

# YBCO PHASE TRANSFORMATIONS STUDIED BY REAL TIME SYNCHROTRON RADIATION



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*The BCS Theory: Central inherence – complex coupling between a pair of electrons of opposite spin and momentum trough an interaction with lattice phonons.*

*Electrons form Cooper pairs; The maximum length at which the phonon coupled attraction occur - the coherence length  $\xi$ .*

*Since the electron coupling is weak the energy difference (normal-superconducting state)  $2\Delta$  is small ( $2\Delta = 3.5kT_c$ ) & appears in the density of states centered about the Fermi energy at 0 K.*

*When T raised the Amplitude and Frequency of atomic motion increase, interfering with the propagation of phonons between correlated Cooper pairs; The attraction between electrons is diminished and  $2\Delta \downarrow$ .*

*Any perturbation in structure/composition extending over the  $\xi$  can alter  $T_c$  or  $2\Delta$ , placing a practical limit on useful superconducting behavior.*

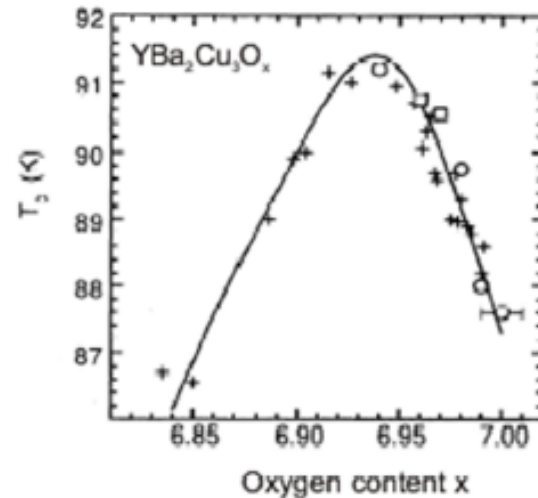
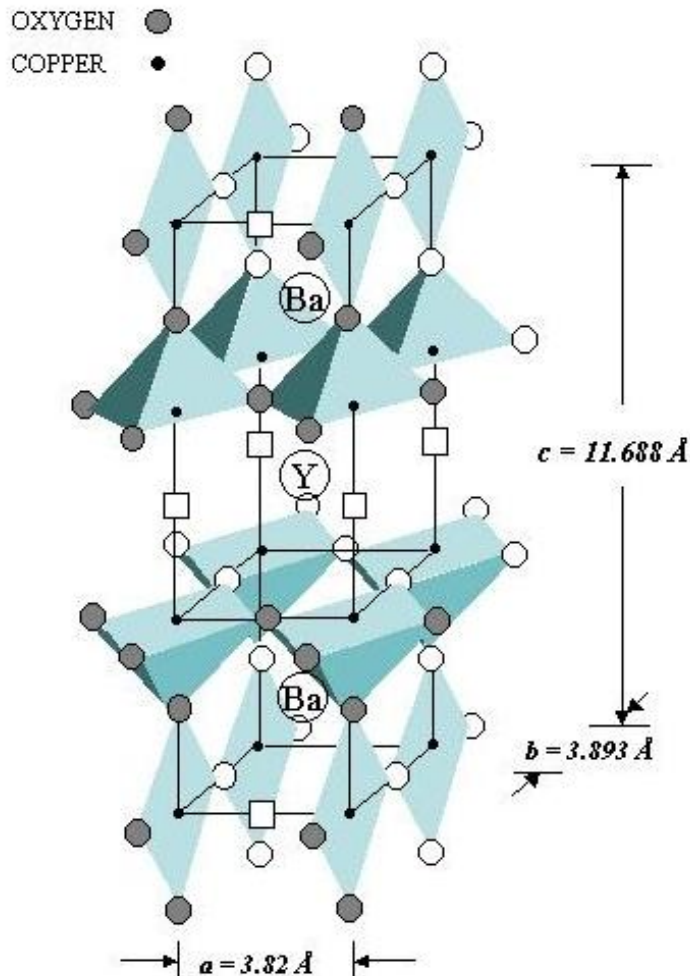
A link between  $T_c$  and the fundamental nature of the material is suggested by the BCS formula,  $N(E_F)$  is the DSST at Fermi level,  $U$ -magnitude of the attractive electron-lattice interaction,  $\gamma$  -the lattice frequency.

