	Experiment title: Growth and structure of Fe Doped tetragonal CuO/STO(001)	**Experiment number**: MA-5164
Beamline: BM32	**Date of experiment**: from: 24 May 2022 to: 30 May 2022	**Date of report**: 09/09/2022
Shifts: 18	**Local contact(s)**: Lucio Martinelli	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Maurizio De Santis*, Tom Mocellin*, Xavier Torrelles$, Veronique langlais-Abad#

**Institut Néel, CNRS, 25 rue des Martyrs BP 166, 38042 GRENOBLE*

$ ICMAB (CSIC) , Bellaterra, 08193 BARCELONA

 # CEMES (CNRS) 29, rue Jeanne Marvig B.P. 4347 FR - 31055 TOULOUSE

Report:

The aim of the experiment was the growth of Fe doped CuO tetragonal thin films and the study of their structure and composition by GIXRD and resonant x-ray diffraction *in situ*.

The common melaconite CuO phase is monoclinic, but a tetragonal CuO phase was recently grown in epitaxy on $SrTiO_3$(001) substrates up to a thickness of about 3 nm. In this phase the superexchange interaction increases due to the bonding angles configuration. Therefore, peculiar magnetic properties are expected for a tetragonal CuO film doped with magnetic elements.

CuO synthesis requires, in MBE regime, the presence of an oxygen plasma or of atomic oxygen.

In our experiment we used an oxygen cracker to split molecular oxygen and oxidize Cu evaporated on clean $SrTiO_3$(001) substrates kept at a temperature between 400°C and 600°C. Unfortunately, the INS2-BM32 UHV chamber geometry does not allow to approach the gas cracker very close to the sample, and we discovered during the experiment that this configuration was unable to produce CuO. Moreover, the cracker was polluted with sulfur, due to a previous experiment on BM32, which was deposited on the sample while operating. We were therefore obliged to change the sample growth strategy. We followed then a two-step procedure. Cu was deposited on STO by MBE and exposed to 10^{-6} mbar of molecular oxygen in the BM32 UHV chamber. Then it was annealed *ex situ* at 700°C in 1 bar O_2 for 4 hours. GIXRD was then measured on BM32. Fig1. Shows an in-plane GIXRD map of one of the samples, with the reflections indexed in the STO unit cell. An epitaxial film in observed, with monoclinic CuO structure and orientation (010). Several domains are presents; however, it looks like that the β angle in-between a and c axes is reduced compared to the bulk value due to the epitaxial constraint, which is promising for improving the superexchange interaction. Further diffraction analysis is in progress.

