ELEMENTAL MAPPING AND MICRO-IMAGING BY X-RAY CAPILLARY OPTICS

Dariush Hampai,1,2,3,* Sultan B. Dabagov,2,4 Giorgio Cappuccio,2
Antonio Longoni,5 Tommaso Frizzi,5 Giannantonio Cibin,6
Valeria Guglielmotti3 and M. Sala7

1)INOA-CNR, L.go E. Fermi, 6, I-50125 Firenze, Italy
2)INFN-LNF, Via E. Fermi, 44, I-00040 Frascati (RM), Italy
3)MINASlab, Dipartimento di Scienze e Tecnologie Chimiche,
   Università di Roma “Tor Vergata”
4)RAS - P.N. Lebedev Physical Institute, Leninsky pr. 53, 119991 Moscow, Russia
5)Politecnico di Milano, Dipartimento di Elettronica e Informazione,
   Via Golgi, 40, 20133, Milan, Italy
6)Diamond Light Source Ltd., Chilton, Didcot OX11 ODE, UK
7)Dip. di Scienze della Terra A. Desio, Univ. di Milano, Italy
*Corresponding author: dariush.hampai@lnf.infn.it

Abstract

Recently, many experiments have highlighted the advantage of using polycapillary optics for x-ray fluorescence studies. We have developed a special confocal scheme for micro x-ray fluorescence (µXRF) measurements, which enables us to obtain not only elemental mapping of the sample but also simultaneously its own x-ray imaging. We have designed the prototype of a compact x-ray spectrometer characterized by a spatial resolution of less than 100 µm for fluorescence and less than 10 µm for imaging. A couple of polycapillary lenses in a confocal configuration together with a silicon drift detector (SDD) allow elemental studies of extended samples (∼ 3 mm) to be performed while, a charge-coupled device (CCD) camera makes it possible to record an image of the same samples with a 6 µm spatial resolution, which is limited only by the pixel size of the camera. By inserting a compound refractive lens (CRL) between the sample and the CCD camera, we hope to develop an x-ray microscope for more enlarged images of the samples under test.

OCIS codes 340.7440, 340.7460, 180.2520,170.1790,180.1790, 040.6040

Accepted to Optics Letter at 10th of October 2008
1 Introduction

Nondestructive analysis of archaeological artifacts by micro x-ray fluorescence spectrometry (µXRF) is a promising multi-elemental technique that has rapidly developed during the last few years [1,2]. With the exception of synchrotron radiation, which is a suitable probe for micro spots, it is often challenging to perform table-top µXRF analysis. However, the difficulty of producing and focusing small-size x-ray beams can limit the feasibility of this type of analysis.

The polycapillary lenses designed for x-ray beam focusing now offer alternative laboratory solutions[3–7]. The combination of a polycapillary lens and a fine focus x-ray tube (with a source spot diameter less than 50 µm) can provide the high-intensity radiation flux necessary to perform elemental analysis. Compared to a pinhole or to a monocapillary [8], an optimized “x-ray source-optics system” can result in a radiation density gain of more than three orders of magnitude (by radiation density gain we mean the ratio of x-ray intensity at the focal spot with and without the optics) [3]. In such a way, due to the rather high-intensity irradiation on a limited space, non-destructive µXRF analysis becomes possible. The most advanced way to get this result is to use a confocal configuration with two x-ray polycapillary lenses. One lens is responsible for the fluorescence excitation on the sample; the other allows detection of the secondary emission from the sample (see for instance [4]). With a µXRF instrument based on x-ray micro-focusing by two polycapillary lenses, 3D elemental mapping can be obtained [9]. Our final aim is to design a portable x-ray microscope to provide simultaneously on-site µXRF mapping analysis. To focus the x-ray beam we are actually using polycapillary optics, but in the near future we plan to use a polycapillary lens as a concentrator and a compound refractive lens (CRL) for the magnification [10]. Since this kind of spectroscopy is non-destructive, we believe it could be useful in the analysis and interpretation of archaeological artefacts and remains.

