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

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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 ($\sim$ 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.

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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 $\sim$ 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 mm^2 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).

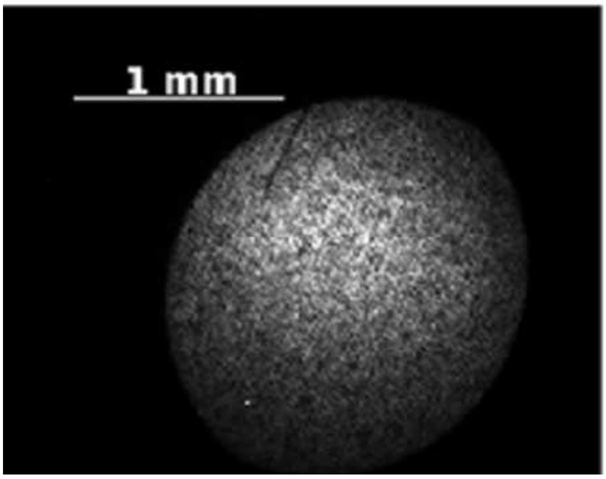

Figure 1: High resolution image of a gold mesh 1000 recorded by the detector (FDI 1.61:1) placed at a distance of $\sim$ 44 cm from the sample.

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 $\sim$90 μm and 50% transmission; the second has a spot of $\sim$ 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

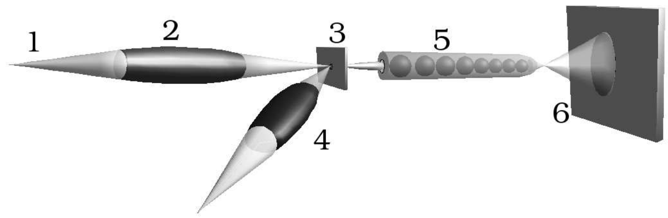

Figure 2: Final prototype layout for μXRF and imaging: 1 - the X-ray source; 2 and 4 - the 1st and 2nd polycapillary optical elements, respectively; 3 - the sample; 5 - a Compound Refractive Lens (CRL) for Imaging; 6 - CCD detector.

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]).

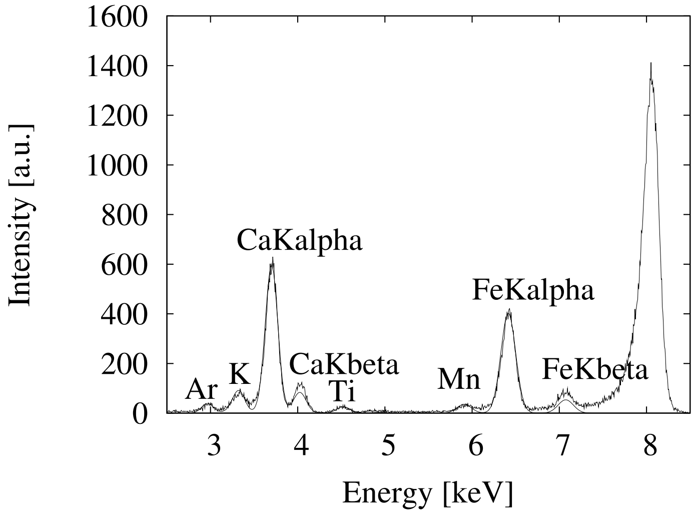

Figure 3: μXRF spectrum of a neolithic human bone powder sample obtained in confocal configuration.

A real mapping of μXRF spectroscopy was obtained on standard sample of ferric oxide (Fe_2O_3) [9]. The standard monophasic minerals of Fe_2O_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 μm. 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).

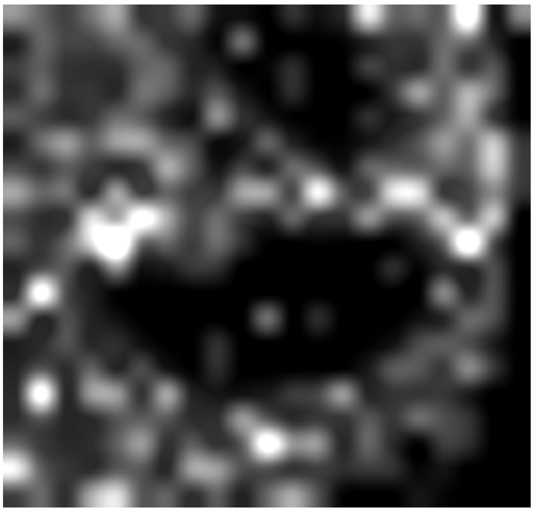

Figure 4: μXRF mapping spectrum Ferrum Oxide sample, deposited on a silicon wafer. The gray pixels represents iron (in a scale of gray), while the white bright spots are manganese trace. The measured area is 4x4 mm with a step sized of 200x200 μm.

3 Conclusions

We have described a new prototype for μXRF 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 μm. 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.

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