Lab 1: Scanning Electron Microscopy

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Working Principles

The main components of a Scanning Electron Microscope are –

i) **Electron column**
ii) **Detectors**

Electron Column

Here electron beam is produced, accelerated and focused before hitting the sample. This further consists of –

a) **Electron gun** – Here electron is produced by either thermal or field emission. In Zeiss Ultra FESEM, a Schottky Field Emitter is used.
b) **Condenser lenses** – These are electromagnetic lenses which focus the electron beam.
c) **Scanning coil** – This is used to displace the electron beam horizontally, and raster scan the sample.
d) **Stigmator** – If the beam is not perfectly shaped, the image obtained will be distorted; this is known as astigmatism. Stigmator lenses are used to correct the beam shape and eliminate this artefact.
e) **Beam booster** – Zeiss column includes an extra component called beam booster, which accelerates the beam to a higher energy while it travels through the column. This helps reducing chromatic aberration.

Detectors

The electron beam interacts with the sample to produce different kinds of electrons which are, in turn, detected by the following detectors –

a) **BSE detectors –** These are used to detect backscattered electrons and gives atomic number contrast of the sample. There are two types – ASB (Angle selective), placed near the pole piece and ESB (Energy selective), placed inside the column.
b) **SE detectors** – These are used to detect various kinds of secondary electrons, giving the topographic contrast of the sample. Two types of SE detectors are used – In Lens, mainly used for SE1 detection and Everhart-Thornley detector, mainly used for SE2 detection.
c) **EDS detectors** – Energy Dispersive Spectrometer
d) **WDS detectors** – Wavelength Dispersive Spectrometer
e) **CL detectors** – Cathodoluminescence detector

Objectives

1. **Identifying all parts of our ZEISS Scanning Electron Microscope –** Done in the lab. The components discussed above are identified.

2. **Understanding how cleanliness is important –** If the working environment or the sample used is not clean enough, dirt can get accumulated in various parts of the electron column which may cause the whole system to render unusable. Also, if the sample is an organic substance, as electron beam hits on it, it may produce some organic vapour which spreads throughout the electron column and causes the pressure inside the column to increase. This

in turn decreases the mean free path of the electron inside the column and creates problem in focusing the electron beam on the sample properly.

3. **Understanding various ways of mounting the sample –** The sample can be mounted in several ways –
    - <u>A section only</u> - If a conductive sample can be sectioned to a size that fits easily into the jaw holder, then it's enough. Most of the time, a sample can be mounted on a plug using tape or adhesive. If the sample is non-conductive, it should be mounted before coating so that the coating will cover both sample and plug. Otherwise a conductive strap has to be painted from the plug to the top of the sample.
    - <u>Bakelite/epoxy mounts</u> – Often a sample is mounted in Bakelite or epoxy for optical examination before performing SEM. Such samples should fit into the cylindrical holders exactly. A spring in the bottom holds the surface flush with a retaining ring on the top of the holder. As Bakelite and epoxy are not conductive, a conductive strap has to be painted from the conductive sample to the sample holder, if it is not carbon coated.
    - <u>Powders</u> – They are always very difficult to mount. Most of the time carbon tape or paint is used as the adhesive base for powders. Powder is sprinkled with a spatula and pressed lightly to seat. Then canned air is sprayed to remove the loose particles. For particle size image analysis, it is important for the particles not to touch one another. In such case, fine powder is dispersed in some polar liquid and several drops are put on a glass slide. The liquid is allowed to evaporate and the particle gets mounted on the surface.
    - <u>Biological specimens</u> – The sample is degassed and put in a desiccator for several days so that no more vapour is left in it. Then it is placed on a mount on a spot of carbon paint to hold it in place and then coated with a layer of gold.

