ROBL-CRG	**Experiment title:** "Phases transformations of multi-layered HTS /dielectric/ manganate and HTS / manganate / HTS structures in real-time synchrotron X-ray scattering study"	**Experiment number**: 20_02_047 EU M05
Beamline: BM 20	**Date of experiment**: from: 13.07.2001 to: 17.07.2001	**Date of report**: 25.02.2002
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Report:

The goal of this project was to investigate the phase transformation of thin multi-layer structures from High Temperature Superconductor - $Y_1Ba_2Cu_3O_{7-\delta}$ (YBCO) and manganate - $La_{0.7}Sr_{0.3}MnO_3$ (LSMO) both deposited on different substrates by means of real-time synchrotron X-ray scattering. The SR-XRD experiments are in pursuit of complex and fundamental questions concerning the development of new devices, which should encounter new demands. We studied the temperature influence on the phase formation and phase interaction process. The latter was possible only using SR at ROBL, because the process demands short measuring times in order to follow the process adequately. Additionally, we found the temperature range of existence of the superconducting phase analyzing the lattice parameter and the peak evolutions. The formation of new unexpected phases was also registered. We established the general temperature range of the superconducting YBCO phase formation. To attain this purpose, a special chamber was constructed to fit the goniometer and to work in oxygen atmosphere at elevated temperatures. The measured structure was 40 nm quenched in vacuum YBCO deposited on $SrTiO_3$ (STO) substrate. The experiment carried out was designed to simulate the process of oxygen uptake and superconducting phase formation such as it takes place in the deposition set-up. A set of scans was measured from 650^{o}C to room temperature in oxygen pressure of 900 mbar. The oxygen content is correlated with the lattice constants of the YBCO layer. Its structure consists of three cubes, with yttrium or barium at the center, copper at the corners, and oxygen at the middle of each edge with the exception of the middle cube, which has oxygen vacancies at the outer edges. The critical feature in this structure is the presence of two sheets of copper-oxygen ions, located above and below the oxygen vacancies, along which superconduction takes place. The transport of electrons perpendicular to these sheets is not favored, making the YBCO structure severely anisotropic. One of the challenges in fabricating crystalline YBCO ceramics capable of passing large currents is to align all the grains in such a manner that their copper-oxygen sheets line up. We calculated the lattice parameter "c" in function of the temperature, which is related with the oxygen content. Fig. 1 displaces the one-to-one dependence, which is quasilinear from 600^{o}C to 450^{o}C and then leads to saturation. The relative peak shifts give valuable information concerning the lower temperature limit, which was found to range from 420-450^{o}C – Fig. 2. The peak amplitude was also studied due to its correlation with the coherent domain size of diffraction. The peak amplitude evolution, presented on Fig. 3, becomes significant from about 570^{o}C and saturates at about 420^{o}C.

Taking account of all considered data we can deduce that the temperature range of phase formation ranges from 570°C to 420°C. It is worth noting here, that when a film is grown at insufficient oxygen supply other phases could be formed, such as e.g. $BaCuO_2$. This phase was confirmed both by SR-XRD and high precision RBS simulation. The latter method states that this layer is thick about 20 nm and lies on the interface. In Fig. 4 the beginning of the process of oxygen uptake is shown at the highest temperature. With red arrows the reflection from Y_2O_3 and $BaCuO_2$ phases are shown. Here, all the planes (00l) are shifted left (to lower 2θ) in comparison with the orthorhombic superconducting phase. Moreover, peak (004) is missing. At the end of this process – Fig. 5, the reflections from the $BaCuO_2$ phase disappear and the (004) YBCO peak appears. The (00l) peak positions correspond to the peak reflections representative for $Y_1Ba_2O_7$.

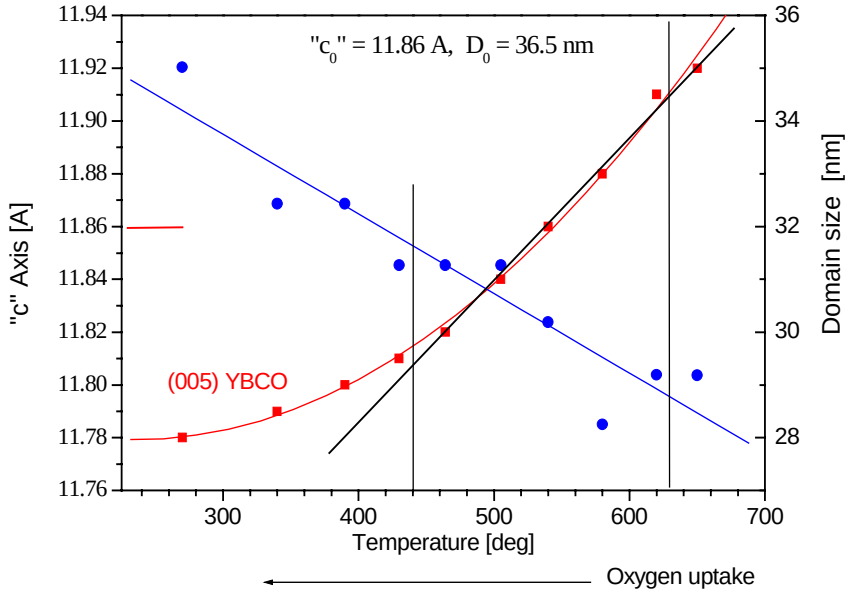

Fig. 1: The lattice parameter "c" as a function of the oxygen uptake. On the left axis, the domain size evolution is shown as a function of the temperature.

