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APPLICATIONS OF FOCUSED ION BEAM IN MATERIAL SCIENCE

UPORABA FOKUSIRANEGA IONSKEGA CURKA V ZNANOSTI MATERIALOV

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The focused ion beam (FIB) microscope is a tool that has a widespread use in the field of material science because it is able to micromachining with high resolution imaging thus therefore enhancing a broad range of both fundamental and technological applications in material science. The FIB is based on a beam of Ga ions which sputter the sample enabling precise machining at the nanometer/micrometer scale. The FIB instruments received particular attentions in the 1980s when the semiconductor industry used it as offline equipment for mask or circuit repair, but only in the 1990s the FIB was used in research laboratory. Nowadays there are commercial instruments (Dual Beam FIB / SEM) that integrate the precision cross section power of a FIB with the high resolution imaging of an SEM creating a powerful cross section and imaging tool. The combined SEM capability allows for real time monitoring of the FIB cuts with a higher resolution.

Key words: focused ion beam, ion source, dual beam instruments, ion-solid interactions, applications

Mikroskop s fokusiranim snopom ionov (FIB) je orodje s široko uporabnostjo v znanosti materialov, ker omogoča mikroobdelavo in opazovanje z veliko ločljivostjo in odpira široko področje temeljnih raziskav materialov in tehnoloških aplikacij. Podlaga FIB je curek Ga-ionov, s katerim se obstreljuje tarča, kar omogoča mikroobdelavo pri redu velikosti nanometer-mikrometer. Naprava FIB je vzbudila posebno pozornost v 80-ih letih, ko so jo uporabljali v industriji polprevodnikov za popravilo mask in tokokrogov, v 90-ih letih pa se je razširila tudi v raziskovalne laboratorije. Na voljo so komercialne naprave (Dual beam FIB), ki združujejo natančnost FIB z visoko ločljivostjo SEM, kar ustvari učinkovito orodje za obdelavo in opazovanje. Kombinirana naprava omogoča v realnem času opazovano FIB-obdelavo z veliko ločljivostjo.

Ključne besede: izvor ionov, naprava z dvojnim curkom, interakcija ion-trdna snov, uporaba

1 INTRODUCTION

In his famous talk on nanotechnology "There's Plenty of Room at the Bottom" held at the 1959 meeting of the American Physical Society at Caltech [1], Richard Feynman considered *"the problem of manipulating and controlling things on a small scale"*. As an example of this kind of manipulations, he considered the task of writing *"the entire 24 volumes of the Encyclopaedia Britannica on the head of a pin"* and for the purpose he imagined a machine which could afford this task: *"we can reverse the lenses of the electron microscope in order to demagnify as well as magnify. A source of ions, sent through the microscope lenses in reverse, could be focused to a very small spot"*.

Even if Feynman describes an interaction of the ions with the sample that is different from the one we know to happen (he postulates a deposition while we know that the result is a removal of atoms), all the same his words sounds like a very rough but essential picture of the focused ion beam (FIB).

In this paper we describe how this very intuitive idea has become a real instrument (in **Figure 1** we reproduce the result of Feynman's "experiment" with a modern FIB). In particular we will consider the state of the art of dual-beam instruments, where the FIB is coupled to a scanning electron microscope (SEM) to realize one of the most powerful tools available in the field of nanotechnology.

In Section 2 we will give an account of the main elements which build-up the FIB, namely an ion source and a set of lenses and scanning coils used to produce a finely focused ion spot which can be rastered and

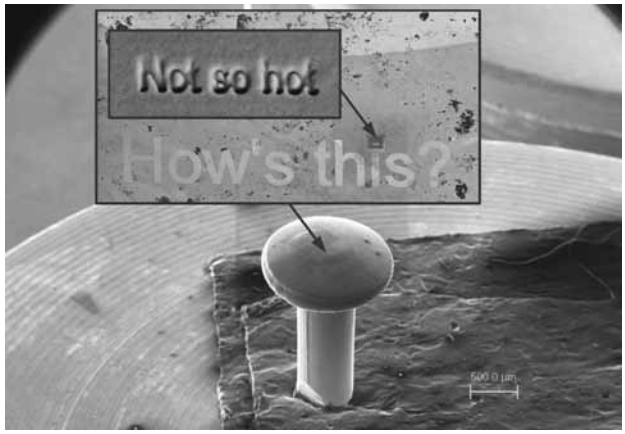

Figure 1: Feynman's experiment: "High school competition". The "o" letters in the smallest box have an internal diameter less than 400 nm.
Slika 1: Feynmannov eksperiment: "Tekmovanje visoke šole". Notranji premer majhnih črk o v majhnem okvirju je manjši od 400 nm.