2 Experimental Setup and First Results

An experimental setup with a cabinet and an optical table was designed and developed specifically for R&D of x-ray optical systems at the Laboratori Nazionali di Frascati (LNF) [11]. The radiation source is a 50 W Cu x-ray tube (Oxford Apogee 5000) with a source spot size of about 50 µm. There are four different detector units available: a scintillator with an effective working area of ~ 1 square in.; two Photonic Science charged-coupled device (CCD) cameras with the software package “Image Pro” for x-ray rough/fine imaging; a silicon drift detector (SDD) with a 5 mm² working area[12,13]. The
first ccd camera (FDI 1.61:1) has a sensitive area of 4x3 mm with a 3.5x3.5 µm resolution; the second (FDI 1:1.61), an active area of 14.4x10.8 mm with a resolution of 10.4x10.4 µm.

The first microscope designed recently at LNF [14,15] was made with an x-ray source, a CCD detector, and a semi-lens. By choosing a semi-lens we hoped to get a very small blurring effect because of the fairly small radiation divergence behind the optics (without taking into account diffraction effects on the sample edges, i.e., far from a wave zone, as well as multiple scattering radiation in matter). To evaluate the highest resolution available, we used a sample of standard mesh: Au 1000 with a hole width of 19 µm and bar width of 6 µm (Fig. 1).

![Image](image-url)

Figure 1: High resolution image of a gold mesh 1000 recorded by the detector (FDI 1.61:1) placed at a distance of ~ 44 cm from the sample.

As an additional option, a second polycapillary lens inserted between the sample and the detector made it possible to implement the μXRF unit with a prototype of an x-ray projection microscope. Polycapillary optics (Unisantis S.A.) with a full lens shape were used to focus the divergent beam from the x-ray Cu tube onto the sample. Another lens in “confocal geometry” allowed detection of the sample x-ray fluorescence with a nominal focal spot size of less than 100x100 µm. In recent years a lot of reported work has shown the advantages of this optical configuration to perform simultaneously micro-fluorescence mapping and imaging[4,6,5]. To obtain the confocal configuration we used two full lenses, chosen so as to have the smallest focal spot and the highest transmission. The first lens provides a focal spot of ~90 µm and 50% transmission; the second has a spot of ~100 µm and 42% transmission. The prototype scheme is shown in Fig. 2.

Fig. 3 reports our first μXRF spectrum collected in 60 s on a Neolithic human
bone sample in powder form where all main elements are well resolved with an excellent collected intensity and good signal to noise ratio (Cf. [16]).

A real mapping of $\mu$XRF spectroscopy was obtained on standard sample of ferric oxide ($\text{Fe}_2\text{O}_3$) [9]. The standard monophasic minerals of $\text{Fe}_2\text{O}_3$ was prepared in a 1000 class clean room at DISAT, University of Milano - Bicocca. So as not to overlap two close measurements, we made a scan, by a remote system, of 4x4 mm of the region with a step movement of 200x200 $\mu$m. Fig. 4 shows the image result: red represents iron (in intensity scale of gray); the white bright pixels are manganese (contamination is probably present so we are designing a glow box for performing measurements in a helium atmosphere).
3 Conclusions

We have described a new prototype for $\mu$XRF analysis. We have shown that a confocal optical scheme used with a low-power (50 W) conventional tube and high-resolution SDD detector becomes a powerful instrument for x-ray fluorescence measurements of micro-size spot. Polycapillary lenses in a “confocal configuration” deliver with a micro-size spot a high photon flux on a sample, so they are ideal candidates for overcoming some of the main problems of laboratory x-ray instruments, including signal-to-noise ratio, resolution, mapping, and portability.

We have shown the first results both for x-ray imaging and for XRF spectroscopy. With regard to imaging, the use of a polycapillary semi-lens helps us get a spatial resolution of about 6 $\mu$m. To decrease the x-ray spot dimensions, we would like to substitute the present polycapillary lenses with the latest generation lenses since they provide a focal spot of a few microns in diameter. To increase the x-ray microscopy resolution, we are planning to combine a full polycapillary lens with a compound refractive lens.

References


