Technologies to Characterize Nanostructured Particles and Bulk Materials

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Elastic and inelastic scattering at single atoms and excitation of electrons in solids are the fundamental processes that influence the range of electrons, the depth distribution of ionisation and the emission of SE and BSE electrons. New technologies are introduced to separate the different information coming from different scattering processes and hence better characterize nanostructured materials.

Due to the GEMINI principle 1,2,3 , the information, coming from the different scattering processes, is separated and projected via the GEMINI lens to different detectors (SE and *EsB*) on the beam axis. The signal is *directly* detected without any losses from conversion plates. This principle enables the detection of very low Z–contrast differences, far below from a standard BSE detector. Latest tests revealed a detection limit of ppm range dopants in low Z material (Boron in Al2O3). Even different polymers can be differentiated with this technology, due to the fact, that the signal coming from the sample, is amplified by a beam booster. The principle here is *Energy* selected BSE detection (*EsB*).

New improvements come from an integrated GEMINI lens detector, selecting and separating the BSE signal via Z contrast and angle contrast. While the high angle BSE electrons, which are detected in the unique in column *E*sB detector, where the electrons are separated via the *E*nergy, the large and very large angles, coming from different scattering processes are collected in the *A*sB detector, or *A*ngular selective **B**SE detection system.

In homogene crystalline bulk materials like metals, or ceramics, we have strong demands to characterize this material and determine the treatment history (cooling, walking, strain). Chemically etching the polished surface is a common technology to describe grain structure (Fig. 3). Sub grain information is lost due to damage. The channelling contrast, coming from mainly Mott scattered electrons, highlights this mechanism, which is used in this detector. The GEMINI lens separates the single scattered electrons from large angle *multiple* scattered BSE electrons. As a result we detect unmatched crystalline contrast, where normally nothing is visible (Fig 1,2,4). The change in the lattice orientation in some samples is typically less than 0.5 degrees in sub-grains, which is difficult to be detected with EBSD technique (Fig. 1, 4).

Similar to our general GEMINI detection technology – fishing the true (interesting) signal and suppressing the noise – we separate the different scattered electrons in the multimode STEM detection system. Elastic (BF) and inelastic (DF) scattered electrons are separated and detected as usual. Beyond this, we image large angle scattered (LAADF) electrons and look for the orientation of crystals. Similar to the TEM imaging of a series of reflexes, different orientations, strain or defects are visible at a glance. We can detect features, absolutely not visible in normal BF or DF. The interference of Bloch waves in stressed crystals give information about growth stress or doping stress in nano particles (Fig.5, 6). Altogether six basic imaging modes are used in this detection system, which is an extremely useful technology to characterize nano-structured materials. References

[1] Jaksch H. et. al. Microsc. Microanal. (2004) 1372 CD

[2] Jaksch H. et. al. Microsc. Microanal. 9 (Suppl) (2003) 106

[3] Jaksch H. Field emission SEM for true surface imaging and analysis, Materials World, Oct. 1996

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Fig 1:Subgrains and sharp grain boundary in AluFig 2:AsB channeling contrast in pure AluminumPolished but unetched surface. AsB detector imageOnly polished but unetched surface.



Fig. 3:Classical etched steel with grain boundaries Fig. 4: *AsB* image of steel showing finest grain and subgrain information. Only fine polished.



Fig. 5: Classical DF image of LaFePd-Oxide.

Fig. 6: The same sample clearly characterized in the orientation DF mode of the STEM system.