pointed in the desired position to form images of the sample or mill specified shapes on it. In Section 3 we briefly describe the complexity of ion-solid interactions which for example allow the processes of sputtering and secondary electron emission (plus a plenty of other physical effects) used in the instrument operation. Finally, in Section 4 a sample of typical application will be described.

The aim of this paper is not to give an exhaustive description of the physics and the technology of focused ion beams, but rather to provide a fast introduction with mention to recent applications. For complete descriptions of the topic Refs. [2–5].

2 THE FOCUSED ION BEAM

The most important characteristic usually required to a FIB is the capability to mill very precise shapes in samples. This operation is performed by focusing in a very small spot the ions and moving this spot to the position where this localized sputtering is required. This is achieved through an ion source and an ion column where a set of lenses and scan coils are housed.

The performances that can be obtained in this "primary" task are first of all related to the spot size, the intensity and the stability of the beam. In principle the smallest and most intense is the ion spot, the most precise and fastest will be the job. In practice, limitations in the maximum removal rate that can be achieved may occur as effect of unwanted phenomena like redeposition [4], but this will be considered later.

2.1 The ion source

Like in optical systems, the fundamental ingredient to get a small and intense spot is a *bright source*, where the brightness is defined [7] as the differential current intensity d^2I emitted by the surface element dA of the source into the solid angle $d\Omega$, i. e. $B = d^2I/dA d\Omega$.

The most efficient and practical way to satisfy the requirement of a high brightness is through the so called *Liquid Metal Ion Sources* (LMIS). LMIS are nowadays the common choice for general purpose instruments, where no special demand exists for the ion species and we will limit our description to instruments were this solution is adopted.

In a liquid metal ion source, a field emitter (typically a tungsten needle with a tip radius of a few microns) is connected to a reservoir containing a liquid metal (or alloy). A heating system is provided for the reservoir if the chosen metal is not liquid at the temperature of operation. The metal, which can flow to the emitter, need to be a wetting liquid. In this case, if a voltage (the *extraction voltage*) of the order of 1 kV is applied by a nearby electrode the liquid assumes a conical shape which is the equilibrium configuration under the competing forces produced by the surface tension and the electrostatic field. In an ideal situation, where no further effect would occur, it has been shown [8] that an ideal cone with a half angle of 49.3° (called a "Taylor cone") would be created. In practice, the liquid cone is pulled by the electrostatic field until its end radius R reaches a value small enough to have an electric field that can start field evaporation [9] in proximity of the tip. In this situation any further reduction in the tip radius is inhibited and it has been shown [10] that the cone apex takes a rounded shape with $R \approx 1$–10 nm.

According this scheme, ion sources of Ga, Au, Si, Pd, B, P, As, Cu, Ge, Fe, In, U, Be, Cs, Li , Pb, etc. have been produced with brightness $B \approx 10^6$ A/cm^2 sr.

Particularly relevant is the implementation of the Gallium LMIS, which is currently the most common choice in commercial instruments. The reasons for this choice are manifold: the low melting temperature (29.8 °C) is certainly fundamental, since it simplify the system and prevent or reduce chemical and physical interactions between the liquid metal and the field emitter. Gallium atomic mass (69.72) has an "intermediate" value between light and heavy elements; this makes gallium ions suitable for efficient sputtering with a wide choice of substrates (the maximum energy transfer in a scattering event occurs when the target and the projectile have the same mass). Its physical properties like surface tension, vapour pressure and vacuum properties maximize the source lifetime. Overall gun performances have been demonstrated to be excellent with respect characteristics like angular intensity and energy spread.

