and Surfaces
Centre of Excellence
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Monday 13 and Tuesday 14 December 2004

Dipartimento di Fisica Sperimentale, Via Pietro Giuria 1, Torino

Scanning Probe Microscopy and Nanotechnology

Monday 13 – Aula Magna

- 14.30 - 14.45 **Claudio Manfredotti** – Introduction
  NIS Torino (I)
- 14.50 - 15.30 **Alexander Shlüger** – Measuring the force of individual surface ions
  University College London (UK)
- 15.30 - 16.10 **Florence Marchi** – Atomic Force Microscopy and related techniques: new ways and tools to explore the nanoworld
  University of Grenoble, UJF-LEPES/CNRS (F)

BREAK

- 16.40 - 17.20 **Paolo Samorì** – Scanning probe microscopies beyond imaging
  ISOF – CNR Bologna (I)
- 17.20 - 18.00 **Serena Bertarione** – Imaging the surface morphology on nanostructured materials by atomic force microscopy
  NIS Torino (I)

Tuesday 14 – Aula A

- 9.00 - 9.40 **Ugo Valbusa** – The nanostructure zoo
  Università di Genova (I)
- 9.40 - 10.20 **Giorgio Mori** – Chemical and transport properties of nanostructures fabricated by local anodic oxidation
  INFM - TASC Trieste (I)
- 10.20 - 11.00 **Chiara Manfredotti** – Nanofunctionalization of diamond surfaces by local anodic oxidation
  NIS Torino (I)

BREAK

- 11.30 - 12.10 **Pasqualantonio Pingue** – The *Nanoworker*: a user-friendly approach to SPM-based nanolithography and nanomanipulation
  NEST - INFM Pisa (I)
- 12.10 - 12.50 **Thomas Muehl** – STM-based nanolithography of diamond-like carbon films
  IFW Dresden (D)

Il coordinatore del Centro di Eccellenza NIS
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Measuring the force of individual surface ions

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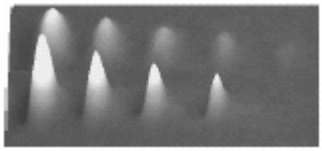
Recent experiments using dynamic force microscopy (DFM) demonstrate that the short-range interaction forces can be measured selectively above chemically identified sites on surfaces of insulators. These experiments and atomically resolved DFM imaging of insulators rely on advanced theoretical models for their interpretation. We will discuss and compare the results of calculations of the tip-surface interaction and modelling of DFM imaging of insulating surfaces using different tips. The theoretical models available now are sufficiently refined to provide information not only about the surface, but also the probe tip, and the physical changes occurring during the scanning process. We will compare the mechanisms of image contrast formation on ionic and covalent surfaces and the transferability of image interpretation between surfaces of similar structure. The applications of DFM to study the structure and spectroscopic properties of surface point defects, spin ordering and molecular manipulation will be considered.

Atomic Force Microscopy and related techniques: new ways and tools to explore the nanoworld



Florence Marchi

University of Grenoble, UJF-LEPES/CNRS, France

In the last few years, a lot of related techniques of AFM (Atomic Force Microscopy) have been developed in order to associate local topography information with physical and/or chemical properties.

With the huge development of Nanotechnology field and more precisely nano-electronics and nanomechanics, two main AFM related techniques and improvements, present a great interest.

Firstly Electrical modes as EFM (Electric Force Microscopy), SSRM (Scanning Spreading Resistance Microscopy), SCM (Scanning Capacitance Microscopy), Kelvin mode, secondly development of specific user interface using haptic feedback as nanomanipulator or modified AFM tips.

After an introduction to these electrical modes, the first part of this seminar will focus on the characterization of electrical properties of semiconductor nanostructures, surfaces and manipulation-detection of few electric charges even single charge [1,2].

The second part will focus on two points linked to nanomechanics:

- characterization of very low forces as Casimir and Van der Waals forces which play a crucial work in the MEMS/NEMS* behaviour thanks to modified AFM tips,
- real-time interaction with a non flat sample, a drosophilae leg which can be considered as biological MEMS, thanks to a nanomanipulator system.

* MEMS/NEMS : Micro/Nano Electro-Mechanical System

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[2]"Single electron tunneling to insulating surfaces detected by electrostatic force", L. Klein and C. Williams, APL, **81**, 4589 (2002).

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Scanning probe microscopies beyond imaging

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Unraveling physico-chemical properties of molecule based architectures across a wide range of length scales represents one of the major goals of materials science. Scanning Probe Microscopies (SPMs) permit not only the imaging of surfaces, but most interestingly they also make it possible to gain insight into a variety of physical and chemical properties of molecule-based structures occurring in scales ranging from the hundreds of micrometers down to the sub-nanometer regime. Moreover they allow the manipulation of objects with a nanoscale precision, thereby making it possible to nanopattern a surface or to cast light onto the nanomechanics of complex assemblies. Thus, they can provide crucial information for the optimisation of functional materials.