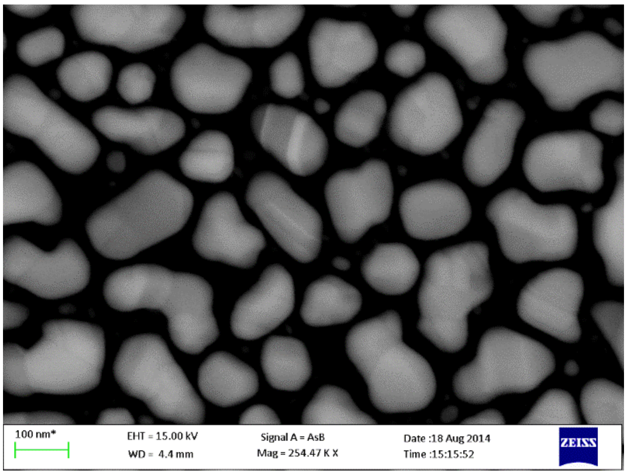
4. **Understanding various ways of coating the sample –** Carbon is the most common coating material because it is cheap and almost invisible to most x-rays. Gold or gold-palladium is preferred if the sample has a very irregular surface and compositional analysis is not required. Both coatings are usually applied at a thickness of about 20 nm, which is too thin to interfere with dimensions of surface features. Carbon at that thickness will have little or no effect on elemental analysis.

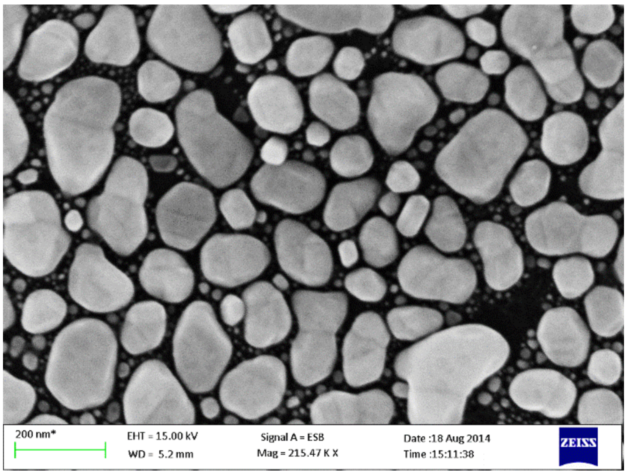
5. **How tilting the sample can damage the pole piece –** When the sample is tilted, it can actually hit some of the sensors present on the pole piece and thus can damage them.

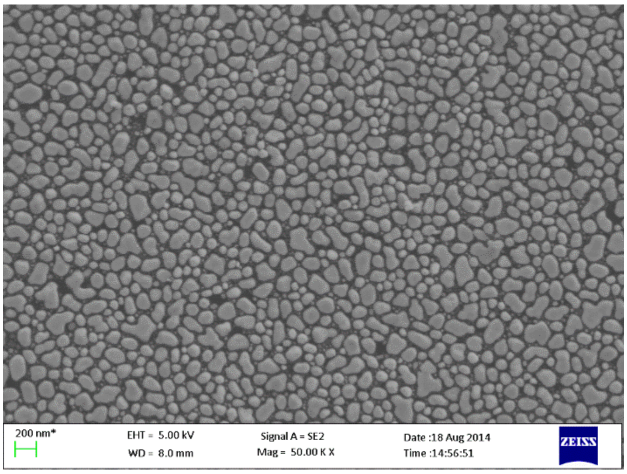
6. **Introducing sample in the SEM chamber –** The chamber is always maintained in very low pressure. Creating such high vacuum takes a lot of effort. That's why, before the chamber is open, the electron column is separated by an air tight partition to preserve its vacuum. After mounting the sample, the chamber is closed and the air is pumped out using rotary pump and then turbo pump. Only after the pressure in the sample chamber is sufficiently low (~ 10^{-5} mbar), the partition between electron column and sample chamber is removed and further pumping is carried on to reach the desirable pressure value.

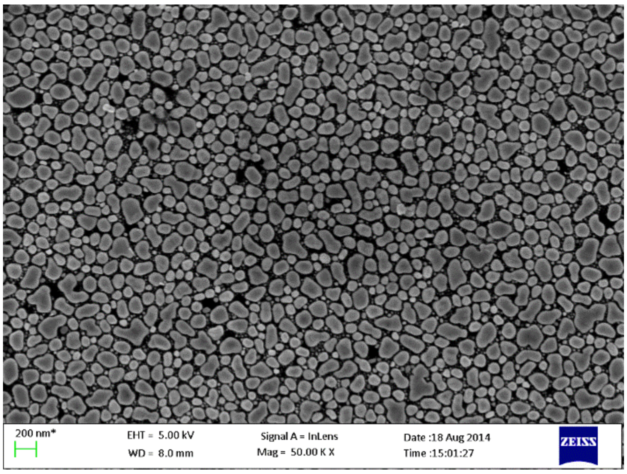
7. **Au nanoparticles on C, imaged with ET, In-lens SE, AsB and EsB detectors** –
    a) ET and in-lens SE detectors provide images with better topographical information, the ET image being superior.
    b) EsB detector provides good atomic contrast.

c) AsB detector provides channelling contrast, eg internal features like grain boundaries are more prominent.

