Nuclear Instruments and Methods in Physics Research B xxx (2015) xxx–xxx

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb

Proposal for a prototype of portable μXRF spectrometer

C. Polese [a,b,*], S.B. Dabagov [a,c,f], A. Esposito [a], A. Liedl [a,d], D. Hampai [a], C. Bartùli [b], M. Ferretti [e]

[a] *INFN – LNF, XLab Frascati – Via E. Fermi, 40, I-00044 Frascati, Italy*
[b] *Dip. DICMA, Univ. Roma "Sapienza", Via Eudossiana 18, Roma, Italy*
[c] *RAS P.N. Lebedev Physical Institute, Leninsky pr. 53, 119991 Moscow, Russia*
[d] *Dip. Scienze, Univ. Roma3, Largo San Leonardo Murialdo 1, Roma, Italy*
[e] *ITABC, CNR, Montelibretti, Roma, Italy*
[f] *National Research Nuclear University MEPhI, Kashirskoe shosse 31, 115409 Moscow, Russia*

ARTICLE INFO

Article history:
Received 23 December 2014
Received in revised form 10 February 2015
Accepted 12 February 2015
Available online xxxx

Keywords:
Micro-XRF
Polycapillary optics
Cultural heritage

ABSTRACT

μXRF is a powerful instrument for non-destructive characterization of materials of cultural interest. At the XLab Frascati Laboratory this technique is already well performed thanks to the polyCO set equipment allowing simultaneous μXRF 2D mapping. However, due to the strict demand for *in situ* analysis in this particular field, a new portable μXRF spectrometer equipped with a full polycapillary lens conjugated with a transmission anode X-ray tube is proposed.

Many cultural objects are characterized by elements (Ag, Sn, etc.) with high energy fluorescence K-lines. Thus, the capability of a full lens to deliver a high energy fraction of X-ray spectrum, in order to excite the fluorescence K-lines of such elements, is tested.

1. Introduction

X-ray fluorescence (XRF) spectrometry is widely used in *in situ* campaign for non-destructive characterization of materials of cultural interest [1]. Medium-Z elements (Ag, Sn, Sb, Ba etc.) are abundant in many different types of these materials such as paintings [2], glasses, and metals [3]. Their optimum detection involves the capability of exciting their fluorescence K-lines [4] by means of a high energy primary beam. Dealing with this kind of materials, the spatial resolution is also important because the details to be investigated can be very small [5] (Fig. 1).

Advances in X-ray optics have made micro-beams available. Among them, polycapillary lenses [6–11] are very efficient in delivering high flux beams thanks to their peculiar geometry and large acceptance solid angle. They consists of many hollow glass channels that work by total external reflection (TER), as a guide for X-ray photons, and are arranged in barrel (focusing full-lenses) or half-barrel shapes (collimating semi-lenses). Commercial polyCO (XOS, IFG, XCT, etc.) act as a sort of low pass filter, suppressing the high energy fraction (>20 keV) of the beam [12,13], due to the severe energy dependence for TER critical angle (θ_c)

$$\theta_c \approx (\hbar\omega_p)/E, \tag{1}$$

namely inverse proportional to the photon energy ($\theta_c < 1^\circ$ for $E > 2$ keV).

By theoretical calculation we can estimate a gain in the radiation density with respect to a pinhole focusing in the same size spot at the same distance according to the equation [6]:

$$G = \frac{\Omega_l^2}{\Omega_p^2} k_T, \tag{2}$$

where Ω_l is the solid angle captured by the lens at the input focal distance (IFD), Ω_p is the solid angle viewed in the spot obtained with the pinhole at distance L from the source, and k_T is the polyCO transmission efficiency.

In this paper we present the preliminary results in achieving relatively high energy micro-beams for portable systems. The rationale is to harden the input spectrum by means of filters and to use the central section of the lens, more efficient at high energies.

2. Experimental setup and preliminary results

The measurements were carried out at XLab Frascati [14], a LNF INFN facility [15] dedicated to the study and characterization of X-ray optics, with particular attention for polyCO lenses, and various X-ray analytical techniques [16,17], such as μXRF, μXRF 2D/3D imaging, X-ray micro-tomography (μ-CT) [18,19], total reflection X-ray fluorescence (TXRF) [20], and X-ray diffraction (XRD).

* Corresponding author at: INFN – LNF, XLab Frascati – Via E. Fermi, 40, I-00044 Frascati, Italy.

http://dx.doi.org/10.1016/j.nimb.2015.02.043
0168-583X/© 2015 Elsevier B.V. All rights reserved.

Fig. 1. Picture of a medieval enameled silver plate.

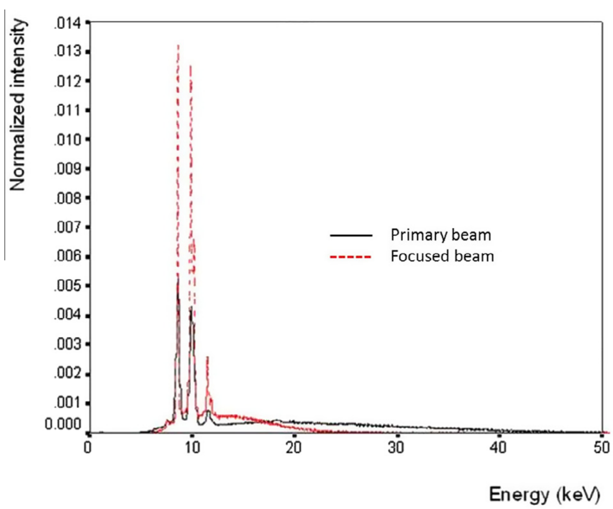

Fig. 2. Spectra of the primary beam and the focused one, normalized with respect to the total count rate.

