Focused Ion Beam – Fundamentals (Part 2)

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Large variety of applications of Focused Ion Beam (FIB) instruments arise from the combination of their features such as: imaging with secondary electrons or secondary ions with novel contrast mechanisms, precise sputtering of the material and thus creating a cut or cross-section (few tens of nanometers to a few hundreds of micrometers in size) and introducing different gases into the vacuum chamber to either deposit materials or selectively etch the samples.

The semiconductor industry was the origin purpose for FIBs invention [1-3] and nowadays is still one of the major beneficiary among many others. The selective sputtering or deposition of the material opened several applications such as circuit modification, diagnostics, failure analysis, specimen preparation, photomask repair and lithography.

The FIB application in materials science started in 1983 and in 1985 was supported with high resolution chemical mapping using secondary ion mass spectrometer (SIMS) [4-7]. At the beginning it was rather dedicated to SIMS or transmission electron microscope (TEM) lamellae preparation. However, the imaging itself with three primary contrast mechanisms appearing in FIB (orientation (channeling) contrast, topographic contrast and material contrast) and using modern dual beam instruments of high spatial resolution started also to attract more attention. The orientation contrast (Fig. 1) is especially helpful for the cubic metals while the material contrast is very frequently used in the case of corrosion or grain boundary oxidation studies as the secondary ion yields can be even three orders of magnitude higher for the metallic species in the presence of oxygen [8].

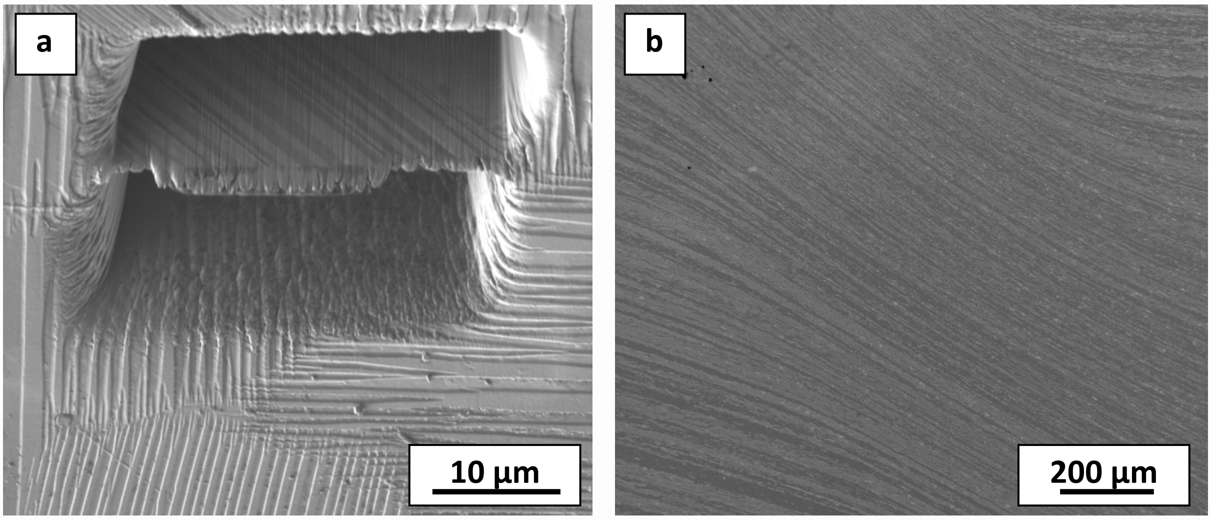

Fig. 1. Examples of the channeling contrast in FIB: self-accommodated martensite structure of Ni-Mn-Ga alloy (a) and aluminum alloy AA1050 deformed by Equal Channel Angular Pressing (b).

The TEM lamella preparation of the thin coating can be extremely difficult if possible using conventional techniques. The Focused Ion Beam deals with such coatings within few hours time and allows to observe the interface with the substrate beneath (Fig. 2).