My lecture will review recent progress in the use of SPMs beyond imaging on soft materials,[1] with a particular emphasis on the study of the mechanical properties of isolated polymer chains [2] as well as on the monitoring of interfacial dynamics processes such as phase segregation phenomena in a polydisperse molecular systems physisorbed at the solid-liquid [3] or solid-gas interface,[4] on the perturbation of the electronic states of molecules adsorbed at surfaces [5,6] and on the reaction chemical occurrence [7]. Moreover, the use of the SPM tip to trigger the mechano-chemical switch between two different nanostructures in a supramolecular ensemble will be discussed.[8]

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Imaging the surface morphology on nanostructured materials by atomic force microscopy

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In recent years an increasing interest has emerged in the investigation of the surface morphology of a variety of polycrystalline materials, in order to correlate processes and phenomena, occurring at atomic level, with the real structure of the different surfaces. Once the morphology and the structure of the exposed faces is known, a better understanding of the surface properties of the investigated systems can be obtained.
It is known that a precise knowledge of the surface atomic structure is a prerequisite for understanding and controlling the physical processes involved in many industrial applications. As a matter of fact processes like as the growth of epitaxial films on the surfaces of single crystals, the catalytic reactions on dispersed polycrystalline materials or the possibility to modify the surfaces by means of atomic/molecular manipulations are intimately connected with the structure of the underlying surfaces.
Scanning probe microscopies (SPM) have shown a relevant role in characterizing the surface morphology of a wide class of single crystals, in order to identify any type of defect interrupting the regularity of the extended faces such as steps, kinks or to define the nature and the distribution of atomic-scale defects, such point vacancies or adatoms [1,2,3].
On the contrary rare literature data concerning the application of SPM techniques in the analysis of the surfaces of polycrystalline oxides are reported. Moreover in the last years AFM has become a powerful technique for the characterization of nanoparticles and has acquired an important role in allowing to obtain the real particle morphology, including the vertical dimension. In case of analysis of phase mixtures (polycrystalline oxides supported metal particles, mixed oxides etc.), the 3D images allow to distinguish structurally non equivalent particles, the presence of clusters/aggregates on extended and regular faces, on the basis of their size (ratio of lateral and vertical dimension). Although AFM use for particles smaller than 10 nm is limited by the strong influence of the tip convolution, in the 10-100 nm, it gives information camparable with TEM technique.
As far as the aim of this contribution is concerned, some AFM images of polycrystalline oxides (ZnO, NiO, MgO, LaCrO3, etc.) will be discussed and compared with previously obtained SEM and HRTEM data [4,5]. In addition, high resolution images of 'smoke' MgO obtained by atomic force microscopy will be presented. These results will be correlate with the vibrational properties of the investigated materials [6].

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The nanostructure zoo

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By using ion erosion and molecular beam hepitaxy a large variety of nanostructures have been obtained. Quantum dots (1), magnetic wires (2), ripples (3), checkerboards (4), simple (5) and rhomboidal pyramids (6) have been observed in several materials ranging from semiconductors to metals, from glasses to ionic crystals.

The talk will review the elementary mechanisms which are at the base of these phenomena and will illustrate the potential applicability of these techniques in nanotechnology.

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Chemical and transport properties of nanostructures fabricated by local anodic oxidation

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Local anodic oxidation (LAO) is an effective tool for patterning the surface of a conductive sample. In last years, LAO lithography has been employed with success for the definition of mesoscopic devices on GaAs/AlGaAs heterostructures as quantum point contacts (QPCs),[1] quantum dots[2], and Aharonov-Bohm rings.[3] On the other hand only few works have been dedicated to the study of the chemical properties of the LAO-oxides.[4-7] This type of knowledge represents the first step in order to control the chemistry of the LAO-oxides.

In this presentation the attention will be focused on the transport and chemical properties of nanostructures fabricated with LAO. In particular, a QPC has been fabricated with LAO on a high mobility GaAs/AlGaAs two dimentional electron gas.[1] At low temperature the QPC shows well defined plateaus due to the conductance quantization. We investigated also the evolution of the conductance plateaus as a function of perpendicular magnetic field and found that the observed behavior well agrees with the one predicted by the model of Berggren et al.[8]

The chemical properties of the LAO nanostructures have been investigated with microscopic X-ray photoemission spectroscopy.[7] We find that the LAO-structures desorb under irradiation wih soft X-rays (130 eV). We analyzed the desorption process by time-resolved photoelectron spectroscopy. We observe that even in the first stages of light exposure LAO-oxide is mainly composed of Ga_2O, with a small fraction of Ga_2O_3 and As-oxides. The As-oxides are located only in the surface layers of the LAO-oxide where they account for 10 % of the oxide. Moreover, we find evidence for the presence of unoxidized GaAs in the LAO-oxide.