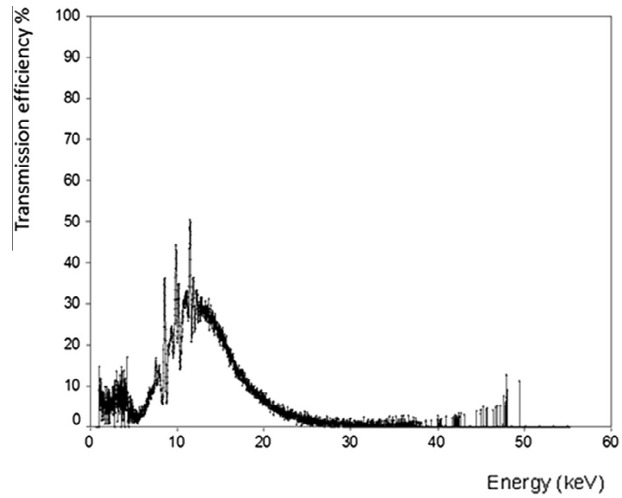

Fig. 3. Transmission of the polyCO.

Principal goal of these preliminary measurements was to characterize the primary beam focused by the optics and to evaluate its efficiency in exciting high energies fluorescence K-lines.

An Oxford Apogee W anode X-ray tube, set at 50 kV and 1 mA, was used.

The outcoming spectrum was recorded by placing a SDD detector (to be used also in the prototype: 10 mm^2 active area, 450 μm thickness, FWHM 175 eV @ 5.9 keV) at 39 cm from the tube focus. The spectrum was corrected for the detector energy efficiency, and the time acquisition was 120 s.

A filter made of a 70 μm thick sheet of stainless steel (Fe 74%, Cr 18%, Ni 8%) and a 60 μm thick sheet of copper was placed at the entrance of the lens, in order to enhance the high energy fraction of the spectrum.

The polycapillary full-lens parameters correspond to the input focal distance (IFD) of 56.5 mm, the output focal distance (OFD) of 44 mm, the length of 12 cm, the input diameter of 4 mm, and a 100 μm focal spot size. The nominal values of OFD and beam size have been verified by taking some CCD images at various distances from the lens.

The polyCO lens reduces the bremsstrahlung component at high energies and shifts its maximum to lower values (from ∼19 to ∼14 keV) (Fig. 2). The polyCO results to have a good efficiency in the energy range of 6–20 keV (Fig. 3) [21].

The spectra of focused beam with and without filter are shown in Fig. 4. It is evident that the filtering allows enhancing the high energy fraction of the primary beam.

To estimate the setup capability of exciting high energy fluorescence K-lines, some XRF spectra for a standard bronze alloy have been recorded. Fig. 5 shows a comparison between the XRF spectra recorded filtering the primary beam, using a pinhole, the above mentioned polyCO and another full-lens optimized for high energies (HE) and with similar geometrical parameters. The first lens results to be not efficient in the high energy region, but both the polyCO provide a better excitation in the low energy region with respect to the pinhole. Fig. 6 shows, for the HE lens, the improvement in the excitation given by the filter. In this case the intensities of the two XRF spectra have been normalized in order to be comparable, because the unfiltered spectrum has been recorded at 60 μA.

According to the experimental results, the first polyCO provides a gain of 3 orders of magnitude for Cu Kα and 1 over 10 keV, while the HE polyCO 4 orders for copper and even 3 up to 25 keV. It is evident that a lens with such parameters as the HE one could be suitable for the realization of a portable μXRF spectrometer able in the detection of high energy K-lines.

3. Prototype proposal

To further improve the high flux beam focused by a polyCO, a transmission anode X-ray tube [11] could be used. It is characterized by a strongly reduced focus-window distance, and this property combined with the use of a fifth generation micro-lens, optimized for the transmission of high energy photons and with low IFD (<25 mm), would lead to capture a larger acceptance angle.

To realize such a prototype, we propose a Moxtek transmission W anode X-ray tube: 50 kV, 200 μA, focal spot size 400 μm, 0.25 mm thick Be window, 92° emission solid angle, and a total weight of 950 g (important for the portability). The target is recessed of ∼2.29 mm from the front end, but with the lens of an input diameter smaller than the external Al collimator diameter (4.5 mm) it is possible to get closer to the target.

As an alternative, also a polyCO like the HE one could be used by placing it closer than its optimum IFD value and using only its central section, which consists of channels with a low curvature radius and therefore is more efficient for the total reflection of the higher energy photons.

Please cite this article in press as: C. Polese et al., Proposal for a prototype of portable μXRF spectrometer, Nucl. Instr. Meth. B (2015), http://dx.doi.org/10.1016/j.nimb.2015.02.043