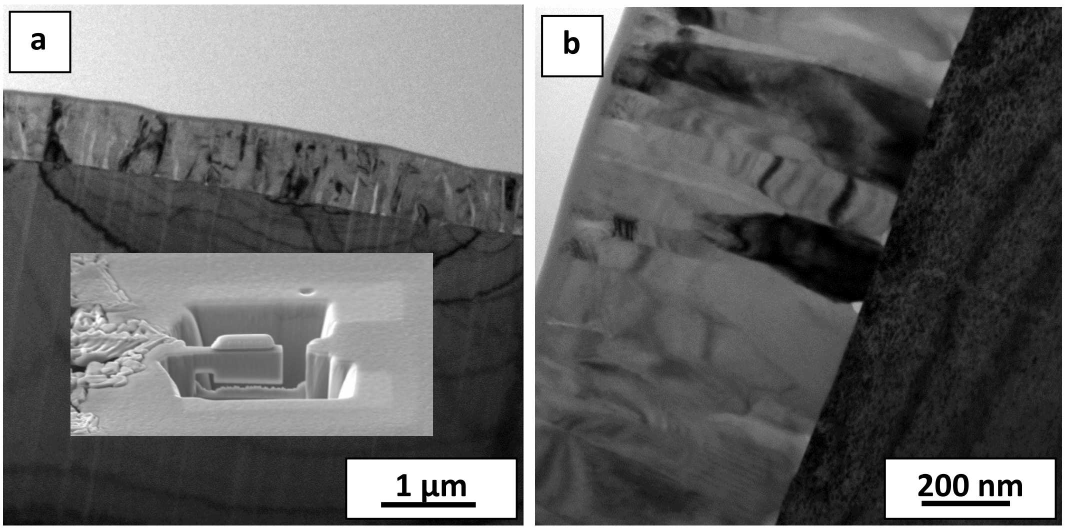

Fig. 2. TEM bright field images of the alumina and $ZnAl_2O_4$ layers on ZnO single crystal substrate under lower (a) and higher (b) magnification with the box showing one of the preparation steps in FIB.

Already in 1995 zinc coated steel was examined thanks to the FIB instrument, and later the whole spectrum of materials such as plasma sprayed metallic coatings and phenomena – corrosion, indentation, wear etc. has been studied. As it was mentioned above imaging with secondary ions is especially useful in corrosion studies and one may find the examples of them in the work of Phaneuf [9]. Observation of the stress-corrosion crack in an X-65 grade pipeline steel was performed by changing the bias from +400 V to -1500 V in ‘D’ shaped, coaxially mounted microchannel plate detector which allowed to obtain either the secondary electron mode or secondary ion mode giving drastically different contrast. Wang et al [10] came a step further employing FIB sectioning and imaging to study stress-corrosion cracking profiles.

Gentle milling with focused ion beam helps to avoid the deformation or closing of existing cracks by the mechanical abrasion. It also leads to the proper interpretation of the crack profiles and structure as well as the fracture specimens. It allows to observe many places at a bulk sample by the precise FIB sectioning revealing the internal sub-structure of materials. However, the redeposition can be an issue in such cases which can be overcome by final polishing using small currents (350 pA). An example of fracture surfaces inspection with FIB can be followed in work of Cairney et al [11] where the extent of interfacial debonding and matrix deformation of composite material consisting of iron aluminide matrix Fe-40 at. %Al reinforced with the ceramic particles (TiC, WC, TiB_2 or ZrB_2) was intensively studied.

Micro- and nanoindentation profiles can also be studied with focused ion beam. FIB serial sectioning was successfully applied for comprehensive description of the nanoindents within the Cu-Al multilayers on alumina substrate [12] or within W/TiN multilayers and directionally solidified eutectic oxides [13].