$$T_c = \frac{1.14h\gamma}{k} \exp\left(-\frac{1}{N(E_F)U}\right)$$

Efforts has been expended in correlating the crystal structure and oxygen content with  $T_c$ .



Oxygen affect the atom spacing and modify the valence of Cu as well as the Cu-O bond length.

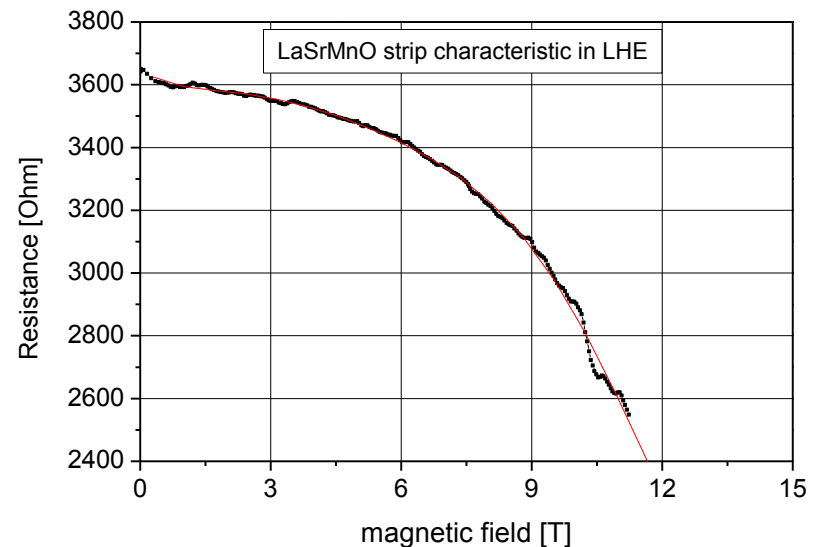
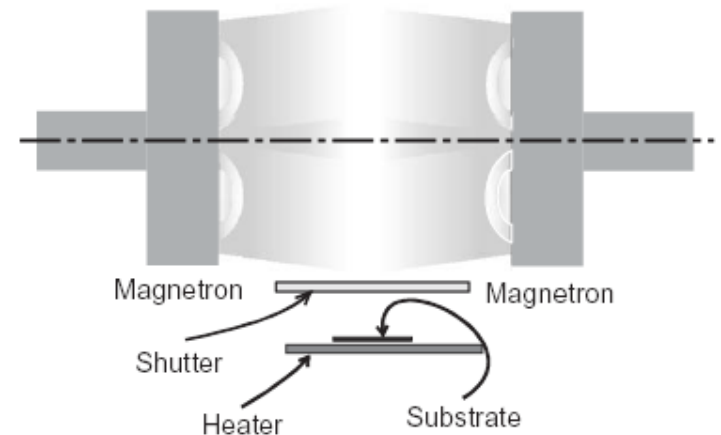
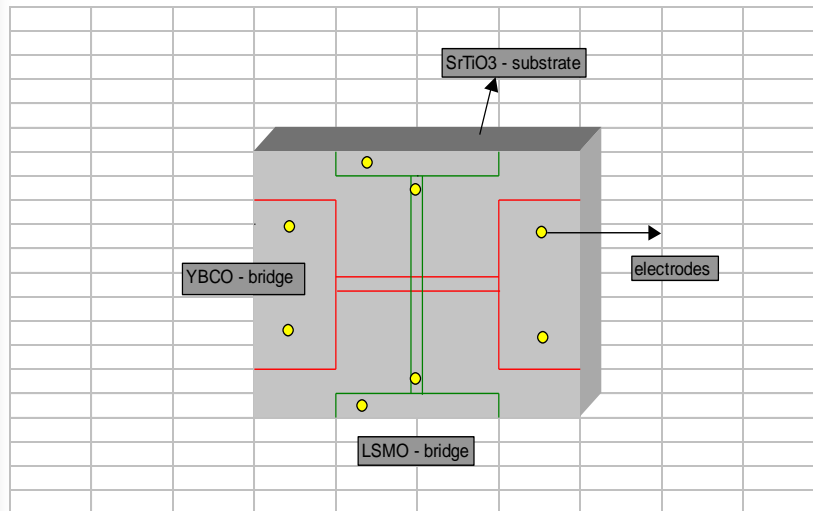