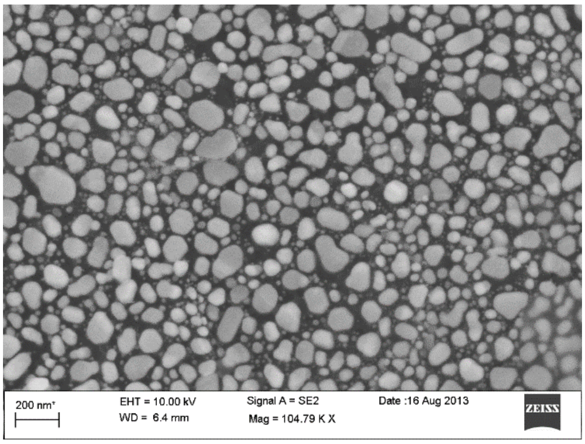


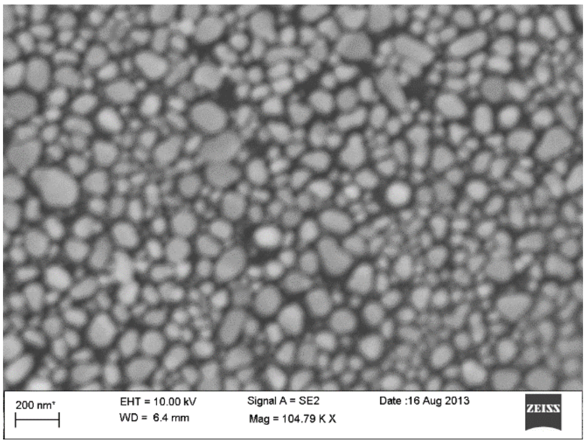




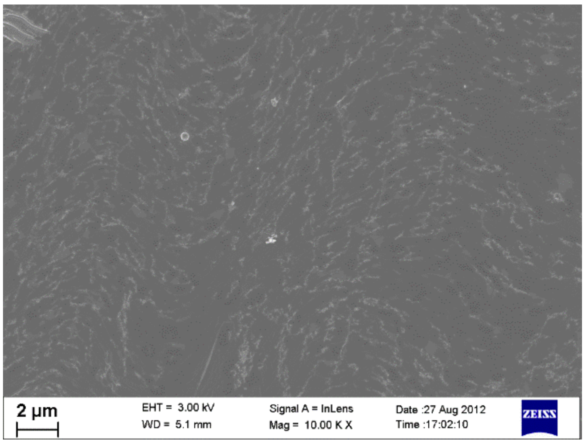


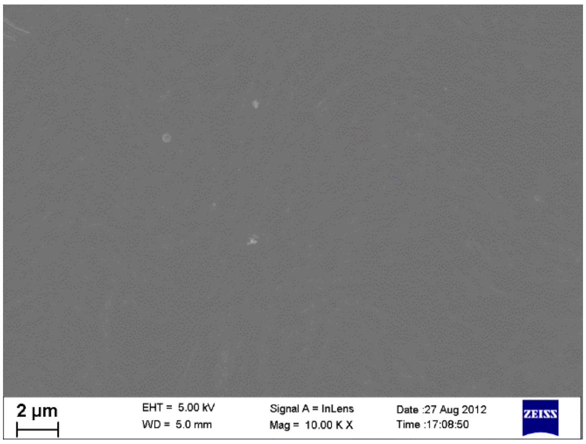
8. **Images of the previous sample is taken with poor and corrected stigmation** – With poor stigmation, the image is blurry, which is resulted from imperfect beam shape. Astigmatism is corrected using stigmator lenses, by adjusting two stigmation parameters, one along x, and the other along y direction.

