

COMBINED DIFFRACTION AND REFRACTION IMAGING STUDY OF A ROMAN KNIFE

W. Paszkowicz¹, T. Wroblewski², and W. Weker³

¹*Institute of Physics, Polish Academy of Sciences, al. Lotników 32/46, 02-668 Warsaw, Poland*

²*Hasylab/DESY, Notkestrasse 85, Hamburg, Germany*

³*State Archaeological Museum, 52 Długa str., 00-950 Warsaw, Poland*

Keywords: MAXIM, Diffraction Enhanced Imaging (DEI), microdiffraction, archaeometry, iron, corrosion

e-mail: paszk@ifpan.edu.pl

Contemporary physics has developed various techniques applicable to solving difficult tasks in archaeometry. Many of them can determine specific material properties in a nondestructive and non-invasive way, and can operate with spatial resolution down to micrometer scale. Such techniques involve ion-beam based analytical techniques, *e.g.* Particle Induced X-Ray Emission (PIXE), Rutherford Backscattering Spectrometry (RBS), Nuclear Reaction Analysis (NRA), Elastic Recoil Detection Analysis (ERDA) [1], Electron Spin Resonance (ESR) [2] with attractive applications, for example determining the nature of ancient gold objects [3,4]. X-ray spectroscopic, fluorescence and diffraction methods are of particular importance, due, in particular to fast development of synchrotron-based techniques including various X-ray microtechniques (see *e.g.* [5-8]). For their success it is crucial that some of X-ray methods can be combined at a single beamline, enriching and facilitating the interpretation of measurement results. One of such combined methods is the *Diffraction Enhanced Imaging (DEI)*

employing not only the classical absorption contrast, but also the contrasts due to refraction, extinction and diffraction [8-14]. The synchrotron imaging methods can be used, in particular, for studies of archaeological objects and therefore, they can complete the traditional radiography commonly used in archaeometry. They can reveal, in a non destructive way, both, the surface and in-depth details of the object structure. The depth at which the data are collected by the given method depends on the experimental conditions (incident angle, wavelength value or range).

In the present study, a small part of a Roman iron knife, excavated, together with many other ancient objects, about year 1905 at an (inexistent now) archaeological site in Sierzchów (Łowicz district, central Poland) was an object of the present study. The studied remains of the knife (see Fig. 1) are solid but almost entirely corroded, composed from Fe-containing oxides.

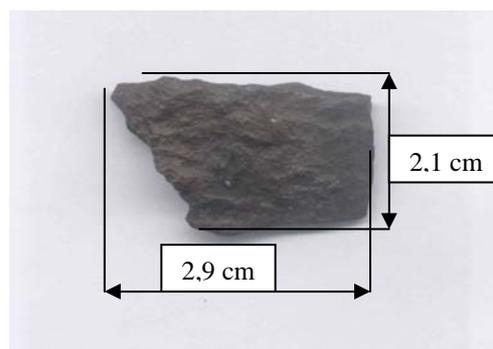
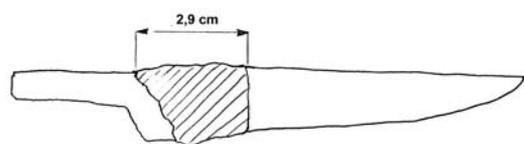


Figure 1. Probable shape of the original knife (upper left). Photographs of the studied part of the knife (upper right), fracture at the end 1 (lower left), fracture at the end 2 (lower right).

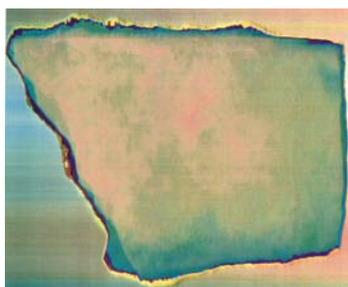


Figure 2. Image of the knife side based on the refraction contrast.

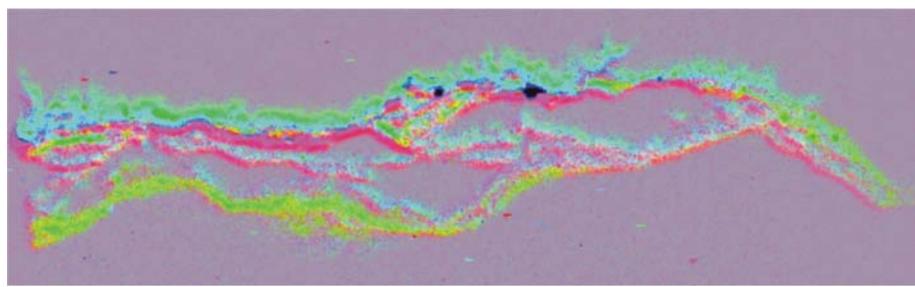


Figure 3. Image of the fracture end 1 surface of the knife determined by the MAXIM method; different colours refer to intensity distribution of peaks characteristic to component phases. Dark dots are attributed to diffracting single crystal grains.