2.2 The column

Although the mechanism of field evaporation is possible only for a very small R, the effective source size results to be much larger due to space-charge effects. In practice a virtual source size of the order of 50–100 nm has been estimated [11].

To reach a probe size on the sample of the order of 10 nm or less the image of the source need to be demagnified. This and other similar tasks are performed by the ion column. We will not enter into the details of this part of the instrument since the argument is mostly technical, but we will limit to a functional description of its components.

After being generated in the gun, ions are accelerated down the column by a voltage typically chosen in the range 5–50 kV. The optical system therein contained is usually composed by two electrostatic lenses. The choice of electrostatic lenses rather than electromagnetic ones like in SEM columns, is due to the fact that ions are much massive than electrons. This implies that if accelerated at the same energy, ions are much slower and thus the magnetic part of the Lorentz force is by far weaker.

The first element of a two lens system is the condenser whose function is to "collect" the ions and to set a suitable divergence for the beam before it passes

the forming aperture. The apertures shape the probe and reduce the current. In this way a range of different probe currents can be selected by changing the aperture (and eventually the condenser voltage). Typically the current intensity can be chosen from a few picoamps to a few tenths of nanoamps. The lens that follows is the objective lens that focus the beam on the sample.

Further elements contained in the column are a deflection/astigmatism unit and a fast beam blanker used for raster and mill operation.

2.3 Dual-beam instruments

As we will describe in Sec. 3, the ion-solid interaction is a quite a complicated process and the intuitive concept of a nano-mill for the FIB is valid only in a very rough approximation.

In practice, the result of even very simple operations like milling a square hole in a flat sample can give unexpected results as shown in **Figure 2**. For "unique" samples this can be a question of great concern for the operator which is responsible for analysis that cannot be repeated. The best solution to this problem has been to provide the way to have real-time visual inspection on how the work is advancing. The technical implementation of this solution are dual-beam instruments, where a SEM can operate simultaneously with the FIB.

In standard dual-beam instruments the electronic column enter vertically the high vacuum chamber where the sample is placed, while the FIB column axis has some angle with respect to the SEM, typically around 50°. In **Figure 3** the interior of a dual-beam instrument is shown, where the vertical electron column and the 54° tilted ion column can be seen.

The capabilities of this workstation can be further increased to make it become a complete nano-factory by

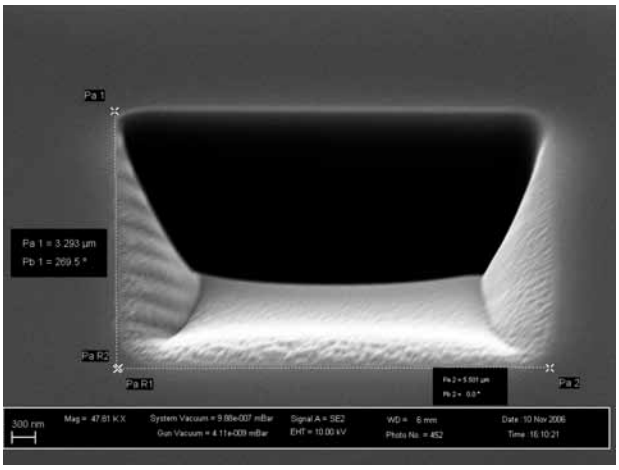

Figure 2: Effects of redeposition: the milling process had been set to produce vertical walls, but the final result shows a "mild" slope on the side milled first.

Slika 2: Učinek redepozicije: proces obdelave je bil nastavljen za izdelavo pokončnih sten, končni rezultat pa je majhen naklon stene, ki je bila izdelana najprej.