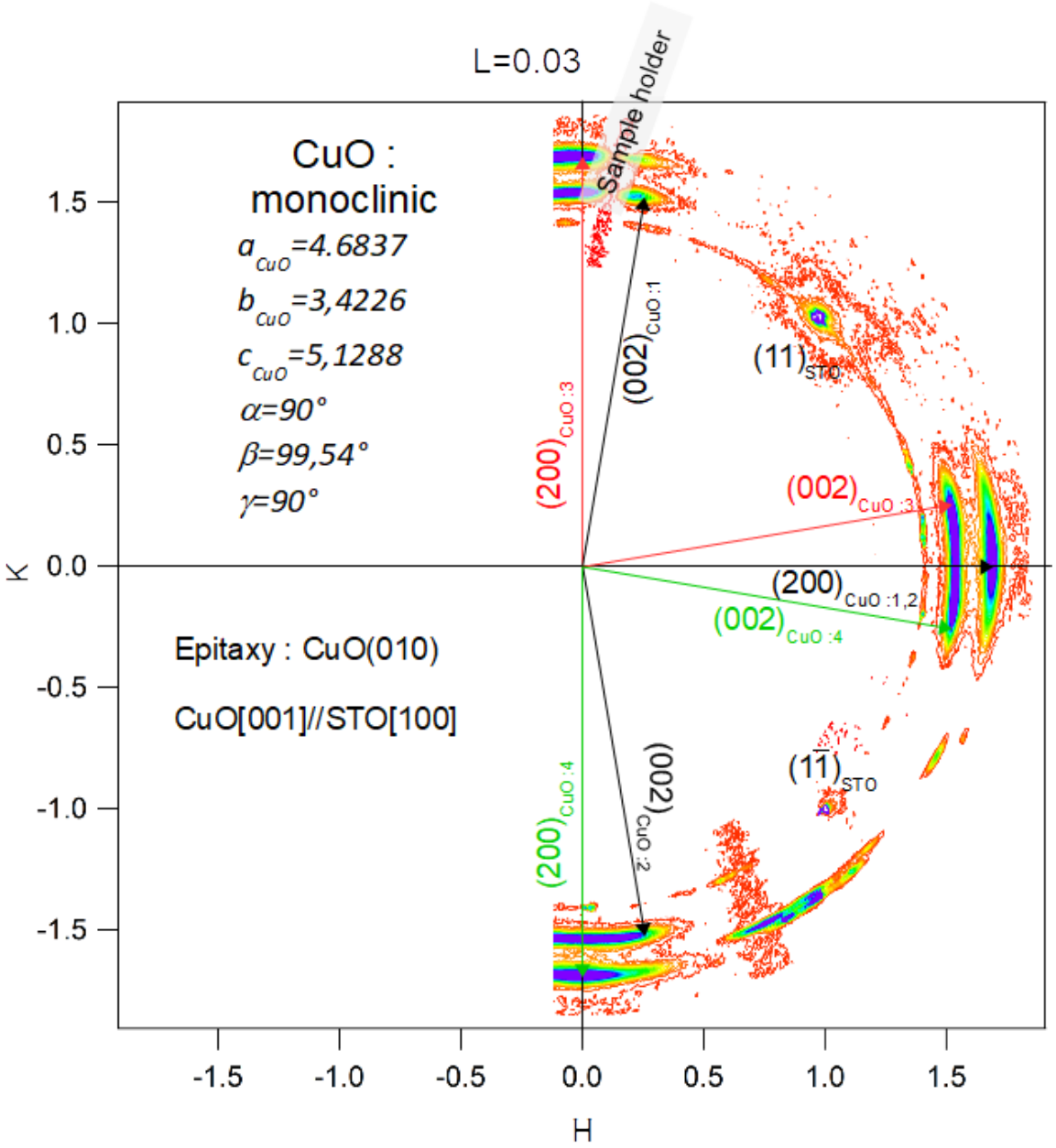

Fig. 1 In-plane GIXRD map of 2.2 nm Cu deposited on $SrTiO_3$(001) after annealing at 700°C in O_2 atmospheric pressure.

Films with Fe doping were grown by a similar procedure, by codeposition of Fe and Cu on STO with a fixed flux ratio, followed by *ex situ* annealing in O_2. All films show a monoclinic structure. The atomic substitution of iron on the copper site of the CuO structure can be studied bu resonant x-ray diffraction at the iron *K* edge. We had time to measure one sample only, elaborated with a large ratio of iron versus copper fluxes (nominal deposited thickness: 1 nm Cu + 0.55 nm Fe). After annealing in molecular oxygen, RXRD was measured both on STO CTRs at different values of the momentum transfer perpendicular to the surface and on peaks of the CuO structure (Fig.2). Data show a phase separation with the growth of an iron oxide layer in coherent epitaxy on the STO. Iron inclusion in the CuO structure is weak, and need to be quantify with theoretical calculations performed with the FDMNES code. Data analysis is in progress.

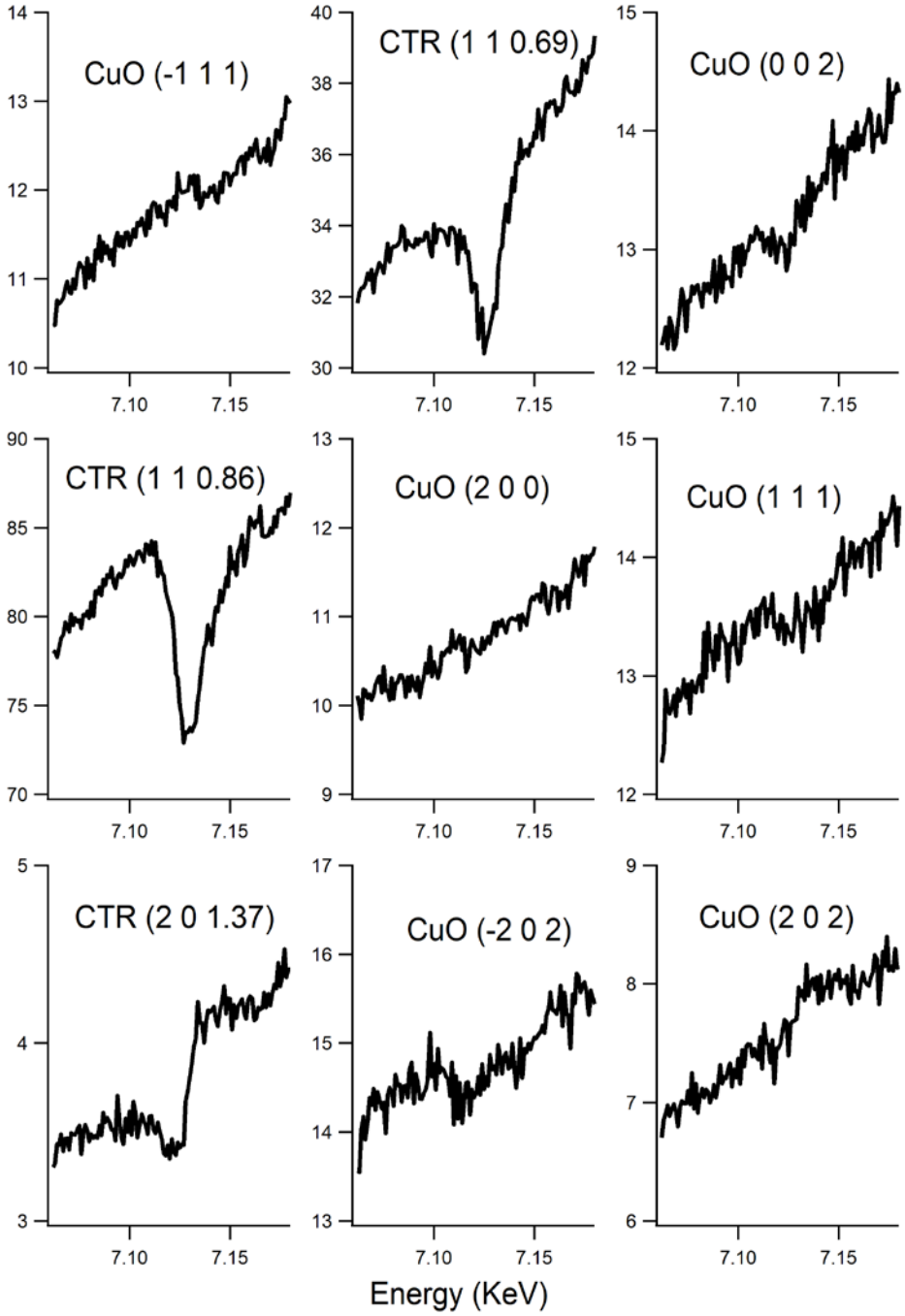

Fig. 2 RXRD measured at the iron *K* edge on STO CTRs and on a few peaks of the CuO structure