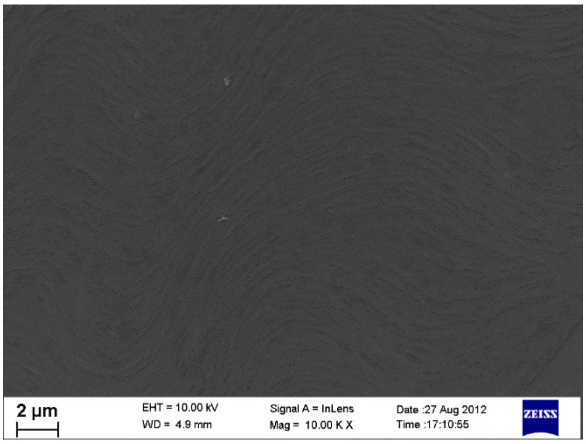


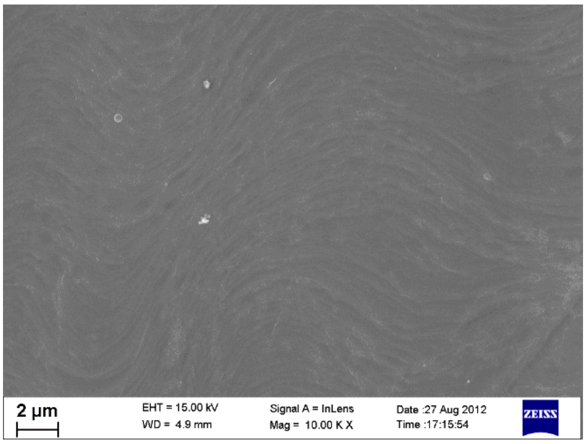


9. **Graphene on Cu sample is imaged with an in-lens SE as well as ET detector at 3, 5, 10 and 15 kV –**
   a) Image from in-lens SE detector is best at 10 kV. At lower kV, surface topography is unclear, and at 15 kV, characteristics of the copper substrate starts to show.