Four type structures; YBCO/STO, LSMO/STO, YBCO/LSMO/LAO and LSMO/YBCO/STO were deposited and characterized in order to solve numerous questions about the growth mechanisms, phase formation and phase interactions.

*Analytical Techniques:*

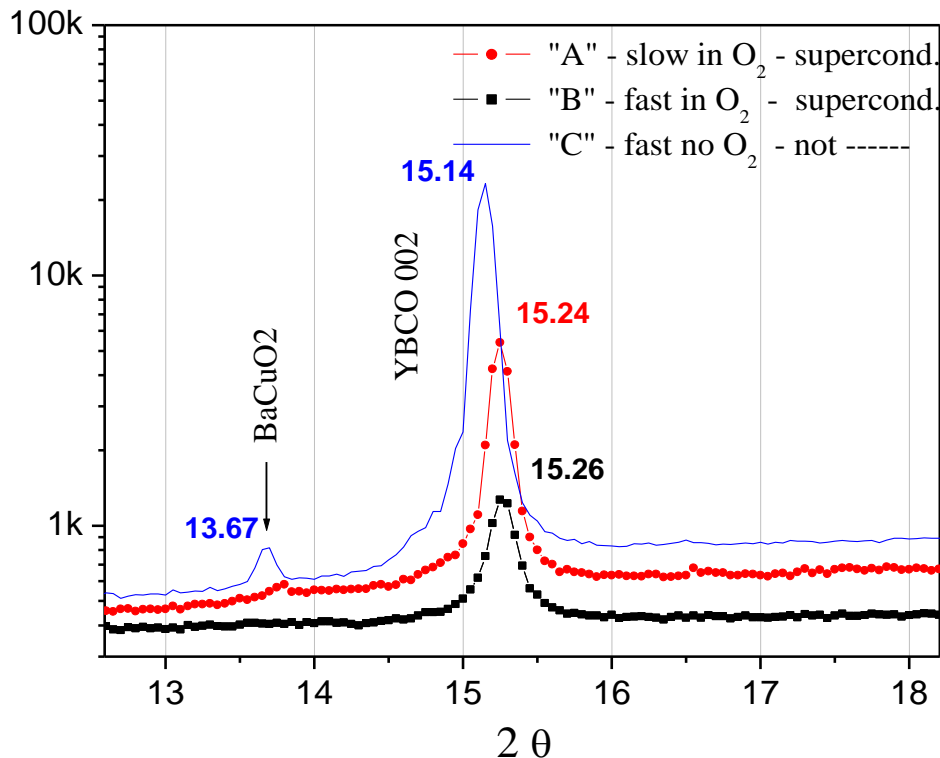
- XRD and Rocking curves;
- RBS in random and channeling geometry;
- Synchrotron scattering X-ray study.

