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LARGE FIELD X-RAY ABSORPTION MICRO-ANALYSIS NEAR AN ABSORPTION EDGE BY IMAGE PROCESSING

E. Bigler and F. Polack*

Institut d'Optique, B.P. 43, 91406 Orsay Cedex, France
*L.U.R.E., Université Paris-Sud, Bât. 209 C, 91405 Orsay Cedex, France

Abstract - Chemical microanalysis is performed by contact micro-radiography with a tunable synchrotron X-ray beam. Large field (3 cm²) quantitative charts of the elements are obtained by selective absorption near an edge. We expose here present performances and future developments of the method: field and spatial resolution, sensitivity and corrective factors.

Microanalytical study of objects with complex structures requires both a large field and a high spatial resolution. In this paper, we examine how these two opposite qualities can be obtained by X-ray differential absorption near an edge and contact imaging.

I - PRINCIPLES

1.1. Chemical analysis by X-ray absorption near an edge \[1, 2\]

The transmittance of a sample is measured with a monochromatic X-ray illumination. The beam is successively tuned on each side of an absorption edge of a given element. The concentration \(C\) of the analyzed element is given by:

\[
C = \frac{1}{m_0 \Delta(\mu/\rho)} \ln(N_2/N_1)
\]

where \((\mu/\rho)\) is the mass absorption edge-jump \((\text{cm}^2/\text{g})\) of the analyzed element. \(m_0\) is the mass per unit area of the whole sample. \(N_1\) and \(N_2\) are the number of transmitted photons on each side of the absorption edge. The result can also be expressed as a mass per unit area

\[
m = \frac{1}{\Delta(\mu/\rho)} \ln(N_2/N_1)
\]

1.2. Experimental set-up

The X-ray source is the synchrotron beam delivered by the storage ring D.C.I. in Orsay. A tunable monochromatic beam \((1 - 3 \text{ Å})\) is delivered by a channel-cut monochromator \((S_4: 220)\). Two contact microradiographs of the sample (one of each side of an absorption edge) are recorded on a high resolution photographic plate. When the concentration of the analyzed element is greater than \(1 \times 10^3\), a simple visual comparison of the two images shows the localisation zones of the element \([3]\).
The field covered is limited only by the section of the synchrotron beam, i.e 3 cm².

II - IMAGES PROCESSING : EXAMPLE OF RESULTS

2.1. Microdensitometer analysis
An area of interest is visually selected on enlarged prints. The original plates are scanned with a microdensitometer. The analytical signal, given by Eq. (1), is obtained by comparison of optical densities of the two contact images.
A calibration scale, recorded on each plate, gives the sensitometric data. The curve plotting optical densities vs number of received photons is linear for optical densities < 1.5. A simple analytical model of the response curve allows a plate calibration up to densities of 2.5. It must be pointed out that we only need a relative calibration, which is much more simple. For example, inequalities of exposure between homologous images can be corrected a posteriori.
A careful registration of images pairs is necessary to ensure that the number of photons $N_1$ and $N_2$ in Eq. 1 correspond exactly to the same pixel of the sample. This problem is solved during the recording of the second image by displaying the image of local differences. The best registration is obtained when differentiation artefacts disappear near the sharp lines of the image.

2.2. Computer processing
The mass per unit area of the analyzed element is computed for each point of the image (Eq. (2)). The result is a numeric array which is displayed as an image in grey levels. Figure 1 shows 3 analytical charts of Mn, Fe and Ni in a 30 µm-thick polymetallic nodule section. White areas correspond to higher concentrations of the analyzed element. Large field correlations between the concentrations zones of the elements appear clearly on these pictures. It would be rather difficult to obtain such images with a microprobe: a large field method is highly interesting to study objects with complex structures like polymetallic nodules.

![Figure 1](image_url)

2.3. Quantitative output : discussion
The sample shown on Figure 1 has also been analyzed with a S.E.M.
The concentration of Mn, Fe and Ni have been measured in their zone of predominance. The sample is known to have an average density of 4: comparison can be made in concentration terms.

<table>
<thead>
<tr>
<th>Element</th>
<th>X-ray absorption</th>
<th>SEM-fluorescence</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mn</td>
<td>28 %</td>
<td>35 %</td>
</tr>
<tr>
<td>Fe</td>
<td>10 %</td>
<td>16 %</td>
</tr>
<tr>
<td>Ni</td>
<td>3 %</td>
<td>4 %</td>
</tr>
</tbody>
</table>

The systematic under evaluation of our results can be explained by different factors:
a non selective detector like the photographic plate records also a fluorescent signal which is additive to the absorption signal. Analysis shows that the contrast between the two images is reduced.

the channel-cut monochromator tuned on wavelength $\lambda$ also delivers the harmonics $\lambda/2, \lambda/3 \ldots$. These high energy components of the beam are not affected by the presence of the absorption edge. Thus, another additive signal is recorded on each image. This phenomenon yields another reduction of the differences between the two images, mostly with thick samples. Let us finally mention that the presence of monocrystals in the sample can bring topographic effects in the contact images. This has never been observed with the nodule samples (Fig. 1.) which are amorphous.

III - PRESENT PREFORMANCES AND IMPROVEMENTS

3.1. Spatial resolution

High resolution plates yield 1 $\mu$m resolution, but the final pixel size is determined by the smallest available microdensitometer slit, i.e. 5 $\times$ 5 $\mu$m$^2$. It must be emphasized that the photographic plate remains one of the only X-ray images detector with good spatial resolution and quantitative output. With the new photoelectron soft X-ray microscope (4), we hope to get a $\lambda$ $\mu$m resolution and quantitives images.

3.2. Sensitivity

Sensitivity is mainly determined by the number of available photons during an absorption measurement. For example, in the case of iron analysis near the K edge in a 30 $\mu$m thick sample (average density = 4), the minimum detectable concentration in a 20 $\times$ 20 $\mu$m$^2$ cell is $C_{\text{min}} = 3 \times 10^{-4}$ with a 4 min synchrotron exposure and an ideal detector. Experimentally, the minimum concentration is found to be $= 1\%$ in the same conditions, with a photographic detector.

To improve sensitivity, we are developing a non saturating scintillation image detector. Our aim is to get 10 $\mu$m resolution with counting output.

CONCLUSION

New synchrotron sources such as undulators will deliver 10 to 100 times more photons than the first synchrotron sources. These very intense beams will offer the possibility of X-ray imaging microanalysis with very short exposures. One must chose between two complementary tools: X-ray absorption, and fluorescence. According to J. KIRZ (5), X-ray absorption is better in the soft X-ray region while fluorescence is more sensitive in the 1 - 3 Å region.

X-ray absorption with contact imaging offers high quality images; on the other hand fluorescence analysis can be more sensitive (6), but image reconstruction needs a scanning microscope. At present, there is no available microscope of this kind with a few $\mu$m resolution (apart from a pioneer work of HOROWITZ and HOWELL (7)).

All the projects of scanning X-ray microscopes include an X-ray focusing optics, and therefore will be limited to the soft X-ray region. In the hard X-ray domain, contact imaging remains the simplest method.

We have shown that quantitative analytical images can be obtained with a photographic detection. We think that the next years will bring important improvements of X-ray images detectors and therefore X-ray absorption microanalysis will be more efficient.

REFERENCES

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{4} POLACK F. This conference