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Development of a synchrotron-based wide-band high-energy resolution spectral K-Edge Subtraction imaging setup

K-Edge Subtraction (KES) imaging allows to quantify the presence of one or more contrast agents embedded within a matrix material, and it can be applied both in planar and tomographic configurations. KES signal derives from the sharp rise in the attenuation coefficient corresponding to the binding energy of the contrast element K-shell electrons. In its simplest formulation, KES consists in subtracting images obtained at energies above and below the K-edge. For this reason, any KES imaging setup must provide spectral information, either by using spectral detectors [1] or by tuning/shaping the X-ray spectrum [2].
Many Synchrotron Radiation (SR) applications of KES fall in the latter category and make use of bent Laue monochromators, selecting an energy bandwidth including the contrast agent's K-edge. Cylindrically bent Laue crystals allow to focus the laminar SR X-ray beam and to diffract a relatively wide energetic bandwidth (few keV), which is (at first approximation) inversely proportional to the bending radius. The resulting beam is energetically dispersed in the diffraction plane, so that different positions in space correspond to different energies. In a typical KES setup, the sample is positioned and scanned through the focal spot while a bi-dimensional detector is placed downstream. In this way, one image axis (x) shows the sample spatial distribution while the other (y) encodes its spectral decomposition (Fig. (a)). If the contrast medium's K-edge lies within the selected bandwidth, the recorded image shows a strong intensity variation along the energy axis. The sharpness of this transition defines the energy resolution of the system. So far, most of the SR-based KES systems have shown poor energy resolution meaning that a large fraction of the beam (typically about 1/3) is involved in "edge crossing"and must be filtered out [3]. This brings to a focal spot blurring, to the need of beam splitter devices to remove crossover energies, hence to a reduction in the X-ray flux, and a-priori excludes the implementation of techniques requiring fine energy resolution as, e.g., X-ray Absorption Spectroscopy (XAS).
In this context, the INFN-funded KISS (K-edge Imaging with Spectral Systems) project aims to develop a wide-band high-energy resolution KES setup at the SYRMEP beamline at the Elettra synchrotron facility (Trieste, Italy). In fact, energy resolution can be maximized if the Laue crystal is asymmetrically cut, and the asymmetry angle is such that the geometrical focus corresponds to the single-ray focus [3]. This "magic condition" [3], is primarily determined by the selected energy while it has a shallow dependence on the crystal bending radius. For this reason, by using small bending radii, a system featuring both a wide-energy bandwidth and high-energy resolution can be pursued. Allegedly, the most critical part of the KES experimental setup is the frame required to bend and hold the crystal avoiding its breaking. Our frame (Fig. (b)) is manufactured, via a numerical control machine, from a single aluminum block and it includes a curved nylon coated surface, where the crystal is strained, defining the final bending radius. To bend the crystal a smaller frame featuring two plastic rods is positioned on top of the silicon wafer and, by using two fine screws, it is pushed against the crystal to make it adhere to the larger frame. Thanks to this design the mechanical stress is more uniformly distributed, allowing to bend a rather thick (0.77 mm) silicon crystal to a small bending radius of 0.5 m without any rupture: this value is smaller than bending radii reported in literature so far ($\geq$ 1 m) [3,4]. Such small bending radius allows the use of multiple contrast agents with K-edges within some keV energy range. Additionally, using thick crystals increases diffraction efficiency, hence the flux. The first experimental planar images obtained with the novel setup have been acquired with a 4-inches Si wafer by using the (111) reflection and the Pixirad1-PixieIII photon-counting detector, featuring a pixel size of 62 μm [5]. The sample was composed by two plastic cuvettes filled with an iodine-based solution (25 mg/ml, K-edge at 33.2 keV) and Xenon gas at atmospheric pressure (K-edge at 34.5 keV).
Energy resolution analysis, performed by fitting the K-edge transitions with an erf, shows a resolution around 100 eV (FWHM) for both elements with a wide energy bandwidth of about 5 keV (Fig. (c)). By applying

a suitable spectral KES decomposition algorithm, based on least squares minimization, a quantitative material decomposition has been performed, singling out the iodine and xenon signals (Fig.(d)). In addition to the reported measurements, the first tomographic images of biological samples containing iodine-based and barium-based (K-edge at 37.4 keV) contrast agents have been performed.
The results obtained with the novel KES setup demonstrate the feasibility of multiple K-edge applications making use, for instance, of iodine, barium, and xenon-based contrast agents, that is appealing for simultaneous pulmonary, cardiac, and gastrointestinal imaging. Additionally, the achieved energy resolution suggests further investigations at lower X-ray energies to perform wide field of view XAS.

Figure Sketch of the experimental setup (a), crystal bending frame (b), K-edges imaging results (c), spectral decomposition (d).

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