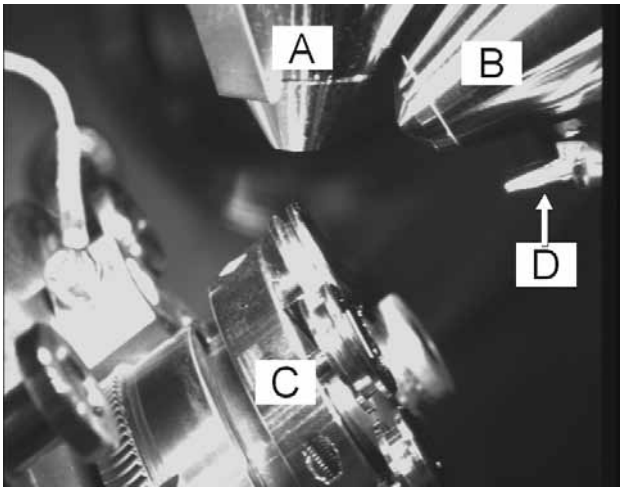

Figure 3: Interior of the sample chamber of a dual beam instrument. A: SEM, B: FIB, C: sample stage, D: GIS.

Slika 3: Notranjost komore za vzorec pri napravi z dvojnim curkom. A: SEM, B: FIB, C: mizica z vzorcem, D: GIS

equipping the FIB with a *Gas Injections System* (GIS) and micro-manipulators and eventually the SEM with an analytical tool like an EDS (*Energy Dispersive X-Ray Spectroscopy*) system or a WDS (*Wavelength Dispersive X-Ray Spectroscopy*) system [12].

The GIS is a mechanical arm inside the sample chamber ending with capillaries connected by valves to external reservoirs where different gas species can be contained. By placing these capillaries very close to the sample and opening one of the valves, the pressure can be locally increased without perturbing excessively the high vacuum condition needed for FIB and SEM operation. The gas injected in the chamber adsorbs on the sample surface. The molecules in this weak bond state can be broken by the ion beam impinging on the surface. Depending on the adsorbed gas, different processes can occur. If the gas is a precursor of some element or chemical compound, a part of the molecule becomes chemically bond to the substrate while the other part is removed by the pumping system. In this way, precise local deposition of metals like Au, Pt, W etc., or insulators like SiO_2 can be realized. Other precursor can instead be injected to enhance or make selective the ion etching process.

Micro-manipulators inside the sample chamber can be used to move portions of the sample extracted by using the FIB. A typical task is the preparation of lamellae for Transmission Electron Microscopy (TEM). In other cases they can be used like electrical probes for conductivity measurement during the work session.

Analytical tools provide a way to establish the chemical composition of the sample. In conjunction with the FIB capability to realize sections, these systems literally acquire a third dimension making it possible a volumetric mapping of chemical species in the sample.

3 ION-SOLID INTERACTIONS

It has already been mentioned that the most important operation that can be realized by a FIB is a spatially selective removal of atoms from the sample. From a general point of view, this process is called *sputtering* and has been studied in detail in a fundamental paper by Sigmund [13].

One of the initial observations reported by Sigmund is that it is unlikely for the first collision of the ion to produce a sputtered atom, since the transferred momentum (at least for normal incidence) has a component in the direction entering the surface.

On the contrary sputtering is one of the results of the so called "collision cascade" initiated by the ion entering the sample. This ion undergoes a series of collisions in the target and each atom which acquires sufficient kinetic energy by this collision can initiate a new series of collisions.

The physics of the collision cascade is complex and not fully understood. Moreover it cannot uniquely be defined since the open scattering channels can be different for different energy of the primary beam. We will limit our considerations to a very schematic classification.

A fundamental division of the processes that take place during a collision cascade is between elastic and inelastic events. Elastic processes are responsible for displacement of lattice atoms, defects generation (*amorphization*) and sputtering. Inelastic events generate secondary electrons, X-rays, photoluminescence and phonons (heat). From the point of view of the incident ion, each event causes a transfer of energy to the solid (a speed reduction) and a deviation from the direction taken after the previous collision. At end (when and if the initial ion kinetic energy is not sufficient to make it move freely in the solid) the ion can stop in the sample (*implantation*) if the random deviations occurred did not bring it again to the surface with enough energy to escape in the vacuum (*backscattering*). The energy losses can occur in "nuclear channels" with elastic events (for typical FIB energies mostly by screened Coulomb scattering) and in "electronic channels" where inelastic interaction with lattice electrons produce excitation and ionization.

