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Plasma and Ions Help Slice and See Surfaces Better

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ABSTRACT

Focused Ion Beam (FIB) devices are versatile analytical tools in physics and materials research. Today, different FIBs, including gallium- and xenon plasma Focused Ion Beam Scanning Electron Microscopes (FIB/SEMs) as well as the Helium Ion Microscope (HIM) help answer research questions that no other technology can. This article looks into the physics of the FIB devices to help understand the difference between the effects of ion species (Ga, Xe, He) and FIB systems.

INTRODUCTION

Richard Feynman's speech "There is plenty of room at the bottom' [1] is considered to be one of the most well-known and influential speeches in science. In 1959, Feynman proposed that one day there will be technology available that can be used as our eyes and hands in the microscopic domain. In his visionary speech, he predicted the use of focused ion beams to see and manipulate matter at the tiniest scales. It would take another 40 years until the first Focused Ion Beam/Scanning Electron Microscope (FIB/SEM) would be available commercially. Gallium FIB/SEMs have established themselves as one of the key instruments in many facilities and labs due to their unique ability to cut a wide variety of materials with nanometre precision while analysing the sample and revealing sub surface features with nanometre resolution. Today, they are the go-to tool for transmission electron microscope (TEM) lamella preparation and are increasingly being used for site specific cross-sectioning, 3D reconstruction as well as nanofabrication [2], e.g. patterning graphene on SiC [3].

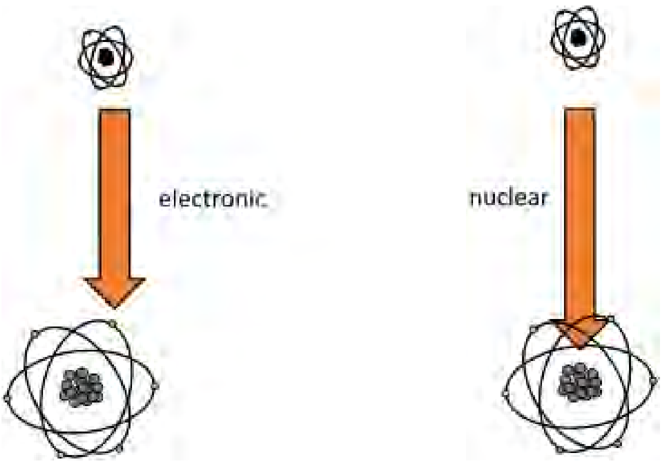

Fig. 1: Electronic and nuclear interaction of an ion with a sample atom.

Throughout the past 20 years, Ga FIB/SEMs have dominated the market. A focused beam of gallium ions, however, can have significant drawbacks. This becomes apparent when looking at the ion-solid interactions. As the ion beam hits the sample, the incident ions interact with the sample atoms in various ways.

ION-SOLID INTERACTIONS

The ions, no matter what ion species, lose their energy in collisions with the sample atoms (Fig. 1). With every interaction, energy is transferred (and/or lost). The entire energy loss dE/dx consists of both contributions, the nuclear (elastic) energy losses $[dE/dx]_{nucl}$ and the electronic (inelastic) energy losses $[dE/dx]_{\mathrm{lec}}$:

$$dE/dx = [dE/dx]_{nucl} + [dE/dx]_{elec}$$

What type of interaction occurs depends on the ion en-

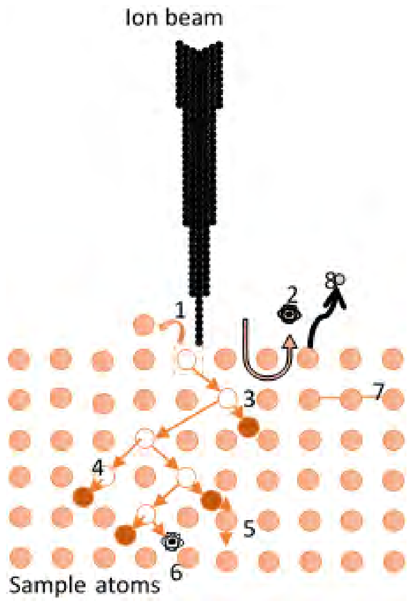

Fig. 2: Schematic of ion-solid interactions: The ion solid interactions can lead to (1) Sputtering, secondary ion emission; (2) back scatter ions, (3) dislocations and vacancies, (4) interstitials, (5) phonons, (6) ion implantation, (7) polymerization as well as (8) secondary electron emission.

ergy, the energy transfer, the ion species as well as on the sample itself (Fig. 2): the collisions can lead to sputtering of the sample surface atoms, secondary ion emission as well as backscattered ions, sample atom displacements such as vacancies, a collision cascade, replacement collisions and phonons. Secondary electron emission as well as polymerization are additionally caused by the ion-solid interactions. The interactions occur until the incident ion has lost all its energy and comes to rest in the sample at a certain depth, leading to ion implantation [4].

