Microsc. Microanal. 8 (Suppl. 2), 2002
DOI 10.1017.S1431927602104004

Quantitative X-Ray Microanalysis with a Low Voltage Scanning Electron Microscope

Paula Horny, Raynald Gauvin, Eric Lifshin* and Di Wu*

Department of Mining, Metals and Materials Engineering, McGill University, Montreal, Canada H3A 2B2
* Albany Institute for Materials, University at Albany, Albany, New York, US

Nanometer scale objects raise more interest than ever in microelectronics and other applications. To characterize such samples, transmission electron microscope is an ideal tool, but the preparation of the required thin foil is a complex and costly operation. Low voltage scanning electron microscope seems to be a good deal to simplify the analysis of such samples, with simpler sample preparation and larger scale analysis. Low voltage electron beam gives better resolution because of the reduction of the incident electrons interaction volume. However the beam current is also decreased and signals become more sensitive to noise. A Monte Carlo program is being developped in order to compute X-ray spectrum with the goal to find optimal conditions for X-ray microanalysis, which are a compromise between resolution and count rates. In this paper, some results and future improvements are presented.

A field emission gun scanning electron microscope Hitachi S-4700 with an Oxford EDS system was used for this work. The studied material is Gallium Arsenide (GaAs). Figure 1 presents a graph of the drift in energy for highest energy emitted photons at different voltages. On that graph, $\frac{\Delta E}{E_0} = \frac{E_c - E_0}{E_0}$ where E_c is the energy of the most energetic photons and E_0 is the electron beam energy. This reveals the impact of charging, which is significant because of the GaAs semi-conductor character. Sample contamination is another problem because beam current and acquisition time must be increased at low voltage. Figures 2 and 3 show both measured and Monte Carlo simulated X-ray spectra of GaAs at 3 and 20 keV. The simulated bremstrahlung was normalized at 2 keV with the experimental ones. It can be observed on these two figures that the relative shapes of the bremstrahlung and characteristic spectra are rather good. However, the relative intensity of the simulated characteristic peaks does not fit exactly to the experimental values, which means that the X-ray cross sections need to be reviewed. Also, at 3 keV, a clear difference between the two spectra at low photon energy reveals that the bremstrahlung cross section has to be refined for low energy incident electrons. At high photon energy, the difference of shape may be related to charging, which is not yet included in these simulations. Very small details of GaAs surface can be seen with that microscope (figure 4) at small working distance (3.6 mm of WD). Even if microanalysis was performed on flatter surfaces than in figure 4, flatness of the sample surface should be verified at low WD before performing X-Ray microanalaysis at longer WD (12 mm).

The microanalysis of GaAs samples gives some challenge in order to get valuable spectra while struggling with charging, high contamination rates, rough surface and weak signals. A future improvement is the refinement of X-ray cross-sections at low electron beam energy. Simulations of lines scans show that it might be possible to detect very thin lines of AlGaAs, as quanta wells, in GaAs matrix at low voltage. Hence low voltage scanning electron microscopy is full of promises, and relevant simulations combined with state-of-the-art microscopy should allow low voltage quantitative microanalysis to become a reality.

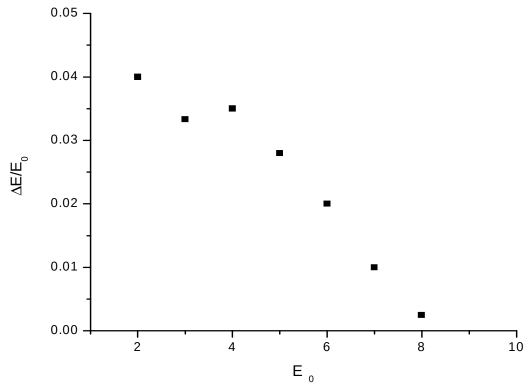

Fig. 1

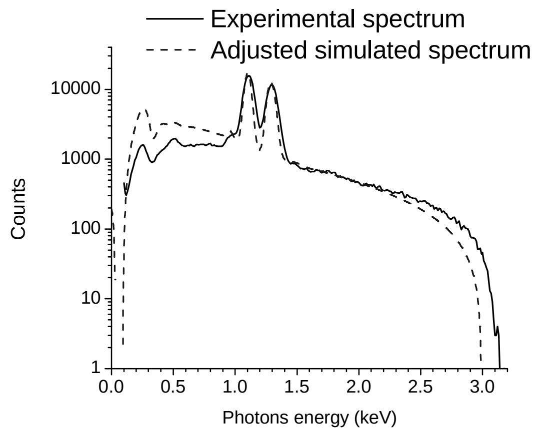

Fig. 2

FIG 1. Energy drift of highest energy generated photons related to electron beam energy.

FIG 2. Comparison of simulated and experimental spectra of bulk GaAs at 3 keV.

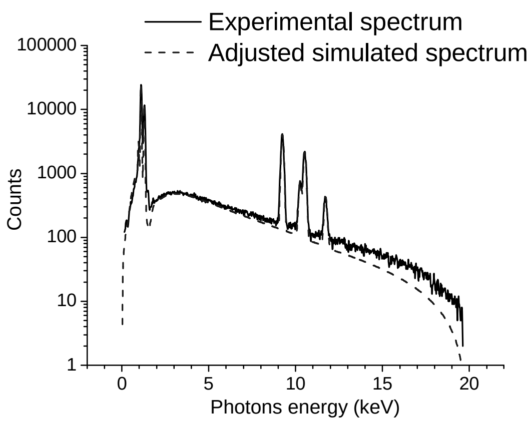

Fig. 3

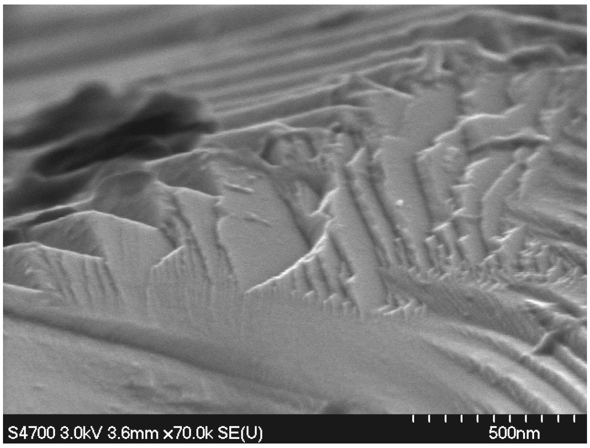

Fig. 4

FIG 3. Comparision of simulated and experimental spectra of bulk GaAs at 20 keV.

FIG 4. High resolution secondary image of the fracture surface of the cutted GaAs sample.

References

[1] P. Hovington, D. Drouin and R. Gauvin,*CASINO: A new Monte Carlo code in C language for the electron beam interactions - Part I: Description of the Program*, SCANNING, 19, 1-14 (1997).

[2] R. Gauvin and E. Lifshin, *Simulation of X-Ray Emission from Rough Surfaces*, Mikrochim. Acta 132, 201-204 (2000).