*Spin-polarized electron injection device*



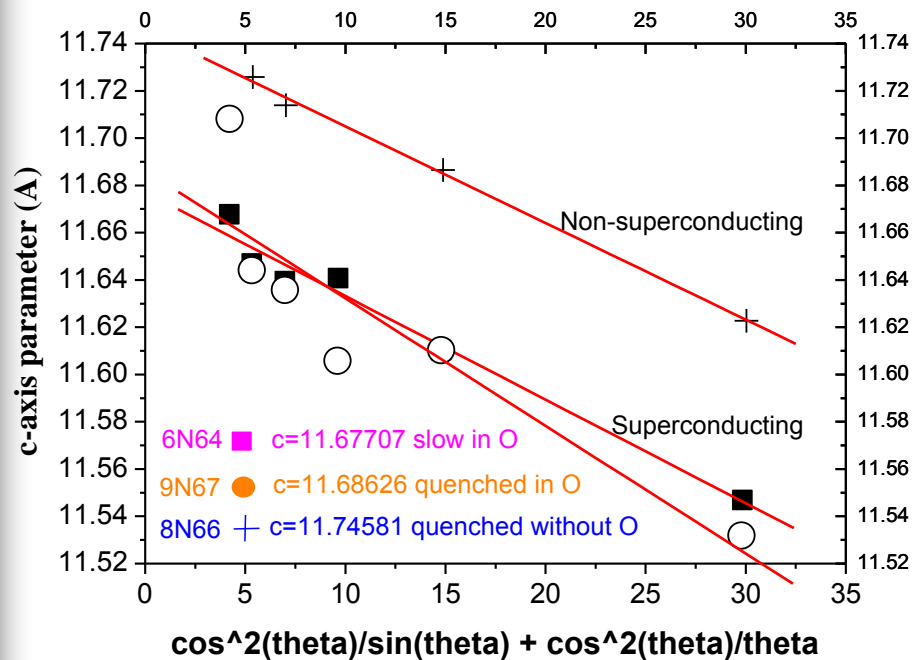
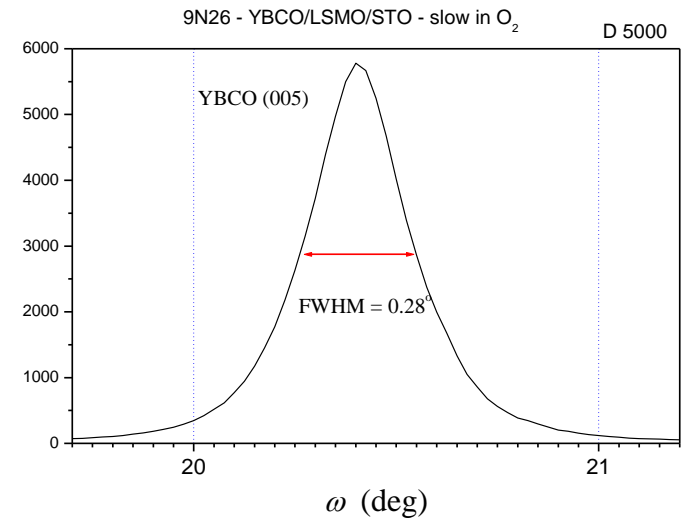
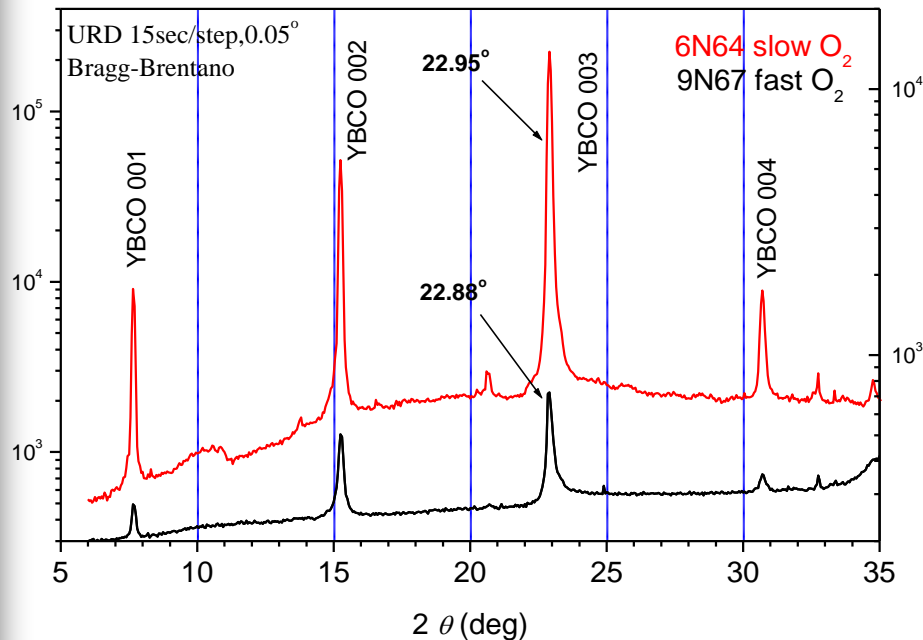
In order to study the relative oxygen content three types YBCO/STO structures were deposited by varying parameter oxygen pressure and annealing time:

1. "Sample A" annealed slowly (1h) in oxygen pressure of 700 mbar;
2. "Sample B" annealed quickly (20 min) in the same ambient;
3. "Sample C" has been annealed without oxygen supply.



	$2\theta$	"c"
↑	15.14	11.69
	15.24	11.62
	15.26	11.60
↓		

Zoomed XRD spectra of YBCO (002) peak for the three types structures -YBCO/STO

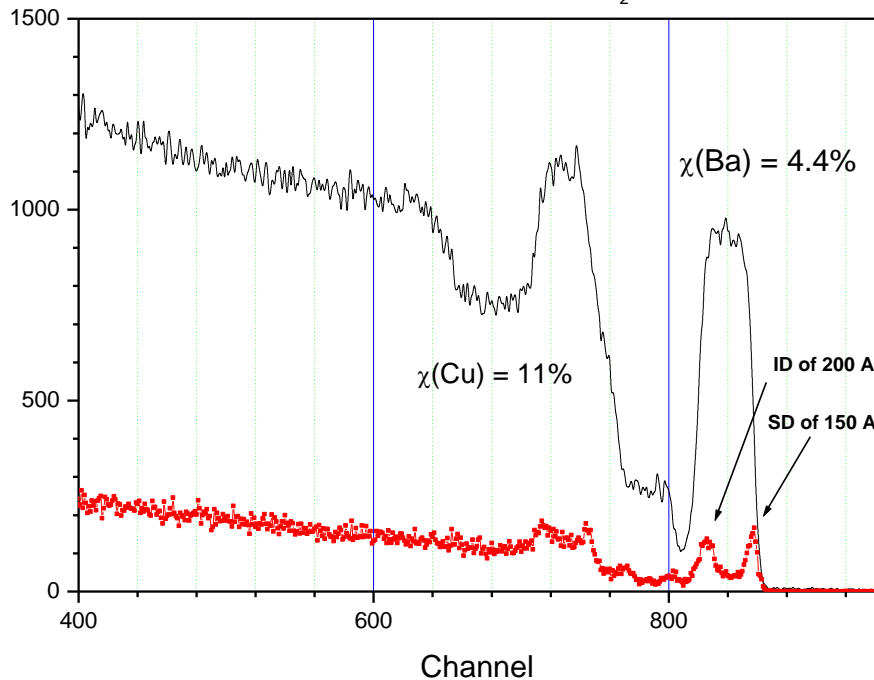


■ The YBCO films have their (a,b) planes parallel to the STO (100), as a result of the presence of well pronounced (ool) peaks.