There are also reports [14,15] on the possible use of FIB in direct observation of the plastic deformation in cubic metals. The researchers concentrated on the inspection of the "mottled" grains contrast of either Al-based MMCs or Ni-based superalloy. Focused Ion Beam serves its stress-free sectioning also for all these samples for which the conventional mechanical polishing would affected their integrity (see example of porous composite material in Fig. 3d). The analysis of the internal delamination or cracking between the particles and matrix can be properly visualized by FIB sectional imaging.

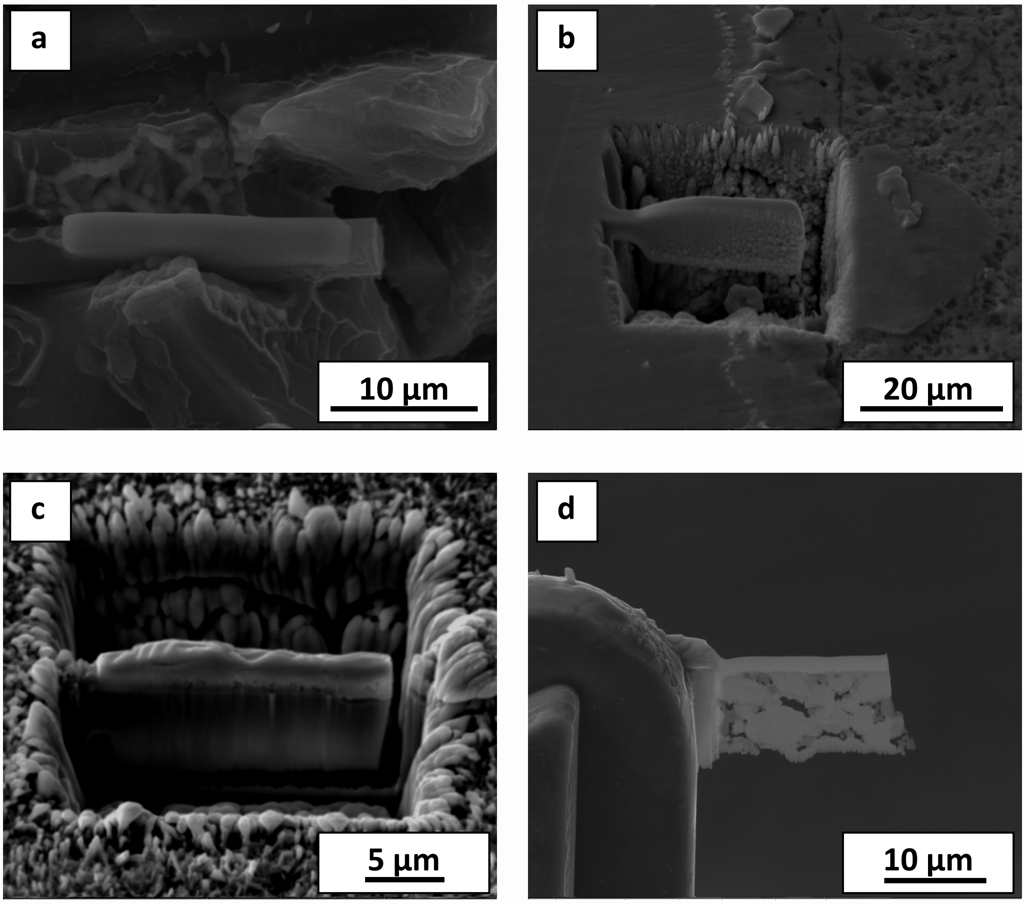

Fig. 3. Examples of the FIB applications for the thin foil preparation in various materials: 2024+Al_2O_3 composite with platinum protective layer placed on the top of alumina fiber (a), substrate (Ag)/solder (In) interface with the intermetallic phase in between after diffusion soldering process (b), corrosion studies of FeCrAl after oxidation in SO_2+1%O_2 (c) and porous lamella of Al-Si_3N_4-C composite obtained with FIB.