The study was performed at HASYLAB beamline G3. Images were collected employing the effect of refraction and using the MAXIM method (mapping of the diffraction effect of constituent phases at selected diffraction angles) [1]. For the MAXIM method, the X-ray energy was 7 keV chosen to avoid iron fluorescence.

Figure 2 shows the knife-side image obtained by the DEI technique. This kind of radiography applies an analyzer crystal between sample and detector. This crystal either suppresses radiation refracted by the sample, if it is oriented parallel to the monochromator crystal, or enhances the refracted part, if it is adjusted to the wing of the reflection curve of the monochromator. The image presented constitutes a false colour composite of three Eigenimages calculated (using a principal component transformation) from exposures taken at the reflection wing at the intensity levels 15%, 50% and an intermediate one. The knife surface is found to be relatively uniform along the knife side (except the edges) showing that the surface-oxide layer is homogeneous.

A wide-range diffraction scan revealed a complex diffraction picture showing that one of components in this Fe-rich sample is likely to be hematite. It is accompanied by a small fraction of quartz attached because of the thousands-year contact with soil. Identification of other phases, presumably Fe-containing oxides, requires further work. Distribution of component phases was observed at the end-2 fracture (Fig. 3) by the MAXIM method. This image is a false colour composite of three Eigenimages clearly showing regions of different structural properties. The diagrams were taken from different regions (green, magenta and violet). The high intensity in the latter (dark spots) is probably due to large single crystal quartz grains. Further work is required to identify the phases connected to the diffraction effects observed. The methods applied seem to be promising, in particular, for investigation of partially or fully corroded archaeological objects.

Acknowledgements: This work was partially supported by the European Community - Research Infrastructure Action under the FP6 "Structuring the European Research Area"

Programme (through the Integrated Infrastructure Initiative "Integrating Activity on Synchrotron and Free Electron Laser Science".) The support by M. Lohmann and J. Metge with the DEI measurements is gratefully acknowledged.

References

- [1] T. Calligaro, J.C. Dran, J. Salomon, P. Walter, *Nucl. Instrum. Meth. Phys. Res. B* **226** (2004) 29-37.
- [2] W.J. Rink, *Radiat. Meas.* **27** (1997) 975-1025.
- [3] M.A.O. Salamanca, B.G. Tubio, M.L. de la Bandera, M.A. Respaldiza, *Nucl. Instrum. Meth. Phys. Res. B* **226** (2004) 199-207.
- [4] M.F. Guerra, T. Calligaro, *J. Archaeol. Sci.* **31** (2004) 1199-1208.
- [5] P. Dillmann, P. Populus, P. Chevallier, P. Fluzin, G. Béranger, A. Firsov, *J. Trace Microprobe Techn.* **15** (1997) 251-262.
- [6] K. Janssens, G. Vittiglio, I. Deraedt, A. Aerts, B. Vekemans, L. Vincze, F. Wei, I. Deryck, O. Schalm, F. Adams, A. Rindby, A. Knochel, A. Simionovici, A. Snigirev, *X-Ray Spectrom.* **29** (2000) 73-91.
- [7] D. Grolimund, M. Senn, M. Trottmann, M. Janousch, I. Bonhoure, A.M. Scheidegger, M. Marcus, *Spectrochim. Acta B* **59** (2004) 1627-1635.
- [8] M. Schreiner, B. Fruhmann, D. Jembrih-Simburger, R. Linke, *Powder Diffrac.* **19** (2004) 3-11.
- [8] T. Wroblewski, *Mater. Sci. Forum* **404-407** (2002) 121-126.
- [9] T. Wroblewski, *Radiat. Phys. Chem.* **61** (2001) 329-332.
- [10] K. Hirano, A. Maksimenko, H. Sugiyama, M. Ando, *Jpn J. Appl. Phys.* **41** (5B) (2002) L595-L598.
- [11] F.A. Dilmanian, Z. Zhong, B. Ren, X.Y. Wu, L.D. Chapman, I. Orion, W.C. Thomlinson, *Phys. Medic. Biol.* **45** (2000) 933-946.
- [12] Z. Zhong, W. Thomlinson, D. Chapman, D. Sayers, *Nucl. Instrum. Meth. Phys. Res. A* **450** (2000) 556-567.
- [13] A. Cedola, S. Lagomarsino, V. Komlev, F. Rustichelli, M. Mastrogiacomo, R. Cancedda, S. Milita, M. Burghammer, *Spectrochim. Acta B* **59** (2004) 1557-1564.
- [14] T. Wroblewski, A. Bjeoumikhov, *Nucl. Instrum. Meth. Phys. Res. A* **538** (2005) 771-777.