ESRF	**Experiment title:** Time resolved relfection mode XAFS study of the oxygenation process in ReBCO superconducting thin films	**Experiment number**: **HS-1190**
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Report:

YBCO films grown by the physical vapor deposition techniques (PVD), are deposited at high temperature in partial oxidizing atmosphere and are tetragonal and non superconducting with an oxygen content lower than x=6.3. The tetragonal to orthorhombic transition is accomplished by cooling the sample to room temperature in high oxygen pressure (≤760 Torr) or by an annealing step at intermediate temperature (500 °C). During this process the oxygen content increases until the superconducting optimally and/or over-doped phases with x~7.0 (OI phase) is reached. It is important therefore to understand the physical process leading to the oxygentaion process. Here we report a direct evidence of local structural changes in the CuO_2 plane in the oxidation process of epitaxially grown high quality $Y_1(Nd_{0.05}Ba_{1.95})Cu_3O_x$ (YNBCO) superconducting thin films. To best of our knowledge, the time resolved X-ray Absorption Near Edge Structure (XANES) spectroscopy has been used for the first time to monitor the evolution of the local structural transformations occurring during the oxygenation of REBCO superconducting thin films. The XANES measurements are made in the reflection mode using grazing incidence geometry to enhance the contribution of the thin film (thickness ~150 nm), that allows us to avoid spurious effects due to the sample substrate. The YNBCO sample was mounted on an heater in a vacuum chamber, placed on a two axis goniometer for the sample alignment. The films were reduced by a high temperature treatment at 570 °C in a pressure of 10^{-5} Torr, leading to the decreasing of the oxygen content from x~7.0 to x<6.3 (tetragonal phase) as confirmed by x-ray diffraction measurements, and allows the evaporations of the contaminants on the surface. Successively the films were cooled down to 500 °C and the oxygenation was promoted by introducing pure oxygen (at the pressure of 600 Torr) in the sample chamber. The temperature and the oxygen pressure were kept constant during the process. The grazing incidence angle (α~0.4 degree) was chosen so that the penetration depth of the incoming beam was lower than the sample thickness. A position sensitive detector (CCD camera) was used for the parallel data detection. The intensities of the incoming and outcoming beams (I_0 and I_1) were measured at the sample position by detecting the direct (empty) and reflected beams respectively. The optimal acquisition time for each spectrum was found to be equal to 17.5 s for the sample and ~250 ms for the empty beam. The total acquisition time was ~4100 s, i.e., 230 spectra were recorded for the oxygenation.

Figure 1 shows reflectivity of fully oxygenated YNBCO thin film, measured as a function of energy across the Cu K-edge, representing the raw data. The reflectivity spectrum shown in the figure was measured 4130 s after starting of the oxygenation. The peak intensity shows a decrease with non-linear behavior as a function of oxidation process (oxygenation time). This happens due to the structural changes within the CuO_2 plane, which involves changes in the Cu-O bonds and Cu-O-Cu buckling angle. Appearance of different oxygen ordering in the Cu-O chain of the YNBCO system are responsible for the local structural transitions like behaviour revealed by the time evolution of the intensity of the main peak in the reflectivity spectrum. We can define at least two clear drops representing respectively the transitions from state α to β and β to γ, accompanied by the plateau regions α to β and γ. The reflectivity spectrum shows an interesting time evolution with step-like changes, revealed by time dependence of the main peak intensity. The step-like changes are due to local structural transitions derived by oxygen intake of the Cu-O(1) plane in the system. Similar step-like (however broad) transitions are found in the oxygen content versus superconducting transition temperature where these steps are found to be due to different oxygen ordering in the Cu-O(1) plane of the REBCO system. Analogously we have argued that the local structural transitions in the oxygenation are due to local oxygen ordering in the Cu-O chains in different configurations.

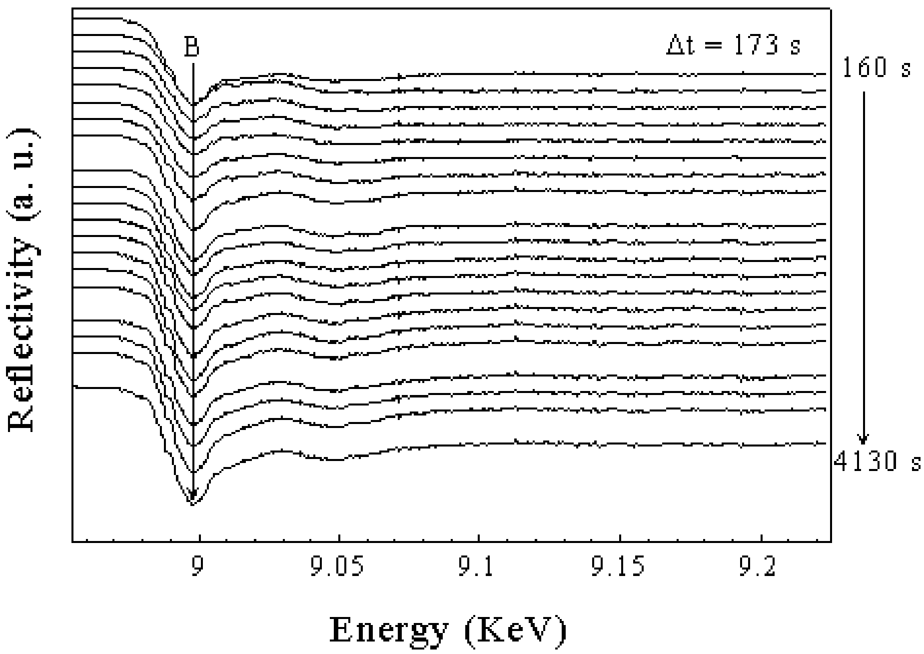

Fig. 1. Reflectivity spectra measured during the oxygenation process