GALLIUM VS XENON

Significant problems arise when processing samples with a Ga FIB/SEM including phase transformations as well as changes in physical properties of the sample as a result of ion implantation [5, 6]. It has long been recognized that Ga changes the physical properties of semiconductors when processing them in the Ga FIB/SEM. It is less known that the gallium ions can accumulate along grain boundaries in aluminium, leading to a completely different deformation and fracture behaviour of the material [7].

To avoid the drawbacks which are associated with Ga FIB/SEMs, different ion species are explored in laboratories and utilized commercially. Today, systems with a

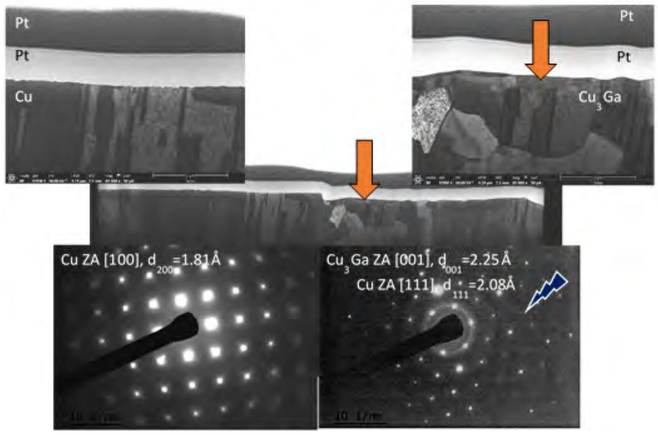

Fig. 3: Top: Scanning Transmission Electron Microscopy images of a TEM foil: Cu (left), Ga FIB irradiated Cu (right). The corresponding electron diffraction measurements (bottom) show that the irradiated area phase transformed to Cu_3Ga.

conventional gallium liquid metal ion source as well as multi-species (liquid metal alloy ion source) can be purchased. The plasma FIB/SEM technology, available since 2012, has had a major impact with Xe being one of the most popular ion species. Xe is inert, avoiding doping of semiconductors or alloying (such as Ga in Cu → Cu_3Ga, see Fig. 3) when processing samples with a Xe plasma FIB/SEM. Furthermore, the combination of a higher sputtering yield for a Xe plasma FIB than Ga (factor 1.5) and the possibility of using μA range currents (rather than nA range currents for the Ga FIB) makes patterning 30 times faster in comparison to a Ga FIB/SEM [8]. As a result, large area cross-sectioning or 3D volume reconstructions, with dimensions around 500 μm (in each direction) become feasible while maintaining nm precision of the cross-section placement. Being able to prepare site specific large area cross-sections/volumes and analyse those with the SEM *in situ* opens up new opportunities, especially in materials engineering, life sciences as well as geology. These applications often require larger area removal which was not feasible with Ga FIB/SEMs previously. A recent report [9] revealed that Xe plasma-FIB is a superior instrument with respect to Ga FIB in slicing materials for TEM analysis. Reduced amorphization layer thickness and the absence of residual Ga contaminants improve the TEM lamella quality significantly.

HELIUM VS XENON

Let us briefly recap the physics of operation of the Helium Ion Microscope (HIM). A lighter ion species like He predominantly interacts with the sample atom electrons

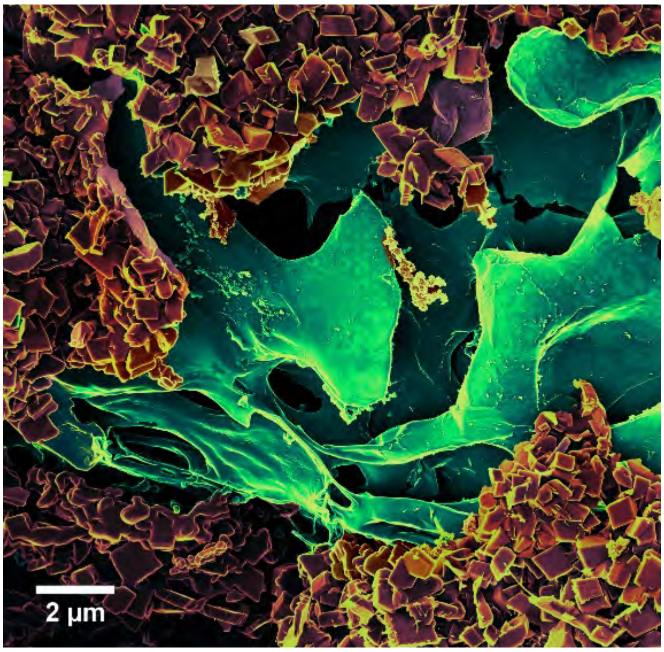

Fig. 4: Colorized HIM micrograph of MoS_2 on graphene.