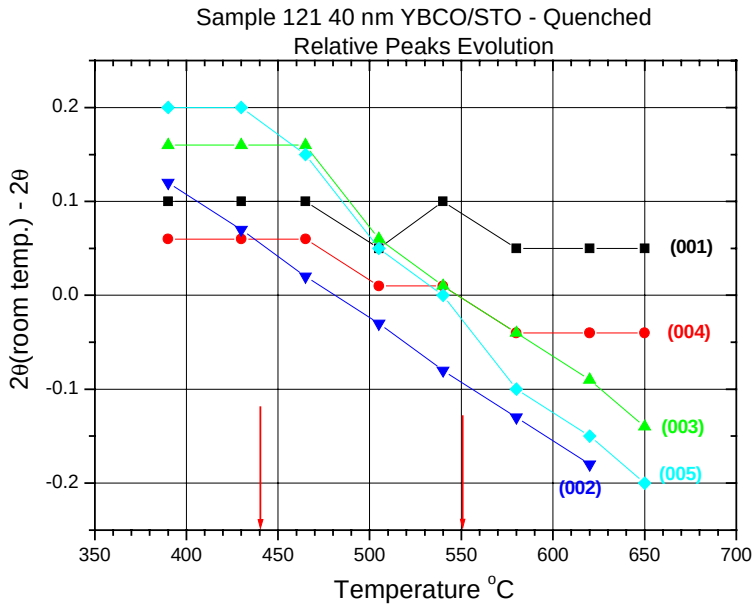

Fig. 2: The relative peaks evoluton for 40 nm quenched YBCO/STO.

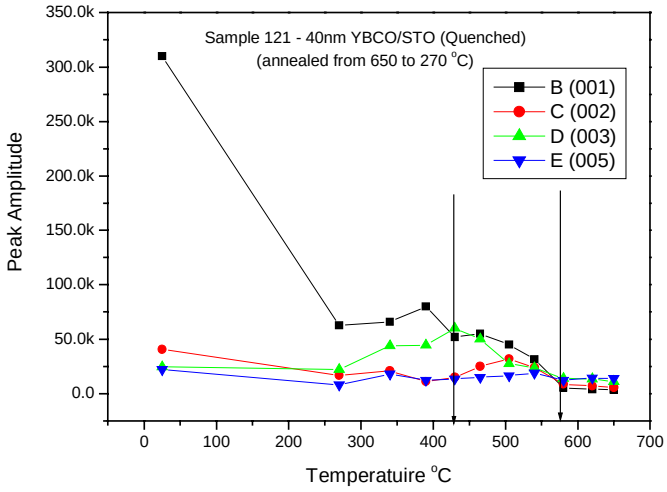

Fig. 3: Peak amplitude variations as a function of the temperature.

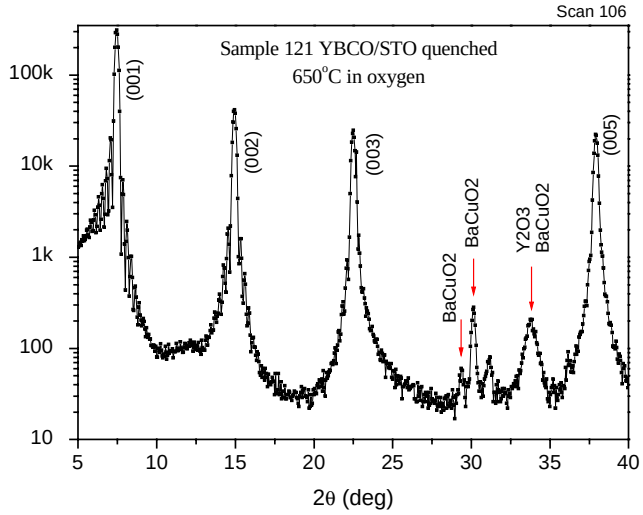

Fig. 4: YBCO/STO scan at the beginning of the oxygen uptake. Scan at 650°C.

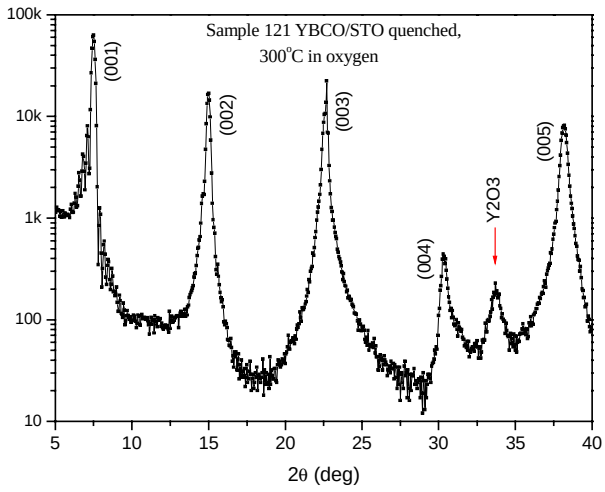

Fig. 5: YBCO/STO scan (at 300°C) at the end of the oxygen uptake process.

Comments to the FZR/ROBL team

This project was put into practice owing to the careful supervising of the project coordinator Dr. habil. W. Matz from the Institute of Ion Beam Physics and Materials Research in Rossendorf.

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