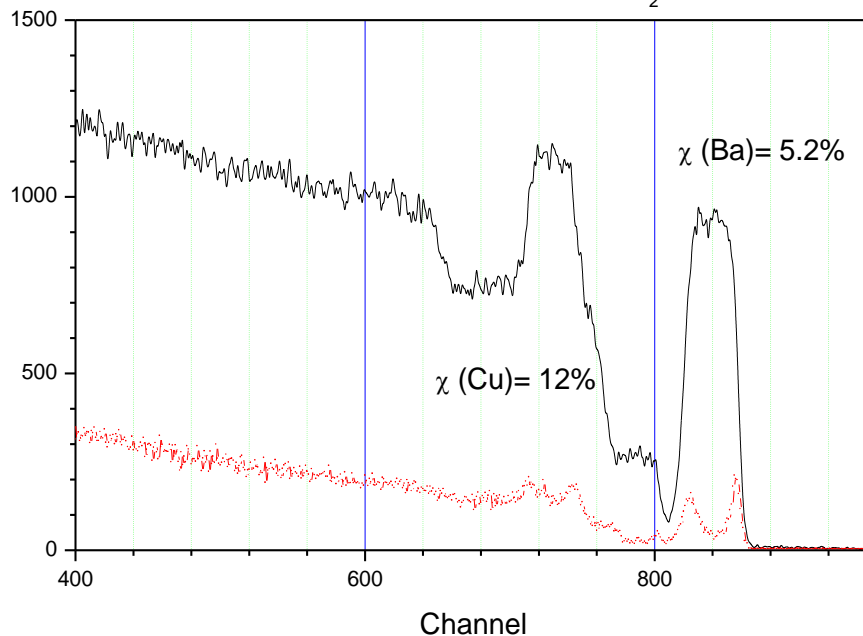
■ Highly texture growth evidenced by (005) YBCO rocking curve. FWHM in the range of 0.2° to 0.3°.

■ The difference in the “c” lattice parameter reveal difference in the oxygen content.

