Nuclear Inst. and Methods in Physics Research, A 897 (2018) 114–119

Contents lists available at ScienceDirect

Nuclear Inst. and Methods in Physics Research, A

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

Temporary implementation and testing of a confocal SR-μXRF system for bone analysis at the X-ray Fluorescence beamline at Elettra

L. Perneczky [a], M. Rauwolf [a], D. Ingerle [a], D. Eichert [b], F. Brigidi [b], W. Jark [b], S. Bjeoumikhova [c], G. Pepponi [d], P. Wobrauschek [a], C. Streli [a], A. Turyanskaya [a,*]

[a] Atominstitut, TU Wien, Stadionallee 2, Vienna, Austria
[b] Elettra - Sincrotrone Trieste, Basovizza, Trieste, Italy
[c] IFG, Institute for Scientific Instruments GmbH, Berlin, Germany
[d] Micro Nano Facility, Centre for Materials and Microsystems, Fondazione Bruno Kessler, Trento, Italy

ARTICLE INFO

Keywords:
Confocal μ XRF
Synchrotron
Bone analysis
Transportable chamber

ABSTRACT

The confocal μXRF spectrometer of Atominstitut (ATI) was transported and set up at the X-ray Fluorescence beamline at Elettra - Sincrotrone Trieste. It was successfully adjusted to the incoming beam (9.2 keV). Test measurements on a free-standing Cu wire were performed to determine the size of the focused micro-beam (non-confocal mode, 56×35 μm^2) and the size of the confocal volume (confocal mode, $41 \times 24 \times 34$ μm^2) for the Cu–Kα emission. In order to test the setup's capabilities, two areas on different human bone samples were measured in confocal scanning mode. For one of the samples the comparison with a previous μ XRF measurement, obtained with a low power X-ray tube in the lab, is presented.

1. Introduction

The multicomponent nature and complicated structure of biological samples often present a challenge in the analysis of such specimens. Due to the different energies of the characteristic fluorescent radiation and its absorption, depending on the sample matrix, the information depth of the elemental components is strongly varying. The method of confocal μXRF overcomes this problem, as it allows the analysis of the elemental distribution within one well-defined layer, e.g. the surface of the sample, in a raster mode. Obtaining the information only from a defined volume (voxel), formed by the intersection volume of the two foci of the X-ray lenses, allows us to eliminate unwanted contributions from subjacent layers of the sample. The use of synchrotron radiation is highly advantageous, especially for the measurement of trace elements, as the high brightness reduces the required measurement time per point considerably and the linear polarization results in perfectly low background conditions.

There are not that many confocal setups available worldwide, and none at Elettra - Sincrotrone Trieste. To exhibit the viability of a confocal μXRF setup at Elettra - Sincotrone, the μXRF spectrometer of Atominstitut (ATI) [1] was transferred and temporarily installed at Elettra X-ray Fluorescence beamline [2]. This setup has already proven useful in the lab for the analysis of bone samples [3], and has the advantage of being small enough for transportation. Additionally, as it is mounted inside a vacuum chamber, detection of low Z elements is accessible.

2. Setup

2.1. Beamline specifications

The Elettra storage ring operates in top-up mode (310 mA at 2.0 GeV or 160 mA at 2.4 GeV). The light source is a bending magnet and currently the available energy range is 3.7–14 keV. For this pilot experiment, the 4 channels monochromator was set on the multilayer RuB_4C ($n = 150$ periods with spacing $d = 2.51$), and the energy was set to 9.2 keV. The bandwidth is ~150 eV and the flux ~3×10^{11} photons/s. Fig. 1 displays a schematic drawing of the experimental setup. The ATI μXRF spectrometer was installed at the end of the beamline, where the beam, exiting via a Be window (300 microns) presents a beam size of roughly ~300×200 μm^2, an angle of 0.5 deg with respect to the horizontal plane and a divergence of 0.15 mrad.

* Corresponding author.
E-mail address: anna.turyanskaya@tuwien.ac.at (A. Turyanskaya).

https://doi.org/10.1016/j.nima.2018.04.012
Received 28 February 2018; Received in revised form 3 April 2018; Accepted 6 April 2018
Available online 25 April 2018

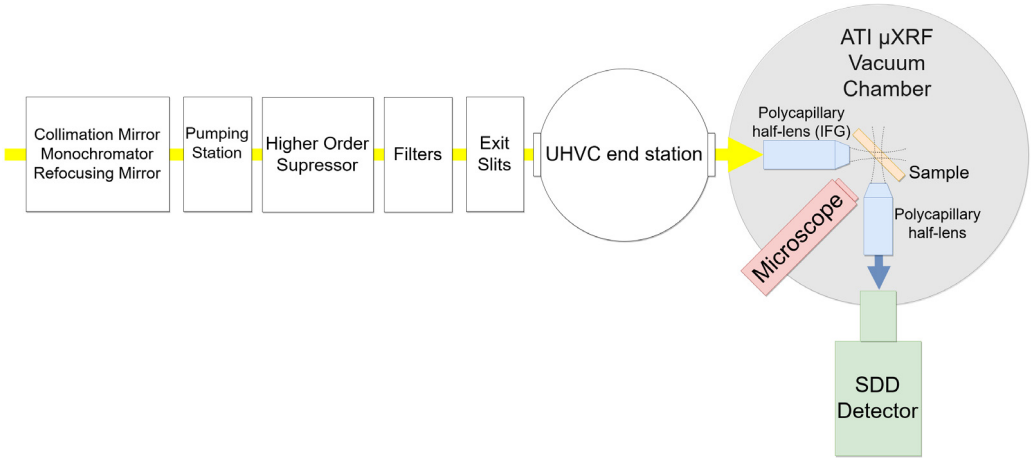

Fig. 1. Schematic drawing of the experimental setup.

