EDS Analysis with Silicon Drift Detectors at High Spatial Resolution

Advances in Low Energy X-ray Analysis

Continuing technological advances require the element analysis of increasingly smaller structures in many fields of materials science, including semiconductors and nanotechnology in general. The element composition of thin electron transparent samples can be analyzed in the nm-range using transmission electron microscopes (TEM) or, specific sample holders provided, in the field emission scanning electron microscope (FE-SEM). Nevertheless both methods often require complex sample preparation. An alternative method is to analyze bulk samples with a FE-SEM. In order to decrease the excitation volume for generated X-rays, low accelerating voltages are required. Consequently, only low energy X-ray lines can be evaluated. Specific examples of this approach are discussed below. They demonstrate that features in bulk structures at the sub-µm scale can be analyzed very quickly using silicon drift detector (SDD) technology and modern data processing.

Technological Advances

Silicon drift detectors (SDD) have become state of the art technology in the field of EDS microanalysis outperforming traditional Si(Li) detectors in almost every aspect. One of the main advantages of SDD technology is the extremely high efficiency, which means that, due to light element windows and fast pulse processing at low electronic noise, most of the incoming photons contribute to the available signal. Thus, low signal intensity, as a result of low accelerating voltage, can be analyzed. This advantage is combined with better energy resolution, also in the lower energy range.

In order to separate overlapping peaks (fig. 2), the line deconvolution algorithms incorporated in the analysis software are of crucial importance. The identification and quantification of low energy X-ray lines can be significantly improved by an extended atomic database. Bruker Nano’s database incorporates more than 300 additional L, M and N lines in the low energy range of 0–6 kV [1], allowing the deconvolution of peak overlaps using complete line series. Specifically adapted routines enhance the standardless quantification of low energy X-ray lines. Table 1 shows the quantification results of a (Ga,Al)N semiconductor sample. The spectra were acquired at 5 kV and the results show a deviation of less than 5–10%. Using reference standards, the deviation can be improved to 0.5–4.2%.

Furthermore, data processing with modern computers sets the stage for spectrum imaging techniques allowing advanced data mining. A hyperspectral EDS database contains a four dimensional data cube (position X and Y, spectrum channel and number of counts in each energy channel). Saving complete spectra for each pixel of the SEM image allows a range of advanced analysis options. For instance, the element identifi-
cation can be improved by using Maximum Pixel Spectrum [2]. This function synthesizes a spectrum consisting of the highest count level found in each spectrum channel. Even elements which occur in only a few pixels of an element map can be easily identified. For minor components, spectrum statistics can be improved using spectroscopic phase analysis, which detects similarly composed areas with the help of mathematical methods.

Examples

Figure 3 shows element maps of semiconductors which were acquired with a Bruker XFlash SDD. The first example (fig. 3a) demonstrates the high spatial EDS resolution that is possible when investigating an electron transparent sample in a SEM at 30 kV accelerating voltage. The second example (fig. 3b) is a bulk CMOS chip, which was analyzed using 4 kV. In the EDS map, which was overlaid with the SEM image, an 85 nm thick layer of an adhesion promoting agent composed of titanium and titanium nitride is clearly visible between the silicon dioxide substrate and the aluminum.

Another example for the importance of small features are CIGS solar cells, where the composition of the absorber material determines the conversion efficiency. The map of the sample in figure 3c was acquired within only three minutes at 7 kV. It displays a heterogeneous distribution of gallium and indium in the Cu(In,Ga)Se₂ absorber layer. The line scan of a second CIGS sample (fig. 4) was quantified using reference standards in order to obtain the highest possible accuracy where the L-lines overlap. The composition of ~50 at.% Se and ~25 at.% for In+Ga and Cu reflects the stoichiometry of the Cu(In,Ga)Se₂ absorber. The absorber layer displays a ~350 nm thick diffusion zone where it borders on molybdenum. This is represented by higher concentrations of molybdenum and selenium. In addition, an exchange of gallium with indium was found in a ~375 nm thick region.

<table>
<thead>
<tr>
<th>Phase</th>
<th>Elements</th>
<th>Area fraction/%</th>
<th>Area fraction/Pixel</th>
<th>Area fraction/µm²</th>
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<tr>
<td>Substrate</td>
<td>Ni, Cu</td>
<td>20.6</td>
<td>47455</td>
<td>30</td>
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<tr>
<td>Solder</td>
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<td>IMC1</td>
<td>Cu, Sn, Ni, Au</td>
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<tr>
<td>IMC2</td>
<td>Cu, Sn, Ni</td>
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<td>25</td>
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<tr>
<td>IMC3</td>
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<td>4117</td>
<td>3</td>
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<tr>
<td>IMC4</td>
<td>Ni, P</td>
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<td>1891</td>
<td>1</td>
</tr>
</tbody>
</table>

Table 1: Expected, stoichiometric values are compared to mean values obtained by quantification with automated background subtraction and an XPP optimized PhiRhoZ model.

(Ga,Al)N semiconductor: 7 kV, 6.5 kcps, 20 s, n=4, reference standards: AlN, GaN, NiP on a continuous IMC layer ~250 nm thick at a solder joint: 6 kV, ~21 kcps, 10 s, n=3.

Table 2: Area fraction and detected elements of solder, substrate and IMC phases.
The last example in figure 5a shows a solder joint connecting components for microelectronic packaging with a tin solder on a nickel and copper substrate. The formation of intermetallic compounds (IMC) at the solder joint plays a major role for its mechanical stability. The microstructural characterization of IMCs is increasingly important mainly because alternative lead-free solder systems are required by international regulations (e.g. RoHS). The Maximum Pixel Spectrum reveals the presence of gold, silver and phosphor (fig. 5b). The spectroscopic phase analysis (fig. 5c, table 2) shows that these elements are enriched in four different IMCs, including nickel phosphide, <100–500 nm in size. The evaluation of the phase spectrum including standardless quantification of this compound at other solder joints (table 1) indicates a stoichiometry of Ni₃P.

Summary

The discussed element analysis using EDS in SEM at high spatial resolution shows that the use of low energy X-ray lines for qualitative and quantitative composition determination has become possible. This is a result of technological developments in silicon drift detectors, research concerning atomic data and modern data processing. The analysis of features at the sub-µm scale using SEM-EDS can provide new insights for various applications in a short time without complex sample preparation.

References

[3] Ferdinand-Braun-Institut Berlin and Institut für Mikroelektronik Stuttgart are acknowledged for providing samples.

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