Imaging exotic nano structured compositions, polymers and protein with Low Loss BSE electrons in a Field Emission SEM

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New materials require new technologies. The smaller the investigated structures, the more sophisticated instruments are required. The imaging of nano-materials and composites is increasing in importance and therefore the role of detecting the smallest compositional differences in these materials is becoming progressively more significant. Due to the high depth penetration of electrons in classical BSE (back-scattered electron) imaging at high landing energies, this technology is not suitable to image nano-compositional materials with very small compositional differences. Electrons penetrate and diffuse deeply into materials at high landing energies. The lower the density of the material, the higher the penetration. This has the potential to make nano-particles and nano-layers "invisible" due to the large scattering volume. Therefore there is a demand for a new detection principle, able to meet the challenge of imaging against the background of these requirements. Below a landing energy of approximately 5 kV, the backscatter coefficient becomes non-linear and reduces with increasing atomic number more strongly than it is the case with elements with low atomic numbers. This behaviour is described in Reimer et al. The graphs in Fig. 1 clearly illustrate this non monotonic behaviour. We see a dramatic drop of the backscatter yield below 5 kV from the high Z elements (A), whereas low Z elements show an increasing BSE yield. Depending on the landing energy we have to expect a contrast reversal from certain elements (Fig 1B). This means that the higher atomic number element appears darker than the element with the lower atomic number. Especially between the elements 20 and 30 we see this behavior at landing energies between 1 and 3 kV. As an example this phenomenon is imaged in the Fig. (2,3,4) between Cu (Z=29) and Zn (Z=30). In the right image acquired at 3.9 kV we see the „normal“ contrast between the alpha and beta phase in brass. The Zinc rich areas are brighter and the Cu-rich areas are darker.

However, at a particular landing energy there is a equilibrium of different scattering mechanisms and no contrast is observed. When confronted with the problem, that the mean free path length of BSE’s from low-density materials such as proteins or polymers becomes extremely small, we have introduced new technologies to make these small signals visible. The most important item of technology in achieving this requirement is the GEMINI® lens with integrated beam booster. Independently of the required landing energy, the beam booster maintains the electron probe at a high energy level over the full length of the column. This essential design detail means the brightness of the electron probe is maintained at low energies and an amplification of the electron signal is achieved in the reverse ray path into the column. Since the GEMINI® lens with beam booster has a strong dispersion in the reverse imaging plane, the SE’s and higher energy BSE’s are separated in real time and are projected towards the individual detectors. A energy filtering grid in front of an axial in column BSE detector tuneable between 0 to 1500 volts allows a separation of electrons with very small energy loss, here called low loss BSE. The technology will be explained in the lecture. Electrons with a very small energy loss originate from the outermost shallow region on the surface. In experiments it was found, that the signal comes from less than 5 Angstrom. This means that we can see mono layers from atoms and molecules.

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Being in search of the ultimate contrast challenge I found that it is possible to see with this technology the contrast between metals and their oxides, -nitrides and -carbides. This is interesting because we have the same number of protons in the nucleus. Examples will be given in the lecture. Due to the filtering process only electrons with a very small energy loss are imaged. These electrons escape from a volume of less than 5 Angstrom. Examples will be shown on Ligand bondings and Graphen carbon layers.

Experiments were made to characterize also hybridisation states of carbon. It is possible to see sp2 and sp3 hybrids (Fig.5,6)! In conclusion we open with this LL-BSE technology a new world for the characterization of really exotic materials. Seeing different oxidization states of metals, hybridization of atoms, ligand bondings and even mono layers of atoms and corrosion enables the nano technology to characterize materials never possible before. Examples will be shown in the presentation.