Powders are other matter possible and are easy to be studied with FIB, either by cutting and imaging the cross section or preparing the TEM lamellae. Examples of studies in this case such as: Mg-Zn mechanically alloyed powder particles [16], Ni powder [17], Zn powder [18] or Ti-Mg-Ni particles [19] clearly demonstrate the potential of the technique. Without any embedding, the powders were placed on the double-sided carbon type and sectioned in the FIB which took few minutes.

One of the most common application of FIB is the micro-machining especially used in semiconductor and data storage industries which comprising of the milling in the submicron scale to fabricate precise features on a sample. FIB makes it possible because of the small size of the ion beam which is additionally combined with imaging of the final result. Modification of the integrated circuits such as cutting or joining of wires and tracks, imaging of the faulty part, measuring critical dimensions and final shaping of pole pieces are just some of the examples of FIB use in semiconductor and data storage industries. Using FIB almost any microtool geometry including curved shapes can be fabricated on a small scale below those reached by grinding methods. In materials science FIB micro-machining helped to produce the tools such as nanoidentor tips, micro-end milling tools [20], tools for mechanical property testing [21], microelectromechanical systems (MEMS) and other devices including photonic devices and various sensors [22].

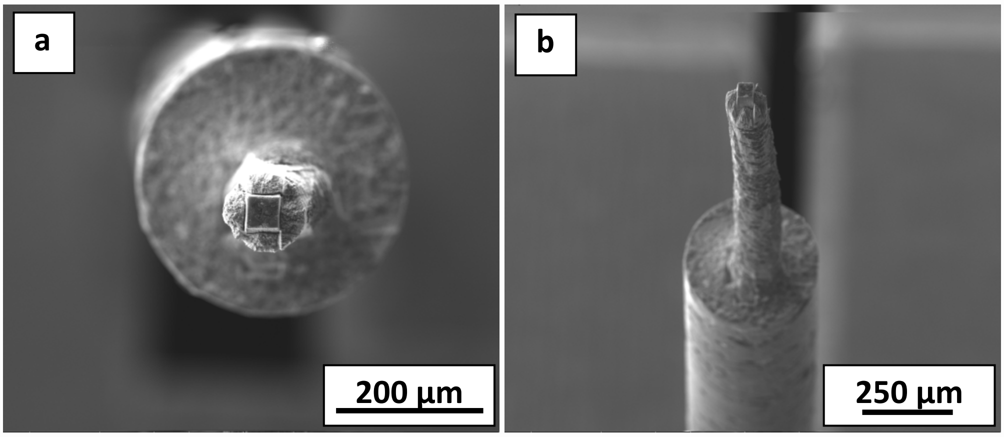

Fig. 4. Example of the FIB application in the micro-machining – shaping of the copper electrode tip: top (a) and side (b) view.

Although FIB sputtering is a relatively slow process it entails no large forces on the tool during fabrication. The time required for the fabrication was calculated in [23] as follows: a FIB column capable of producing 20 nA current would allow for fabrication of a 25 µm wide high-speed steel and tungsten carbide tool in less than 30 min.

Serial sectioning using mechanical or chemical means although commonly used since years cannot reach the level of lateral resolution provided with FIB. Characterization in 3D of the local structure, chemistry and crystallography of materials from couple hundred nanometers to a few microns scale is possible and successfully applied [see for example 24, 25]. Most serial-sectioning experiments rely on the capability of removing a known thickness of material per cycle with simultaneous keeping the surface flat. Many samples possess multiphase and polycrystalline microstructure which generates the problem of uneven material removal, however in single crystal superalloys (matrix and precipitates of similar milling rates) this methodology has successfully been applied.

Still the challenge for the FIB technique are polymers and biological samples. Although, gas assisted etching with xenon difluoride or water was successfully applied by the semiconductor industry to remove the polyimide coating and allow to modify the device modification or failure analysis, the polymer area is so far insufficiently represented. There are reports on results obtained with FIB for polymers nanocomposites [26] or polymers coatings on aluminum or silicon substrates [9,27].

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