Aside from sputtering, among the processes described, secondary electron generation is the most important. By utilizing the SEM detectors is in fact possible to generate images also with the FIB.

The resulting images can show contrast mechanism (like the contrast for the local crystallographic orientation) that are not present when an electron primary beam is used. On the other hand, this imaging mode suffer from a continuous modification of the sample due to the contemporaneous occurrence of sputtering events.

The processes described above are fundamental in nature. The actual phenomenology can be sometimes unexpected for the presence of further mechanisms. We describe briefly three of these mechanisms that can pose some limitation or at least make more complex the process of milling.

3.1 Redeposition

Atoms removed by sputtering or backscattered ions can actually fail to "escape" from the solid and instead they can deposit on the surface of the sample very close to the milling site. The effect is a degradation in the quality of the milling operation that can easily reach unacceptable levels as shown in **Figure 2**. This effect is more pronounced when trying to mill high aspect ratio features and when using high ion fluxes. Possible solution are the reduction of the current and/or the dwell time at the expenses of a longer process time. A different solution is the introduction through the GIS of a reactive gas like F, which binds to the sputtered atoms and facilitate the removal by the pumping system.

3.2 Channelling

Channelling occurs in crystalline material and is an apparent inhomogeneity of the sputtering yield across a chemically homogenous surface (**Figure 5**). It is due to the lower atom density along low index directions, resulting in a lower probability for the incident ion to hit a target ion.

3.3 Auto-organization

Off-normal incidence of the primary beam can make evident the effects of the instability generated by the dependence on the local curvature of the sputtering yield [14–18]. Since the erosion rate in depressions is larger than on surface mounds, any surface deviation from flatness tend to be amplified. The presence of a competing smoothing force due to surface atom diffusion produces typical structures showing long-range correlations. Easy to observe are wavelike structures usually indicated as ripples. In conventional milling processes the generation of these structures does not have particular relevance as long as height corrugations of the order of $\approx$10 nm can be neglected.

4 APPLICATIONS

The FIB has reached a relatively large diffusion thanks to its application in microelectronic industry started in the 1980s. The capability to remove or add atoms in selected sites with submicron precision makes the FIB an instrument which can hardly be replaced in applications like failure analysis, mask and integrated circuit (IC) repair. A classical example is the inspection of a buried IC, where conventional techniques employing mechanical tools to reveal the hidden parts can introduce artefacts that cannot be tolerated if resolution in the nanometre range is required.

As the diffusion in industry increased, in the 1990s, FIB systems started to be acquired by research labs: one fundamental application became the preparation of TEM cross-section lamellae. In this case several advantages can be indicated with respect to traditional techniques like the use of a ultramicrotome. First of all FIB preparation is site-specific: the lamella can be extracted from a selected location in the sample. This possibility is fundamental for structured samples as in the case of biological specimens. Then, in general, a fewer artefacts are introduced above all when dealing with samples showing a non-homogeneous hardness across the section or with very soft materials.

A further well-established application of the FIB is direct three-dimensional micro and nano-machining. These capabilities have used for MEMS (Micro-Electro-Mechanical Systems) and photonics structures realizations both in processes where only the FIB is used for rapid prototyping and in situation where finer details are added to classical lithography works.

Since all these topics are widely covered in the literature, in the remaining part of this paper we will consider relatively new applications like the production of solid-state nanopores, and the study of ion-induce self-organization processes.

They can serve as examples for two different ways to go beyond established limits of state-of-the art FIB instruments.

4.1 Production of solid-state nanopores

Nanopores produced in solid state membranes have been proposed as the key element for a new class of devices deputed to fast DNA sequencing or (in a more general case) characterization (see [19] and references therein contained).

In the basic set-up, an insulating membrane where a hole with size in the 1–10 nm range has been drilled, separates in two part a reservoir containing an ionic solution. By placing an electrode in each part and by establishing a voltage bias between the two regions, a ionic current starts to flow across the pore. If DNA molecules are inserted in the negatively biased region, they will tend to go through the pore and during the translocation a variation in the ionic current is expected. In principle, for pores small enough, it should be possible associate the instantaneous current variation to the single base which is occupying the pore in that instant. Variant of this scheme have been proposed, but all of them require an insulating membrane with a pore that is comparable in size with single-stranded DNA, i. e. a few nanometres in diameter.

