

**Experiment title:**

Determination of thermal reversible and irreversible effects during crystal irradiation at 4th generation synchrotrons

**Experiment number:**

MA-5374

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**Report:**

Radiation damage induced by intense X-ray irradiation is becoming an increasingly important concern with the development of extremely bright sources such as 4<sup>th</sup> generation synchrotrons.

Material modifications can also be exploited for tuning the electrical properties in oxide materials such as superconducting cuprates [1,2,3] and semiconducting oxides [4,5], but the understanding of the microscopic mechanism underlying material modification is still to be determined.

In this regard, one possibility is the effect of intense temperature gradients induced in materials upon X-ray irradiation, which could generate shock waves.

From our FEM simulations, it seems that huge thermal gradients can be generated in materials such as rutile TiO<sub>2</sub> and SrTiO<sub>3</sub>, and these thermal effects seem to be more pronounced at cryogenic temperatures with respect to room temperature because of the lower heat capacity of materials.

To test this hypothesis, we performed single crystal XRD, exploiting the pulsed nature of synchrotron radiation to simultaneously heat the material and collect XRD patterns by using the same bunch of photons. By changing the flux of the incident beam, a shift of the peaks is expected due to thermal expansion and consequent increase in d-spacing.

Different materials have been tested, namely SrTiO<sub>3</sub>, BaF<sub>2</sub> and a PtEuTe multilayer on top of a BaF<sub>2</sub> substrate. These materials were selected because of the low penetration depth that should lead to a higher temperature increase and because of the high thermal expansion coefficient (in particular for BaF<sub>2</sub>).

Indeed, in bulk BaF<sub>2</sub> we detected a change in the diffraction peak as a function of the beam intensity by performing  $\theta$ -2 $\theta$  scans, in the sense that a broadening of the peak towards lower  $\theta$  values (and correspondingly higher d) was measured.

This change in the peak shape is possibly due to a temperature distribution across the irradiated volume.

Interestingly, this change is fully reversible and the peak goes back to the original position once the flux is reduced, suggesting the absence of permanent damage of the material (see Figure 1).

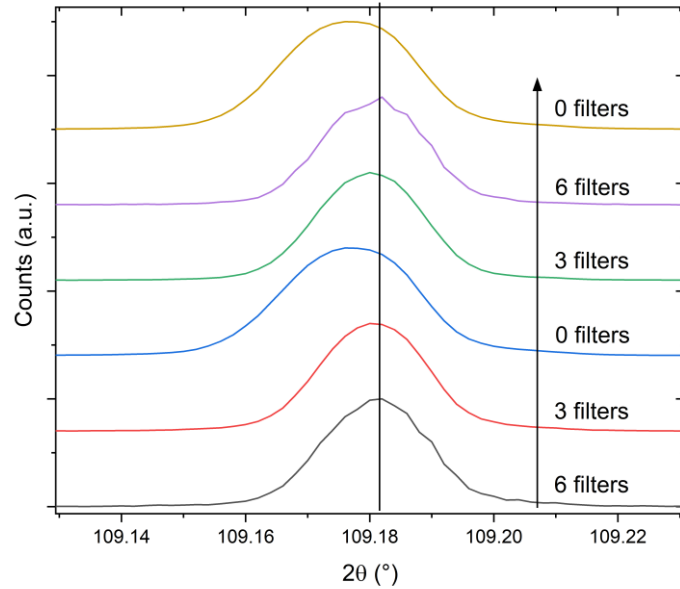


Figure 1:  $\theta$ - $2\theta$  scans for  $BaF_2$  at different photon fluxes.

To have a more uniform temperature field across the volume of interest, we investigated also a PtEuTe multilayer, and in this case a real shift of the peak was detected for the PbTe buffer layer (see Figure 2).

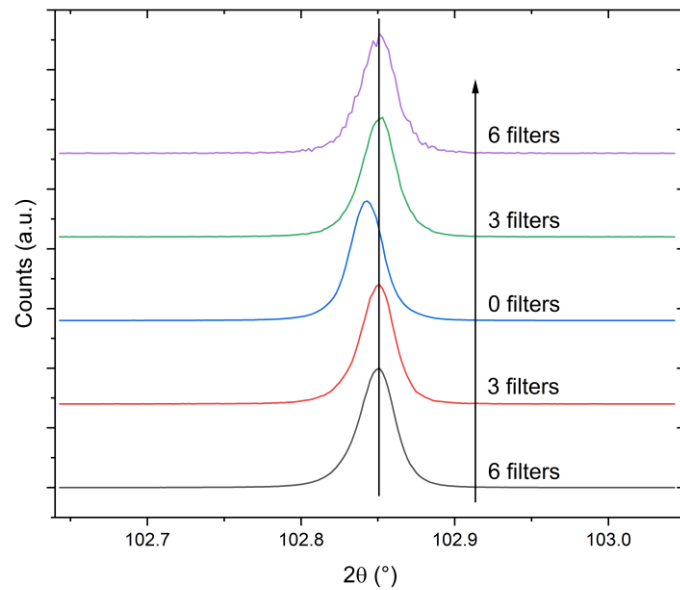


Figure 1:  $\theta$ - $2\theta$  scans for  $PbTe$  at different photon fluxes.

To test the hypothesis that material damage is enhanced at cryogenic temperature, we repeated the same measurements at 8 K, and actually some irreversible change was detected. However, the beamtime was barely enough to check the existence of the phenomenon and nothing can be said at the moment about its details. A more in depth data analysis has still to be carried out, but preliminary results are encouraging and suggest that this technique is suitable to check the modifications induced by X-rays in materials.

## References

- [1] A Pagliero *et al.* Nano Lett., **14**, 1583 (2014).
- [2] M. Truccato *et al.* Nano Lett., **16**, 1669 (2016).
- [3] M. W. Rabbani *et al.* Crystal Growth & Design, **21** (6), 3299-3309 (2021).
- [4] L. Mino *et al.* Adv. Electron. Mater., **5**, 1900129 (2019)
- [5] A. Alessio *et al.* Phys. Status Solidi RRL, **15**, 2100409 (2021)