DETECTION OF Be USING EDS IN SEM

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Energy dispersive spectroscopy (EDS) is the most frequent microanalysis tool used in scanning electron microscopes (SEM). At this technique the energy of X-rays arising from the sample excited by electrons is measured. Due to the nature of the detection and measuring processes, peaks are much wider than those formed at the complement wave-length dispersive spectroscopy (WDS). Consequently, it is more difficult to distinguish elements with overlapping peaks and detect elements present in low-concentrations. Nevertheless, EDS-analysis is quite fast and the equipment is much cheaper than for WDS.

EDS has been used for several decades. At the beginning it was possible to detect only elements with atomic numbers greater than eleven (elements from Na) because of the application of Be-window in Si(Li)-detectors which effectively absorbed all low-energy X-rays. Development of thin polymeric windows and overall improvement of the system resolution below approximately 135 eV (for Mn Kα radiation) allows detection and qualitative analysis of all elements ranging from Be to U. At the moment, the quantitative evaluation is possible for elements from B to U, but not for Be.

With decreasing atomic number (Z) the energy of X-rays lowers dramatically, reaching the energy of only 0.108 eV for Be K-radiation. With this small energy, interaction volume where X-rays are generated in fact increases, but the attenuation length becomes so small, that almost all X-rays apart from those arising from the shallow surface region, become absorbed. Therefore, the Be-peak will be strongly influenced by the concentration, surface condition and operating conditions (accelerating voltage, count rate,...)

In our investigation we studied the influence of Be-content, particle size, surface conditions and operating conditions on detectability of beryllium. We used different samples: standard Be-wire from SPI Supplies, binary Al-Be alloys with 5.5 % mass fraction of Be, Al-Mn-Be alloys with Be₄AlMn and quasicrystalline phases. The study was carried out in Sirion NC from FEI (FEG SEM) equipped with INCA 350 system from Oxford Analytical Ltd (detector resolution for Mn Kα: 129 eV). SEM’s field emission source allowed EDS analysis with as low voltages as 1 kV.

The results clearly showed that pure Be (in alloy Al-Be) can be easily determined with accelerating voltages between 3 kV and 15 kV. The same was also true for standard Be-wire which was even carbon coated. The highest ratio between the height of beryllium and the height of carbon peaks was attained at ~10 kV (Fig. 1). With accelerating voltages of 1 kV and 2 kV the interaction volume lied almost completely in the carbon coating thus no beryllium was detected. Be-peak was clearly found in Be₄AlMn phase (Fig. 2) with small acceleration voltages of 3 kV and 5 kV with small count rates (approximately 5-10 % dead time, count rates ~200 s⁻¹). It was found useful to focus the electron beam on a small area for some period of time. This procedure removed many of adsorbed carbon from the polished surface and resulted in a more pronounced Be-peak. On the other hand, short application of FIB resulted in Ga-implantation in the surface region. As a result Be-peak disappeared.

In quasicrystalline particles which contained ~40 at. % Be (according to Auger electron spectroscopy AES) we have not succeeded to detect Be yet. Therefore, it seems that with the current EDS detectors the detection limit for Be lies between 40 and 66 at. % Be. This limit is further shifted to higher Be-contents with surface contamination and coatings, small particle size
(which can be made less critical with the use of low accelerating voltages in today’s high-resolution microscopes) and not optimal operating conditions. According to our experience, it is much more easily to miss than to confirm Be in Be-containing phases using EDS.

Figure 1: EDS-spectra taken on Be-wire standard sample from SPI Supplies. At 10 kV Be peak can be clearly seen, but at 1 kV it is not visible.

Figure 2: a) Microstructure of alloy Al-Mn-Be with 5 wt. % Mn and 2.5 wt. % Be. Bright phase: Al_{10}Mn_{3}, grey phase: Be_{4}AlMn, dark matrix: Al-rich solid solution. b) EDS-spectrum of Be_{4}AlMn. Be peak is very small, but clearly visible. Conditions: 3 kV, 6 % dead time.