Fast Elemental Mapping in Materials Science

M. Procop¹, W. Hübner¹, R. Wäsche¹, S. Nieland², O. Ehrmann²

¹Bundesanstalt für Materialforschung und -prüfung, ²Fraunhofer Institut Zuverlässigkeit und Mikrointegration, Berlin, Germany

INTRODUCTION

The scanning electron beam in an electron microscope causes the secondary electron (SE) image of the specimen surface and also a microscopic image of the elemental distribution near the surface, if the microscope is equipped with an X-ray spectrometer, to detect the characteristic X-rays emitted from the specimen. This kind of analysis is known as elemental mapping. The SE image and map are different regarding two essential subjects. The first is the resolution, which for the SE image is in the order of the electron beam diameter, i.e. some nanometres. The resolution of the map, however, is determined by the diameter of the X-ray excitation volume, i.e. about 1 µm. The second difference that is important in the following is the time to record an image with the same pixel number and comparable noise figures as the SE image. The SE yield is roughly one, but the probability per primary electron for the emission of an X-ray photon into the solid angle of the detector ranges around 10⁻⁴. Therefore, we have the 1MB pixels SE image within seconds, whereas the accumulation time for a 256 x 256 pixel map amounts often up to a half hour.

MATERIALS AND METHODS

The only way for fast elemental mapping by X-ray spectrometry is a higher count rate. This can be achieved by operation at increased beam currents. Modern spectrometers with digital pulse processing detect more than 10⁴ pulses per second at dead times as low as 10%.

High computer speeds enable the storage of the complete spectrum at each pixel for later evaluation. It was originally introduced as position tagged spectroscopy by PGT [1], and is now offered also by other manufacturers. A relatively quick impression from a specimen inhomogeneity can be gained by a colour coding of X-ray energies. "Cameo" from Oxford Instruments applies this method [2]. Röntec developed an extremely fast spectrometer. Key features are a silicon drift detector [3] and a special analogue pulse processor able to process up to 10⁴ pulses per second.

As outlined above, high processor speed can be used advantageously only when the specimen is exposed to a high beam current. In our laboratory we studied specimens from several fields of materials research to explore the application for such a fast spectrometer.

RESULTS

One of the first specimens was a Cu/TiW double layer on a silicon wafer. The Cu layer was structured to have 30 µm dots in a matrix array. A map was recorded at 20 keV and 15 nA. The acquisition time for a single 512 x 384 pixel frame amounted to 1 min. After that time the dots were visible not only in the Cu-Kα map as bright spots, but also as dark spots in the less intensive W-Lα and Ti-Kα maps. After smoothing of raw data the map should be ready for every laboratory report (Fig 1).

The next example belongs to wear research. Figure 2 shows the wear scar of a corundum ball on a SiC disk specimen after an oscillating sliding experiment at 200°C. These investigations were carried out in order to screen the tribological properties of ceramic composite tribomaterials in the system SiC-TiC-TiB₂ running against alumina. For a quick evaluation of the amount of Al (in form of Al₂O₃) worn on the silicon carbide surface we recorded 256 x 192 pixel images within 45 seconds each. The microscope parameters were as above. The Al map gives a much better impression from the Al distribution than the SE image. Moreover, because the rough surface shadows the X-rays on their path to the detector, the picture appears more vivid. Clearly, the short acquisition time enables a user to inspect more specimens within a limited time.
Figure 3 gives an example how by means of elemental mapping the TiN and alumina inclusions in a CrNi-steel can be made quickly visible. The acquisition time amounted to 3 min at 20 keV beam voltage and 5 nA beam current. The raw data were smoothed and converted into binary maps for the presentation.

At high magnifications image drift due to specimen charging at such high beam currents may become a problem. We studied cross sections from silicon wafer pieces embedded in resin. The charging problem could be solved by contacting the wafer directly by means of silver paste lines. The beam current had to be reduced to below 10 nA, resulting in a total count rate of some $10^4$ counts/s.

Figure 4 gives the elemental maps for a eutectic PbSn$_{63}$ solder bump on top of a Ni(P) under bump metallisation (UBM). The map was completed within 9 min. A metallisation scheme like that in Fig 4 is applied in flip-chip soldering. This technique has gained a high industrial interest because of its potential for cost reduction of electronic devices. Solder bumps can be applied for example by stencil printing of solder paste. For standard applications like consumer electronics a eutectic lead-tin solder is used. In order to investigate the reliability of such material combinations fast mapping method can be useful to detect the diffusion of UBM components into the solder and to observe intermetallic phase formation.

We studied a lot of specimens using the fast XFlash [4] spectrometer equipped with the new silicon drift detector. The maps were finished in a few minutes for beam currents ranging between 5 and 10 nA. The short acquisition time is a clear advantage because more specimens can be investigated in one session. Image drift due to specimen charging at high beam currents could be avoided in most cases by proper contacting of the specimen. We tried to improve the lateral resolution of the map by reduction of the beam voltage. However, for our conventional SEM equipped with a tungsten cathode either the beam current was too low to achieve count rates in the order of $10^4$ and above, or the SE image resolution was too poor at low lens currents. Ideal conditions should be achieved in connection with SEM operating with a thermionic field emitter.

**CONCLUSIONS**

The experiences gained so far with this kind of fast X-ray spectrometer demonstrate a wide field of application where large specimen quantities need to be characterized by their inhomogeneous elemental distribution at low or modest magnifications. Some improvements of the spectrometer would be desirable with its further development. An increased detector area would allow high count rates at lower beam currents. The detector area amounted to about 5 mm$^2$ in this study. Larger areas are now available. Replacement of the beryllium window by a thin film window would extend the detected spectral range toward low energies and would enable maps at low beam voltage for optimum lateral resolution.

**REFERENCES**

4. XFlash ® is a registered trademark of Röntec GmbH.