ESRF	**Experiment title:** Exploring the mechanisms of a novel X-ray nanolithography technique for superconducting oxides by scanning XRD	**Experiment number**: MA-2594
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Report:

The investigation of high-T_c superconducting oxides by means of synchrotron radiation micro- and nano-probes presently represents a very active field. Although multiscale auto-correlation properties have been detected for the O-ion spatial distribution for several systems such as LaCuO [1], YBCO [2] and Bi-2212 [3], another approach more oriented towards possible applications was missing for these materials.

Our group recently filled this gap showing that both the electrical and the structural characteristics of the Bi-2212 superconducting oxide can be modified by high dose irradiations at 17 keV [4]. In a following experiment performed at ID16B-NA, we have exploited this fact to fabricate a proof-of-concept Josephson device [5] by exposing selected regions of a Bi-2212 micro-crystal to a flux of 1 x 10^{11} ph/s at 17.6 keV with a spatial resolution of 55 x 56 nm^2 in order to draw "trenches" regions in our crystals. These results are very encouraging, suggesting the possibility to develop a novel direct-write X-ray nanopatterning approach, whose ultimate resolution could be in the range of the photoelectron inelastic mean free path (~ 10 nm).

To better clarify the atomic-scale mechanism which leads to the O doping modification during X-ray irradiation, during experiment MA-2594 we measured 3 Bi-2212 micro-crystals: 2 free-standing (SBLP12 and SBLP12_2) and one mounted on a substrate allowing electrical characterization (WBLP04). We worked at 13 keV with a beam size of 2.5 x 2 μm^2 and a flux of 6 x 10^{11} ph/s.

For the free-standing samples we irradiated selected regions in subsequent steps corresponding to increasing X-ray doses and we acquired XRD patterns at each dose to monitor the structural variations in the sample. The XRD patterns were acquired spanning a rotation angle of 270° with a step of 0.2°. From these XRD patterns it is possible to perform a single crystal refinement of the structure (see Figure 1) and to determine the cell parameters. The length of the *c*-axis is particularly relevant since it can be directly related to the O content which determines the T_c.

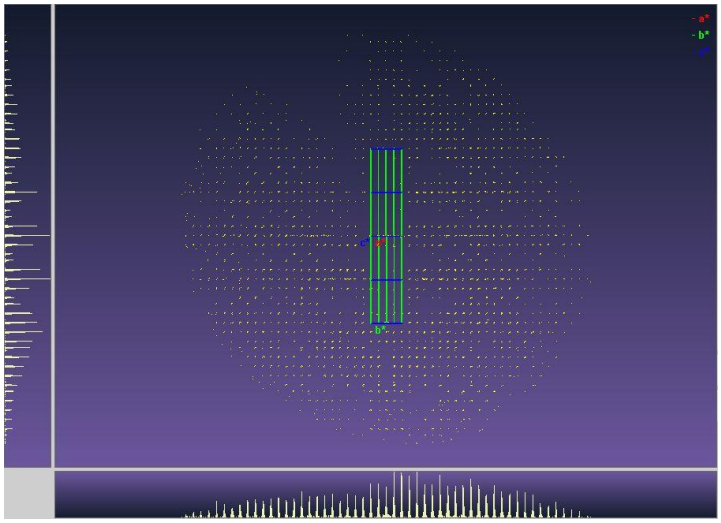

Figure 1. Reciprocal lattice reconstruction from the XRD patterns acquired on the free-standing SBLP12_2 Bi-2212 sample before irradiation. The corresponing refined lattice parameters are a = 5.3639(8), b = 5.4218(8), c = 30.740(5).

Figure 2 shows the evoultion of the system after 3 doses of X-ray irradiation: the initial crystalline pattern (Figure 2a) of the Bi-2212 single micro-crystal seems to evolve into a polycrystalline system (Figure 2b) as a consequence of an increased structural disorder, probably because of the lattice misalignment induced by the oxygen depletion. Further analyses are in progress to quantitatively determine the variation of the lattice parameters (in particular of the *c*-axis) as a function of the X-ray dose.
For the sample mounted on the substrate allowing electrical characterization, after each X-ray irradiation step and subsequent acquisition of the XRD patterns (in this case the rotation angle span was limited to 20°), a resistance vs temperature characteristic was acquired in order to correlate the structural variations monitored by XRD with the chamge in the sample electrical properties.

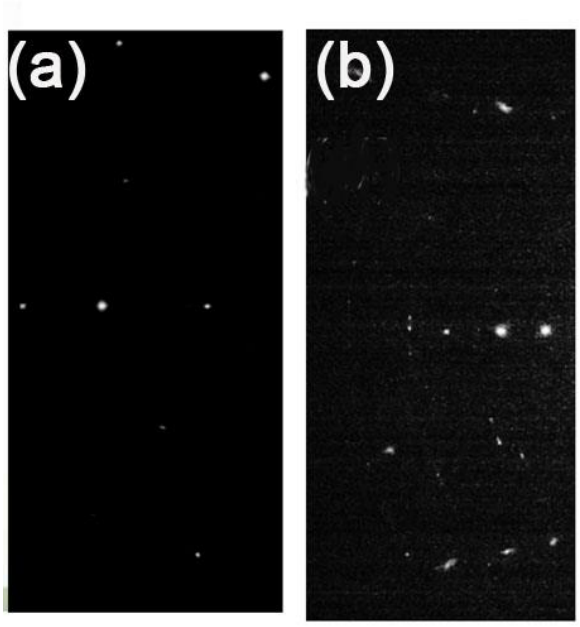

Figure 2. XRD patterns acquired before (a) and after X-ray irradiation (b) highlighting the progressive evolution of the pristine single Bi-2212 crystal to a polycrystalline system.

References

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[5] M. Truccato *et al.* Nano Lett., **16**, 1669 (2016).