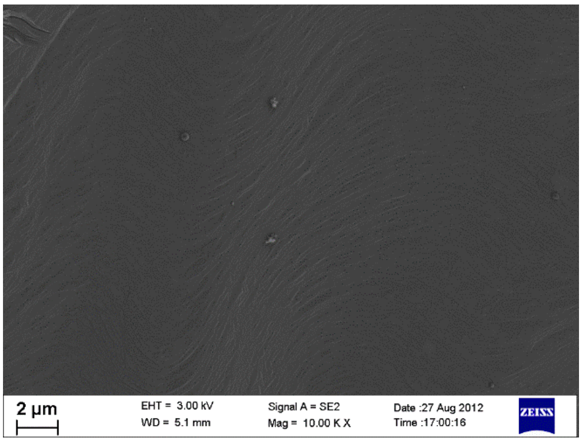


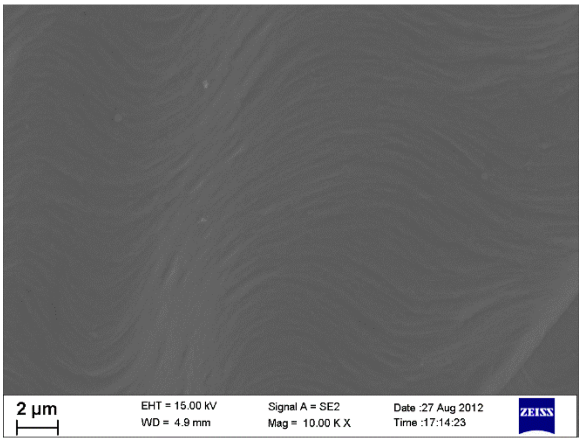


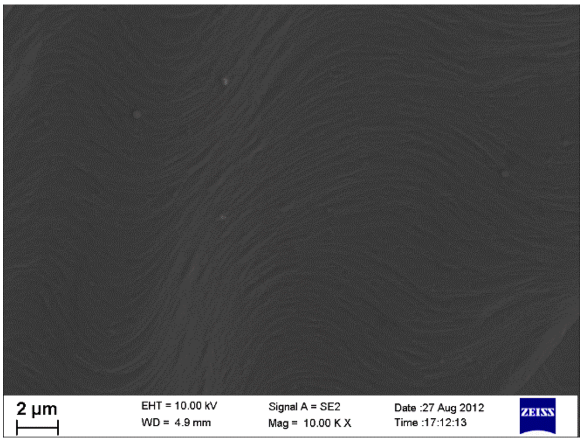


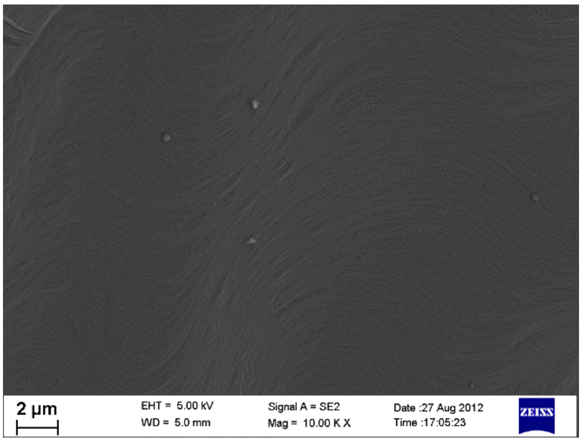


   b) Images from ET, in general have more topographical information compared to in-lens SE detector. Also, the contrast decreases with increasing kV.

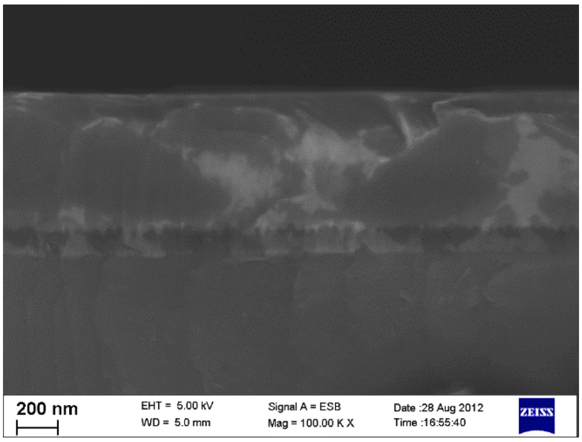


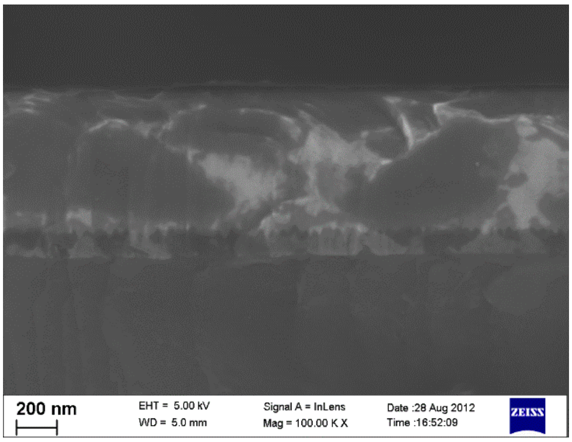






10. **AlGaN/AlN/Si is imaged with EsB, in-lens SE and ET detector** –
    a) The image from EsB provides better atomic contrast, and so the three layers are distinctly visible. Image from In-lens SE is brighter compared to EsB image, as at 5 kV, Se dominates over BSE. Finally, the ET image has maximum topographical information