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Nanofunctionalization of diamond surfaces by local anodic oxidation

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Diamond shows surface properties no other material shows; these properties differ largely as the top surface layer is H- or O-terminated (1). A H-terminated surface has a high p-type conductivity in undoped diamond also (holes concentration is about 10^{13} cm^{-2} for some nanometres depth), is highly hydrophobic and is characterized by negative electron affinity (NEA), while an O- terminated surface is insulating (resistivity is about 10^{11} ohm cm), hydrophilic, and shows a normal electron affinity (PEA). It has been demonstrated that a FET structure can be realized by using the surface conductive layer; moreover, these diamond properties can be utilized for biosensing applications (2,3).

The oxidation of a H-terminated surface at a nanometre scale can be obtained by local anodic oxidation (LAO), using an atomic force microscope (AFM) in contact mode and a conductive tip by applying some volts biases to the sample surface. In such a way, MOS lateral structures (4) have been realized, having an 'oxidized' line 60 nm wide through which the tunneling Fowler-Nordheim mechanism has been observed.

The oxidation of the diamond hydrogenated surface as a function of different parameters like scan speed, ambient humidity and applied bias (5,6) has been investigated, using a homoepitaxial diamond sample, composed by an Ib type HPHT diamond, (100) oriented, onto which an IIa type diamond film (5 μm wide) has been epitaxially grown.

Results indicate a good reproducibility of the oxidation lines, the width of which at scan speeds lying between 20 e 110 nm/s, varies from 80 to 100 nm and from 45 to 90 nm at 60% and 40% ambient humidity respectively. The oxidation process is accompanied by a current decay between tip and sample that ends in about 10 s.

Other investigations are necessary in order to define clear conclusions on the nature of the chemical reaction responsible of the phenomenon, that cannot be simply interpreted as an analogue of what happens e.g. on silicon. Moreover, some aspects of surface topographic variations accompanying the process must be cleared, as about that nonconforming data are present in literature.

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The *Nanoworker*: a user-friendly approach to SPM-based nanolithography and nanomanipulation

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A new scanning probe microscope (SPM) system has been developed and its utilization as a tool for lithography, manipulation and mapping on nanometer scale is reported. User-friendly graphic interface allows to perform nanolithography and to monitor the whole process in real time. We implemented two types of lithographic processes.

The first one is VECTORIAL MODE: mouse leads the tip upon the surface, in the three spatial dimensions. In vectorial mode we import the acquired image in the "Vectorial window", and then we move the mouse upon the image, acting the probe in real time as a pantograph from macroscopic down to nanometer scale, taking advantage from an absolute-positioning stage. We can also select any parameter (i.e. applied force, or tip-surface bias) and applying it locally by a mouse click or tuning it by the mouse scroller. Moreover, the so called "*contour-mode*" allows turning off the feedback, moving the tip in any direction at a fixed relative tip-sample distance, taking in account sample topography. Finally, vectorial mode can also be performed in automatic mode, repeating several times a previously-saved lithographic process.

The second one is SMART-BITMAP MODE: this configuration allows superimposing a bitmap pattern, with 24bit-resolution colour table, on a selected region of the previously-imaged sample region. Three different lithographic tasks are assigned to the 3 (RGB) colour scales, with 256 values each. Lithography is performed in FORWARD scan and imaged in BACKWARD scan. For example, we can plan to perform on forward scan force-distance maps with different dwell times related to the red scale of the bitmap image, applying a force modulated by the green scale and a voltage modulated by the blue tones.

The instrument has been successfully employed in order to perform dynamic ploughing[1], spatially-resolved anodization lithography (LAO)[2] on semiconductor and metallic substrates, dip-pen nanolithography (DPN)[3] and carbon nanotubes manipulation. Any measured properties of the sample can be used by a new and user-friendly lithographic interface to realize interactive patterning of the sample. Other interesting applications of this instrument in nanolithography will be also presented.

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STM-based nanolithography of diamond-like carbon films

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The spatially localized emission current of a scanning tunnelling microscope tip leads to the local oxidation or the local graphitization of diamond-like carbon thin films depending on whether the environment is air or ultrahigh vacuum, respectively. Using this technique, nanostructures smaller than 10 nm can be written.

Due to the different properties of diamond-like and graphite-like carbon, there are a lot of possible applications for graphite nanostructures on diamond films. Systematic investigations of the current, voltage and charge dependence combined with analytical methods like scanning tunnelling spectroscopy and conductive scanning force microscopy provide some preliminary conclusions on the underlying modification mechanism on an atomic scale.

Fig.: STM image of graphite-like nanodots within a diamond-like carbon layer (image size 1.2 μm x 1.2 μm).

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