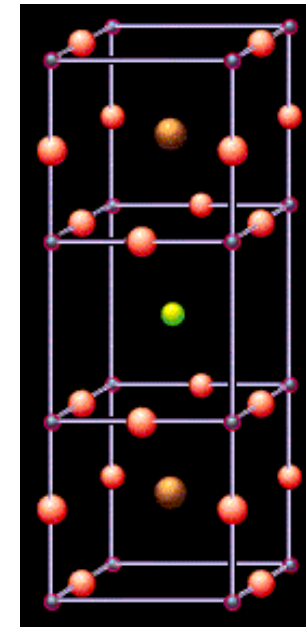
6N64 YBCO/STO slow in O<sub>2</sub>



9N67 YBCO/STO fast in O<sub>2</sub>

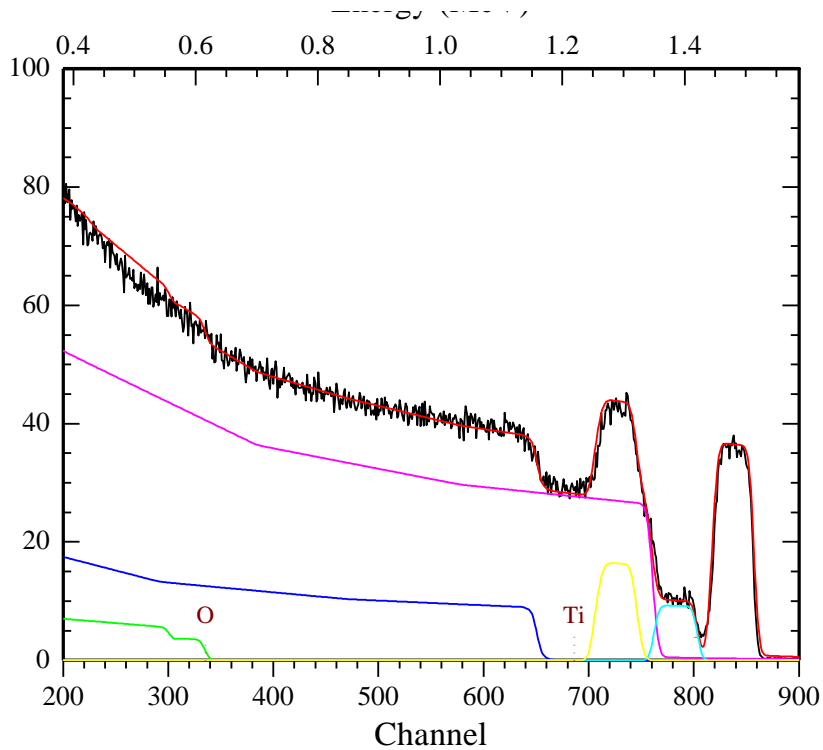


↑  $\chi$  - (weaker channeling) - c ↑ O ↓



- The precise  $\chi_{\min}$  values measured on YBCO films with the same “c”-axis angular spread ( $\sim 0.1$ -  $0.8^\circ$ ) depend upon the oxygen content. In fact, higher  $\chi_{\min}$  values are observed on the films having a higher value of “c” parameter – characteristic of low oxygen content !
- High crystalline quality – near  $0^\circ$  epitaxial growths.

# “Sample A” - RBS – high precision simulation



**La1:**

**Thick 600 A**

**Comp: Y 1.1 BA 1.85 CU 3 O 7 AR 0.02**

**La2:**

**Thick 500 A**

**Comp: Y 1.1 BA 1.85 CU 3 O 7 AR 0.12**

**La3:**

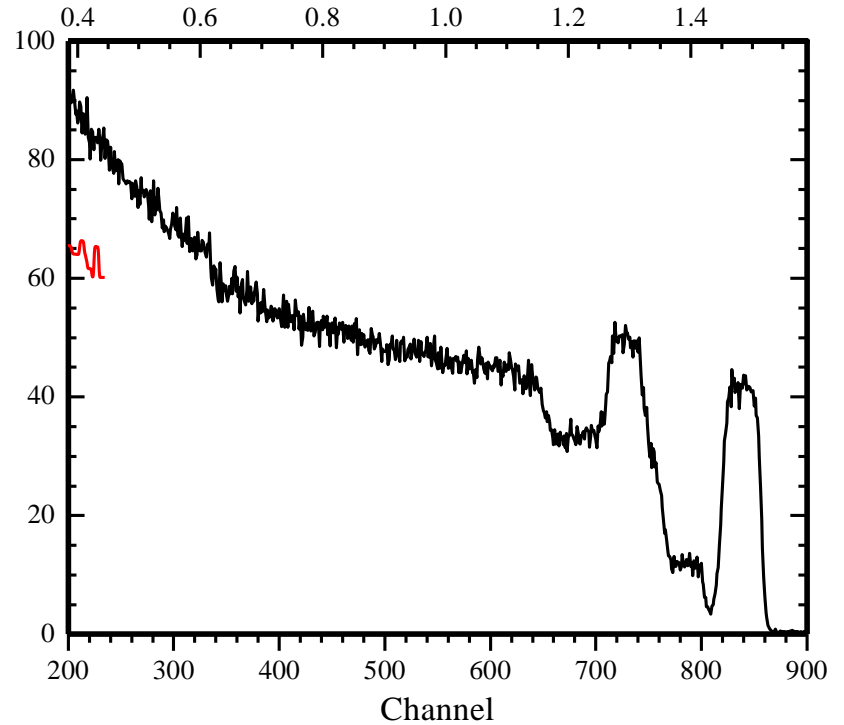
**Thick 10000 A**

**Comp: O 3 SR 1 TI 1**

6N64ra YBCO/STO slow in oxygen - superconductor

# “Sample C” – RBS - high-precision simulation

Relative film stoichiometry for YBCO/STO quenched in vacuum in different depths as deduced from RBS