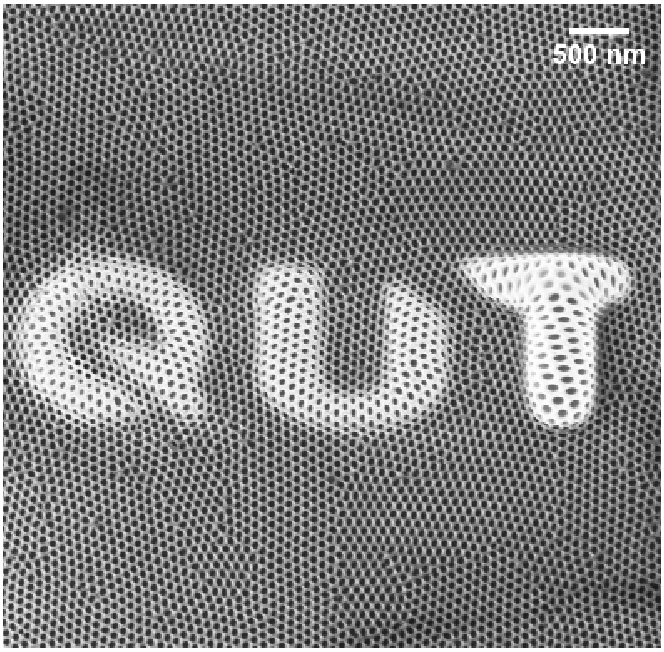

Fig. 5: Defect engineered aluminium oxide on Si, reprinted from Aramesh, M., Mayamei, Y., Wolff, A. *et al.* Superplastic nanoscale pore shaping by ion irradiation. *Nat Commun* 9, 835 (2018). https://doi.org/10.1038/s41467-018-03316-7

(inelastic collisions), producing many 'secondary electrons' (roughly 10 times more signal than is created when using an electron beam in an SEM) which can be detected using a conventional Everhart Thornley detector [10]. The beam stays collimated within the secondary electron escape depth. This in combination with its atomically sharp and cryogenically cooled source gives the HIM its superior imaging capabilities in nanomaterials research (Fig. 4) [10]. Nuclear interactions still occur and become statistically significant and lead to sputtering when using higher ion doses. This gives rise to the sub-10 nm fabrication capabilities of the HIM when these interactions occur near the sample surface. It is not well recognized within the science community though, that nuclear interactions become the dominant interaction type below the sample surface once the He ion's energy has fallen below 1 keV as a result of the ion-solid interactions. As the atoms cannot be removed under such conditions from within the material, dislocations are created and this can be used for defect engineering, creating novel material properties [11]. Figure 5 shows a defect engineered aluminium oxide layer on silicon which exhibits superplastic behaviour, opening an exciting opportunity to write "nano-tattoos" on solid surfaces.

Heavy ions like Xe predominantly interact via nuclear interactions in the entire energy range that is available in FIB/SEMs, making these ions ideal for sputtering samples. The new Xe plasma FIB/SEM (Fig. 6) very recently installed at QUT [12] is therefore ideally suited for preparing and analysing large area cross-sections, large volume reconstruction and TEM lamella preparation with reduced amorphous layer thickness. It also extends QUT's HIM noble gas structuring range, making it possible to precisely fabricate features ranging from sub-10 nm (HIM) to > 500 µm (Plasma FIB).

Fig. 6: Tescan S8000X Xe FIB/SEM installed at QUT. This system is equipped with Oxford Symmetry EBSD and Ultim Max EDS. Image courtesy Tescan.

CONCLUSION

Using different ion species and focused ion beams (FIBs) presents new opportunities to study as well as create materials of tomorrow. From using the Xe plasma FIB/SEM to prepare high quality site-specific cross-sections or TEM lamellae of various materials to the HIM's ability to study the surface features and to defect engineer novel materials, the underlying physics of the ion solid interactions helps choose the appropriate ion species for the job.

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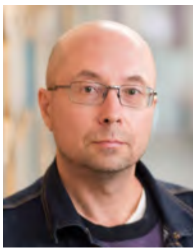

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