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Development of a low-energy X-ray fluorescence system combined with X-ray microscopy

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Abstract. A system for Low Energy X-ray Fluorescence (LEXRF) has been implemented in the TwinMic [1] microscope station operating at Elettra synchrotron facility. The integrated LEXRF is coupled with the STXM operation mode. This allows simultaneous collection of XRF maps, brightfield images and phase contrast images [2], providing complementary information about the sample’s chemical specificity and morphology with high spatial resolution. The system is based on a modular setup of multiple Be windowless Silicon Drift Detectors (SDDs), read-out by a custom designed fast multichannel acquisition system [3]. The number of the SDDs (up-to 8 detectors) will be increased in the future in order to minimize the XRF acquisition time (higher count rate and solid angle) and to increase the XRF detection limits, therefore allowing us to acquire XRF maps with sub-micron spatial resolution.

1. Introduction

X-ray Microscopy (XRM) responds the increasing demands in material research for characterization with lateral resolution at sub-micrometric length scales. Combining XRM transmission imaging with spectroscopy, based on X-ray absorption (XANES) or X-ray Fluorescence (XRF), allows complementing morphological information with chemical specificity. While XANES requires stack of images (acquired scanning the photon energy) to be correlated and computationally analyzed, the XRF provides with a single scan at selected photon energy simultaneous identification of many chemical elements and their lateral distribution. Almost all synchrotron light sources have dedicated experimental stations for XRF microscopy using photon energies in the hard x-ray range (usually > 5 keV). Low energy XRF is still poorly exploited, since it requires in-vacuum operation and short working distances of the zone plate focusing optics. The advantage of LEXRF is that it is very adequate for identifying the presence of light elements, e.g. C, N, O, Mg, Na etc, that are the main constituents of the life matter. To our knowledge the only report for LEXRF setup, which allows fluorescence detection down to the C edge, showed only elemental maps of Ca, Na, Si, K, Mn, Fe and Cu lines [4].

Further-on, by implementing LEXRF in a Scanning Transmission X-ray Microscope (STXM) the acquisition of the XRF spectrum for each pixel in the raster-scan can be combined with simultaneous X-ray absorption and phase contrast maps, which provide complementary information of the specimen morphology, allowing the location of the elements to be unambiguously established. Most of the synchrotron facilities performing XRF analysis typically use Ge or Si(Li) detectors, the developed system reported here is based on the use of SDDs. The advantage of the SDDs is a very low output capacitance (of the order of 100 fF), independent of the active area of the device, resulting in a much lower electronic noise with respect to conventional silicon diodes of equivalent area and thickness [5]. Moreover, the integration of the first stage of the front-end electronics [6] on the detector chip allows high energy resolution even at high counting rates with the SDD operated near...
room temperature. The combination of low noise performance and an optimized entrance window [7] for low-energy photon detection makes the SDD ideal for detection of low energy X-rays down to B-Kα line (183eV). SDDs have also the advantage of not requiring liquid nitrogen cooling, which makes them more compact and thus more suitable for a set-up with limited space and mechanical constraints.

In the following we present the LEXRF set-up for TwinMic and the first preliminary results, obtained in the commissioning period, illustrating the performances of the developed system.

2. Experimental setup
The developed LEXRF system has been successfully installed in the TwinMic station, operated in the 250-2200 eV energy range at the Elettra synchrotron facility, in Trieste, Italy [1]. TwinMic is an X-ray Transmission Microscope which hosts Scanning Transmission (STXM) and full-field (TXM) microscopes, with easy switch between the two modes. In STXM mode the sample is raster-scanned allowing simultaneous detection of transmission (absorption, phase) images and emitted signals (photons and electrons). The optical resolution of the STXM is typically chosen as a compromise between photon flux and lateral resolution required for the specific experiment, ranging from 0.05 – 1 µm.

The LEXRF setup has been coupled with the STXM operation mode: the SDDs are acquiring the fluorescence signal emitted by the sample while absorption and phase contrast images are collected by a special configured electron-multiplied fast read-out CCD camera from Andor Technology [2]. The optical scheme of the microscope setup is shown in Figure 1.

The main limitations implementing LEXRF in a STXM are the geometrical constraints due to the short working distance (a few mm) between the sample and the zone plate-based focusing system. In TwinMic the SDDs are arranged in a symmetric concentric configuration as illustrated in Figure 1 with an angle of 70° between incident beam and detector axis. The SDDs-sample distance is around 28mm. The detectors are mounted on a copper water-cooling base plate to remove the heat dissipation from the Peltier cooling of each SDD. Customized pre-amplifying and biasing electronics (provided by Politecnico di Milano/INFN) are located in close proximity to the detectors. The charge preamplifier is based on pulsed reset technique [3,8-9] and is characterized by extraordinary energy resolution performance, high energy resolution stability (resolution worsening is about 3.5 eV at Mn Kα with input photon rate of 150 kcps) and a very low peak shift (below 0.1% over the range of incoming events from 1kcps up to 150kcps).

3. Results
TwinMic was operated in STXM mode using photon energy of 1440eV, chosen to excite the light chemical elements of interest. The pilot experiments were carried out with only two Be windowless 30mm² active area SDDs. The SDDs have a nominal FWHM of 135 eV at Mn Kα cooled down to -20°C. For the pilot experiment the X-ray beam was focused to 1 µm spot size with a photon flux of $10^{10} - 10^{11}$ ph/s in the probe. The microprobe size was a compromise to achieve sufficient flux for these pilot experiments in the commissioning phase of the LEXRF set-up. Some XRF maps were also collected recently with sub-micron resolution.

Fig. 2 shows the first results demonstrating the expanded potential of the TwinMic station. The transmission and XRF images are of an hepatocyte from human liver, which represent one of the important applications in human biology and virology. The aim of such measurements are detection of changes of sub-cellular processes and cell bio-functionalities due to virus replication by externally administered probes, and these commissioning results confirm the feasibility. The XRF maps show the spatial distribution of C, Na and Mg, constructed by considering the total counts corresponding to the C, Na and Mg Kα lines. They can be directly correlated to the sub-cell constituents, distinguished in the transmission absorption and phase contrast maps. For good statistics we used a dwell time of 20 s per XRF spectrum, requiring a total acquisition time of about 10 hours, which will become four times faster implementing all 8 detectors.
4. Conclusive remarks
With the development and the implementation of the LEXRF system TwinMic has become the first microscope in the soft X-ray energy range (250-2200 eV) capable to combine XRF elemental mapping with simultaneous acquisition of absorption and phase contrast images, proving complementary morphological information. For the pilot experiment a spatial resolution of 1 \(\mu\text{m}\) was chosen in order to have a reasonable count rate and acquisition time. The upgrade of the LEXRF set-up to 8 SDDs will allow pushing the spatial resolution sub-100 nm range by increasing the acceptance solid angle as well as reducing the measurement time when using modest sub-\(\mu\text{m}\) resolution sufficient for probing a great variety of specimen.

In conclusion, the LEXRF setup have expanded the potentials of the TwinMic station, opening new application opportunities in material science, human and marine biology, virology, environmental science and most recently in food science and cultural heritage.

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**Fig. 1:** Optical scheme of the TwinMic STXM mode operation with the LEXRF setup implemented.

**Figure 2.** Scanning transmission and LEXRF maps of an hepatocyte from human liver simultaneously acquired: BF is the brightfield or absorption map, DPC is the corresponding differential phase contrast map. Image size of 40 x 40 \(\mu\text{m}^2\) with 40 x 40 pixels raster scan and XRF acquisition time of 20s per pixel.