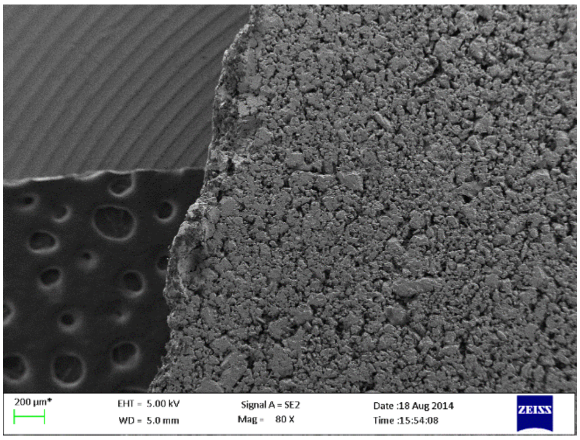


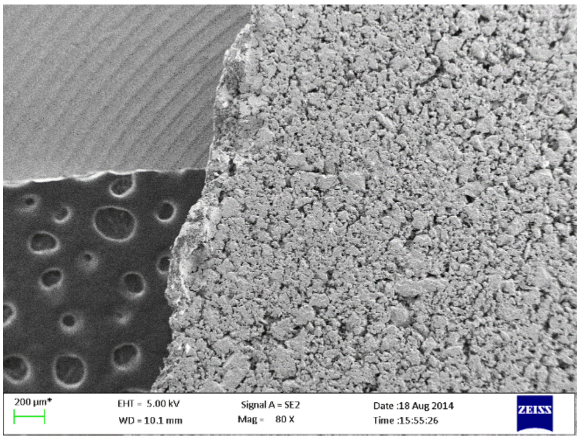
    b) At 25 kV, yield of SE decreases, so the image from ET loses the topographical details.

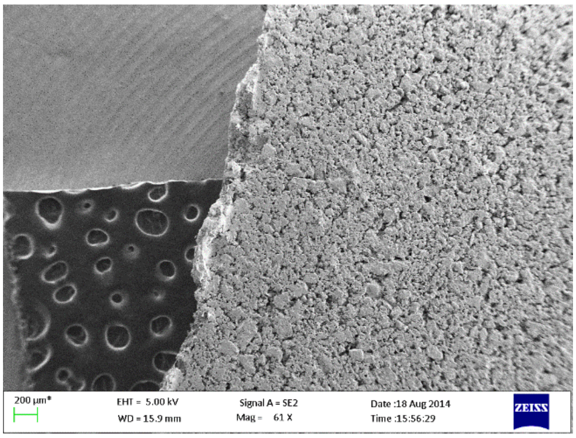
11. **An EDAX map of the previous sample** –
    Percentage of Al, Ga, N and Si is mapped from corresponding K lines. The weight % and atomic % of these elements in the cross section is found as follow –

Element	Weight %	Atom %
N	17.42	38.11
Al	5.20	5.90
Si	33.76	36.82
Ga	43.62	19.17

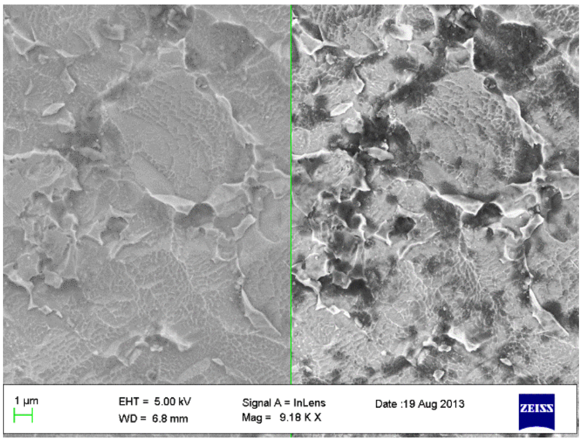
12. **Image of Ti-Sponge mounted on C-tape is taken at working distances 5, 10 and 15** – With increasing working distance, the focus carbon tape improves, while the top surface of the Ti-Sponge sample is always kept in focus. So basically, the depth of focus increases with increasing working distances.

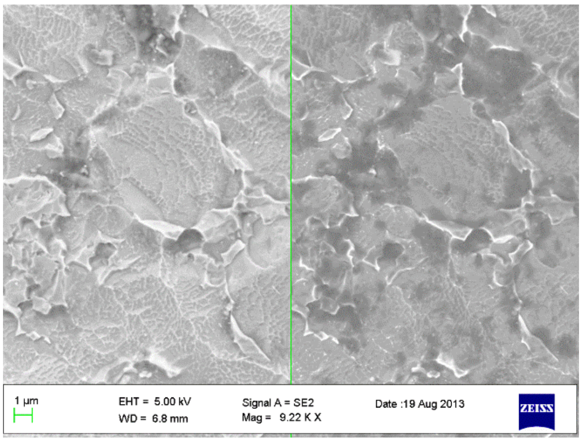






13. **Ti-6Al-4V alloy is imaged at 20 µm and 60 µm aperture sizes** – The brightness of the image increases with aperture size, while resolution decreases for both in-lens SE and ET detectors.





14. **Previous sample is imaged with a tilt of 20^0** – This leads to more efficient detection of topographical information, which are not clearly seen in normal incidence.