High resolution X-ray microanalysis and imaging with state of the art FE-SEM

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The new generation of Field Emission Scanning Electron Microscope (FE-SEM) can perform high resolution imaging at incident electron beam energy below 1 keV. Images with resolution smaller than 2 nm are now guaranties by various manufacturers. Since imaging below 1 keV allows obtaining surface details of nanomaterials and reduces beam damage for sensitive materials, it is clear that electron microscopy is now entering in a new era. With FE-SEMs that can operate in the 50 eV to 30 keV mode with many images modes like conventional bulk secondary electron (SE) or backscattered electron (BSE) imaging or new scanning transmission electron microscopy (STEM) imaging of transparent materials in bright field or dark field mode, the versatility of these microscopes is obvious. Also, if we keep in mind that we can also perform quantitative x-ray microanalysis with state of the art SDD EDS detectors and crystallographic characterization of materials with EBSD detectors, FE-SEM has a very bright future. Its importance in science and technology will growth faster than ever because we have now microscopes that can deliver enough current with high spatial resolution to fully exploit the advantages of low voltage scanning electron microscopy as predicted by Von Ardenne as far as 1942. This paper will present new results for the characterization of various nanomaterials obtained with the Hitachi SU - 8000 cold field FE-SEM. This FE-SEM has 1 SE lower detector, 2 SE upper detectors with various modes of energy filtration, a five quadrant BSE detector, a STEM detector that works in bright field, an electron convertor that allows to use the SE lower detector for dark field STEM imaging, a 80 mm\textsuperscript{2} SDD EDS detector (Oxford Instrument) and the EBSD Nordlys II System (Oxford Instrument). The maximum probe current is 40 nA and this allows to perform quantitative x-ray microanalysis at low voltage as well as EBSD maps at a faster time, eliminating the problems of drift current issues and flashing.

In order to show the STEM capabilities of this FE-SEM, figure [1] and [2] compares bright field images of the same thin foil (thickness about 80nm) of a 2099 alloy in the T83 condition that was made with a Hitachi Focus Ion Beam NB-5000. Figure [1] was obtained with a Hitachi Field Emission Transmission Electron Microscope HD-2700 with a C\textsubscript{s} aberration corrector and figure [2] with the SU-8000. Clearly, the STEM at 30 keV allows to image the T1 precipitates as well as at 200 keV. Moreover, the contrast of the plates perpendicular to the beam are easily seen at 30 keV, owing to the lower beam energy. Figure [3] shows a bulk specimen of the Al 2099 alloy imaged at 5 keV with the SU-8000. The T1 precipitates are clearly visible as well as their localization with the grains. Figure [4] shows Monte Carlo simulations, using MC X-Ray [1], of the In $L_{\alpha}$ line as a function of beam position of a 50 nm thin film of GaN with layers of InGaN of 10, 5 and 1 nm separated by 10 and 1 nm at 200 keV and 30 keV with the probe diameter equals 0,1 nm. Clearly, high resolution x-ray microanalysis at 30 keV will be possible as soon as sub-nanometer probe diameter, with enough probe current, will become available.
References

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**Figure [1]** Thin film of an Al 2099 alloy imaged at 200 keV with a Hitachi HD-2700 FE-STEM aberration corrected. Bright field.

**Figure [2]** Same thin film of an Al 2099 alloy imaged at 30 keV with a Hitachi SU-8000 FE-SEM. Bright field.

**Figure [3]** Bulk specimen of the Al 2099 alloy imaged at 5 keV with a Hitachi SU-8000 FE-SEM. 5 quadrant BSE electron detector.

**Figure [4]** Monte Carlo simulations, using MC X-Ray, of the In L₆ line as a function of beam position of a 50 nm thin film of GaN with layers of InGaN of 10, 5 and 1 nm separated by 10 and 1 nm at 200 keV and 30 keV. The probe diameter equals 0.1 nm.