2.2. Components of the ATI µXRF spectrometer

The ATI µXRF spectrometer is mounted inside a vacuum chamber. In the lab environment a polycapillary full lens is utilized to focus the incoming X-rays that are produced by a low power tube with Rh anode (20 W). For this experiment, the tube was removed and replaced by an aluminium cap with polyimide foil (d = 8 µm) window to retain the option of vacuum measurements. Furthermore, the polycapillary full lens was replaced by a half lens optic with a working distance of 6.6 mm (provided by IFG, Institute for Scientific Instruments GmbH, Berlin, Germany) to focus the parallel synchrotron beam. In the detection channel, a second polycapillary half lens with a working distance of 5 mm (XOS) in front of the detector entrance window confines the detector's field of vision. Both optics are positioned by piezo-positioner stacks. The sample is mounted on a motorized XYZ-stage via a magnetic base. In the lab, the fluorescence radiation is usually detected by a LN_2-cooled Si(Li) detector with ultra-thin window, however, its large size and heavy weight render it impractical for transportation. Therefore, it was replaced by a SDD detector (Ketek) with 25 µm Beryllium window, 80 mm^2 active area and an energy resolution of 145 eV @ 5900 eV (property of ATI). An optical microscope, focused to the confocal volume position, is utilized to accurately adjust the sample. In Fig. 2, pictures of the confocal setup and the ATI µXRF chamber are displayed.

Measurements are performed using the ATI in-house µXRF software package [4]. Acquired data is fitted with AXIL [5] and the resulting data is processed and visualized elementwise using the software LP-map developed at ATI [6]. All elemental maps and spectra presented are dead-time corrected and converted to counts per second (cps) live-time.

2.3. Setup procedure

2.3.1. Modifications to the laboratory setup

After passing through the beamline's permanent setup, the beam leaves the UHVC end station at a fixed position and with a tilt angle to the horizontal plane. Therefore, it was necessary to be able to accurately adjust position and angle of the µXRF chamber accordingly. A construction for transversal adjustment of the chamber in the plane perpendicular to the beam-axis was designed and built at ATI. It consisted of three aluminium plates with a thickness of ~5 mm. To adjust the horizontal position, base and middle plate were connected with a 1D translation stage. To make fine adjustments to height as well as the angle to the horizontal plane, the top plate was connected to the middle plate with a 3-point support, utilizing three micrometre screws. A schematic drawing of this construction is presented in Fig. 3.

The beam was focused onto the sample by a polycapillary half lens (f = 6.6 mm, focus size ~30 µm, intensity gain ~235 for E = 7.5–10 keV). Since its acceptance angle was small (e.g. 3 mrad for the critical angle at 9 keV [7]), the angle adjustment of the polycapillary optic had to be precise. A special adjustment mechanism was constructed, utilizing the XYZ-piezo positioner stack that is normally responsible for the linear adjustment of the primary optic, converting this linear motion into angular motion via a fixed pivot point (Fig. 4). A small V-shaped aluminium plate, mounted on an aluminium column in front of the positioner stack, served as the pivot point of the polycapillary. An aluminium holder was mounted on the piezo-positioner stack, with a small metal sphere attached to the top of it. This metal sphere served as the hinge of the mechanism. An aluminium ring, with an indentation fitting for the metal sphere, was fixed to the capillary casing with a plastic screw. A U-shaped aluminium plate screwed to the back of the holder ensured that the polycapillary does not collapse to either side. With this setup, moving the piezo-positioner in the plane perpendicular to the beam direction, would result in small angular changes of the polycapillary. This is illustrated in a drawing at the bottom of Fig. 4, where up or down movements result in a change of the angle to the horizontal plane.

It is important to note that by using this mechanism the back of polycapillary would move, therefore, the entrance position of the beam would also change slightly. This means that after finding the right angle, it might be necessary to readjust the translational position of the setup in order to hit the polycapillary in the centre.

2.3.2. Adjustment

A laser level was utilized to achieve a coarse adjustment of the chamber.

As a next step, the first polycapillary half lens was installed in the beam path (excitation channel), and a test sample (Gd screen with Cu wire cross glued on top) was fixed to the sample holder. Utilizing the fine angle adjustment mechanism, an angle scan was performed, i.e. the polycapillary angle was varied in small steps (~25 µm) while measuring. If a fluorescence signal (Gd or Cu) was detected, an X-ray film in front of the polycapillary was used to ensure that the beam passes the optic as centrally as possible. Then, chamber position and polycapillary angle were optimized to achieve a maximum count rate. If no signal was measurable, the coarse adjustment was repeated.

A linescan across a horizontal as well as a vertical free standing Cu-wire (d = 10 µm) was performed to estimate the beam dimensions after the first polycapillary optic, i.e. in non-confocal mode. The spectral data was fitted to obtain the count-rates for Cu–Kα, which were then Gauss-fitted to extract the beam dimensions (Fig. 5). The FWHM was 57 µm for