Literature: Reimer L., Scanning electron microscopy 2nd Edition, Springer-Verlag Berlin Heidelberg New York 1998

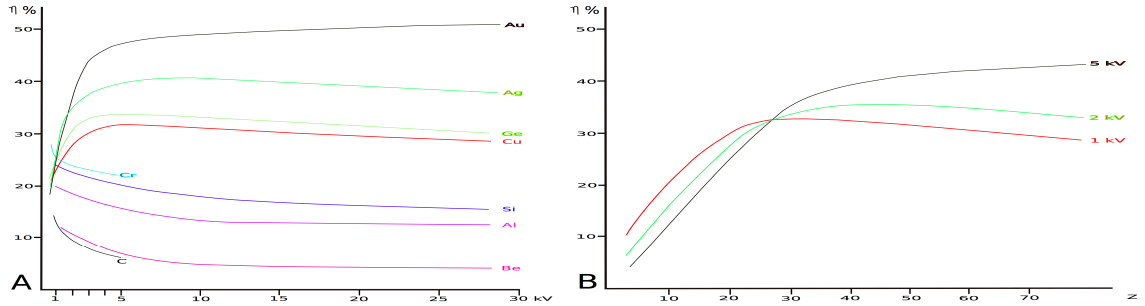

Figure1: Backscatter coefficient as function of landing energy and as a function of atomic number Z (after:Reimer 1998)

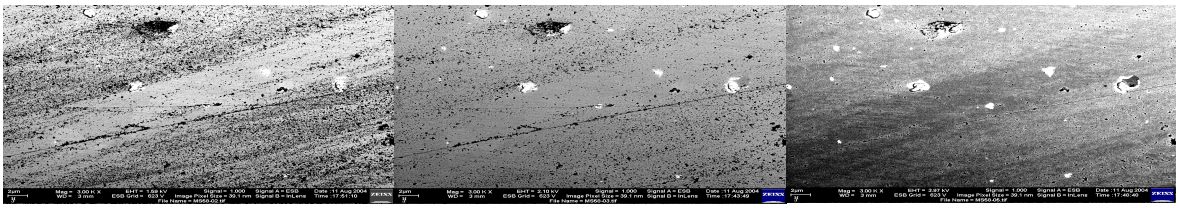

Figure 2,3,4: Contrast reversal with Cu (29) and Zinc (30) in Brass at landing energies 1.59kV, 2.1 kV and 3.9 kV.

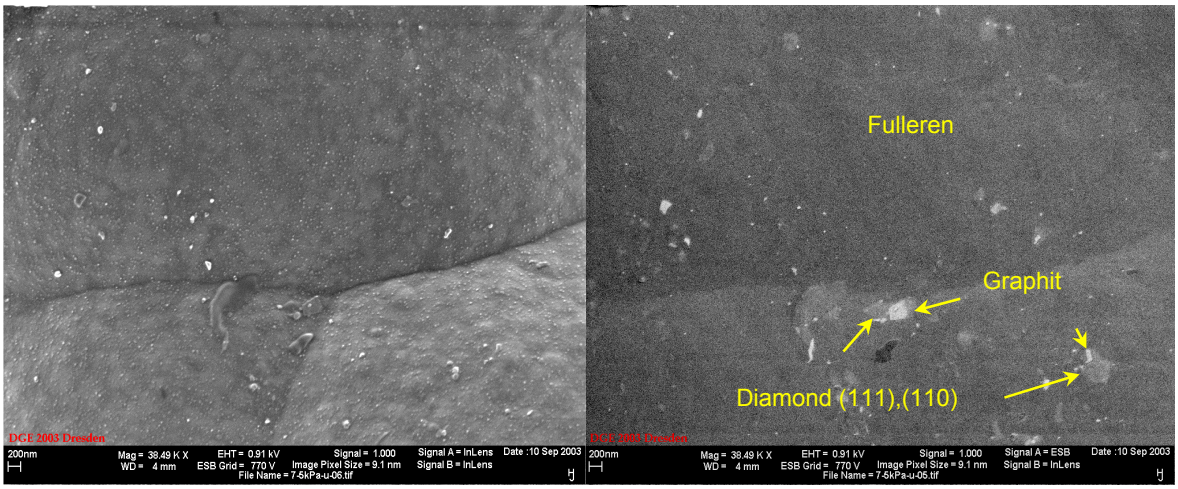

Figure 5,6: SE electron image at 910 Volt landing energy of 3 modifications of carbon (left) and the corres-ponding LL-BSE image with 140 eV low loss energy window, showing the sp_2 - sp_3 hybrids Diamond, Graphite and Fullerene. Note: Contrast inversion between Diamond and Graphite due to bonding energy difference !

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