Classical lithographic techniques do not have sufficient resolution to be used in nanopores production, and, in this case, even the FIB can not perform the task directly. It has been reported in several papers a minimum reproducible size for FIB drilled pores in typical Si_3N_4 or SiO_2 membranes ($\approx$100 nm thick) of about 30 nm. Techniques like *nano-sculpting* [20] and high energy electron irradiation [21–23] have been proposed and an effective shrinkage to the desired size has been demonstrated.

In the first case, a SiO_2 membrane, where pores with a diameter of $\approx$50 nm have been realized by using a FIB, is irradiated with a broad 3 keV Ar^+ beam. Under this irradiation, a gradual closure occurs as can be deduced from real time measurement of the transmitted ion current and final size less than 1 nm can be achieved. The process is explained through a model where adatoms (surface-diffusing mobile species), created by the incident beam, diffuse to the pore and fill it in.

In the second case pores of similar initial size are produced by electron-beam lithography and plasma etching in a SiO_2 membrane. Subsequent exposure to 300 keV electrons in a TEM reduces the size in a controllable way, with a precision of 0.2 nm (resizing occurs while imaging and this allows to follow the process). The claimed mechanism is a fluidization of the SiO_2 under

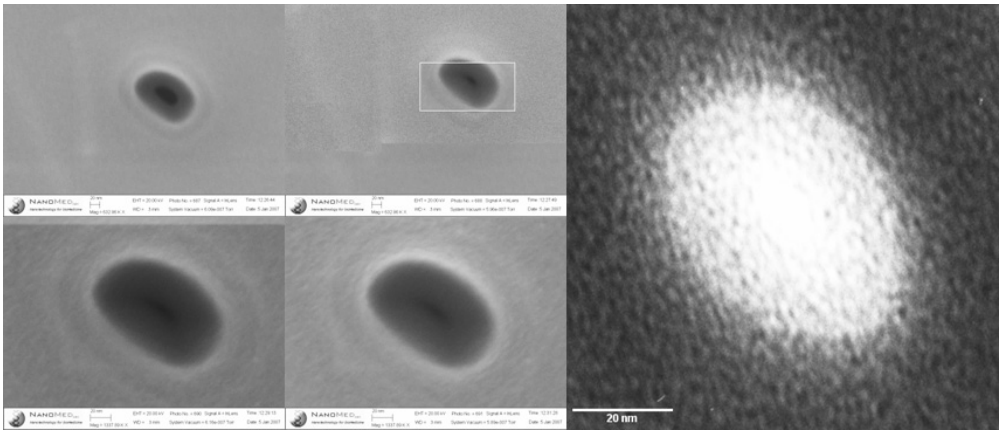

Figure 4: Sequence showing shrinkage of a solid state nanopore in Si_3N_4 under electron irradiation. In the last tab an ex-situ acquired TEM micrograph shows a final size smaller than 10 nm.

Slika 4: Sekvence kažejo krčenje nanopore v Si_3N_4 pri obsevanju z elektroni. Zadnji TEM-posnetek prikazuje, da je končna velikost manjša od 10 nm.

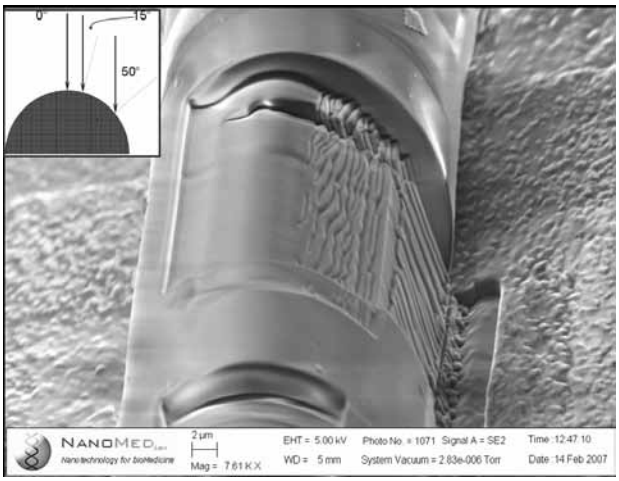

Figure 5: Ripple formation on a silicate bio-structure (spicule). The ion beam is orthogonal to the sample holder, but due to the cylindrical shape of the sample, its incidence angle increases with the radius. Ripple rotation for angles closer to grazing incidence appear, as expected from Bradley and Harper theory [14].

Slika 5: Nastajanje brazd na silikatni biostrukturi (spicule). Curek ionov je pravokoten na nosilec vzorca, zaradi valjaste oblike vzorca pa vpadni kot raste s premerom. Pojavi se zasuk brazd za kot blizu drsnega, kot napoveduje Bradley-Harperova teorija.

the high energy electron irradiation and a pore shrinkage under the action of surface tension.

Recently a new techniques that can entirely be realized inside a dual-beam instrument has been proposed and the results appear to be interesting. In this case the pore size is reduced during SEM imaging and the shrinking rate can be monitored simply following the image evolution. The resolution of this process is limited by the SEM capabilities to values larger than 1 nm. In **Figure 4** a size reduction sequence is shown. The final size is evaluated ex-situ in a TEM.

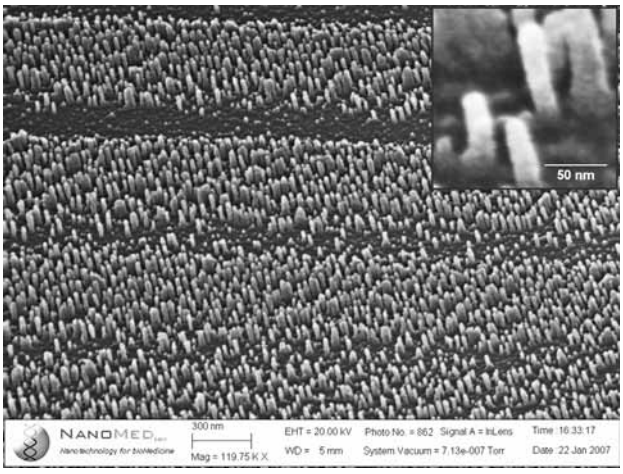

Figure 6: Pillar in silicon produced by off-normal incidence with a FIB. In the inset a particular at higher magnification clearly shows that the dimensions of the pillars is beyond FIB resolution.

Slika 6: Stolpci na siliciju, doseženi z nenormalno vpadnostjo v FIB. Slika pri veliki povečavi v okvirju prikazuje, da je dimenzija stolpcev pod ločljivostjo FIB.

4.2 Nanostructuring by self-organization processes

The self-organizations processes briefly described in Sec. 3 have been often indicated as a possible high throughput method for nanostructuring surfaces. Recently it has been shown that similar structures can be created also by using the FIB and moreover the long range correlation of this structures can be increased if they are produced on a surface where an ordered template has previously been milled. Although the high throughput characteristic of this technique is lost when the process is realized with a FIB (due to the relative low currents used with respect to broad-beam sources) nevertheless some advantages are obtained. First of all, the possibility to use higher local fluxes allows the exploration of regimes which hardly can be accessed with conventional guns. Then, the self-organization process can be induced with different condition in adjacent regions producing structures of higher complexity. Finally, dual-beam instruments offer the capability of following in a real-time mode the process. This can give the necessary feedback to overcome the lack of knowledge of all the variables entering the self-organization process that influences the capability to predict the final morphology of the surface.

From the point of view of the FIB user, the advantage of this technique is in term of increased resolution in producing particular structures as shown in **Figure 6**. Moreover for particular structures the process time can be greatly reduced.

5 CONCLUSION

We have reviewed the main characteristics of Focused Ion Beam technology with particular attention to dual-beam platforms. The applications that we have mentioned demonstrate the widespread diffusion that this kind of instruments have reached, diffusion that goes beyond the fields where the use was initiated, i. e. microelectronic industry. We have finally considered two new research fields where the FIBs can both play a fundamental role and find new ways to go beyond its current limits.

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