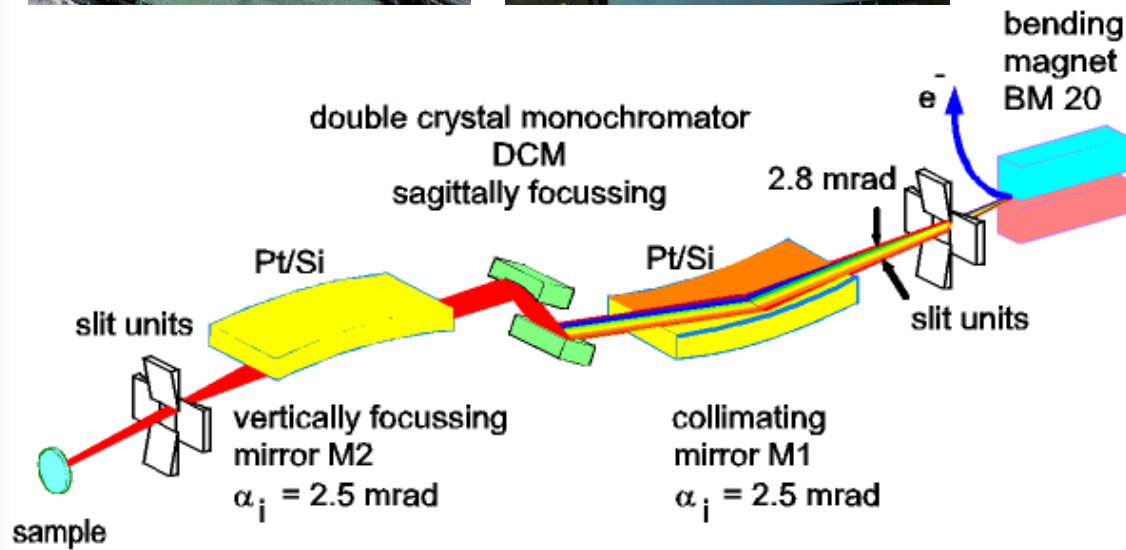
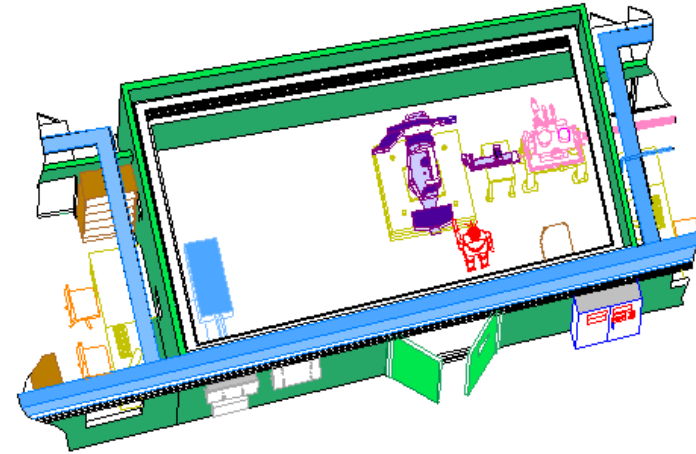
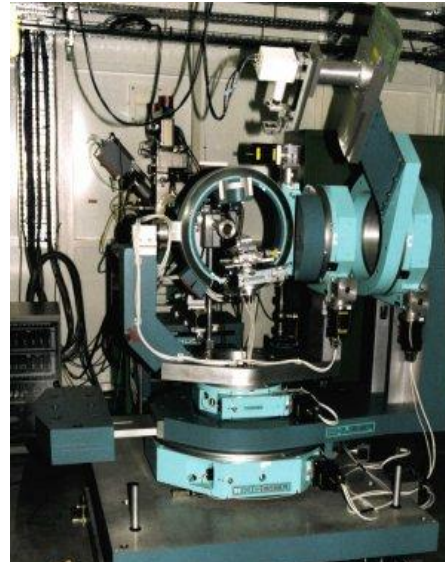
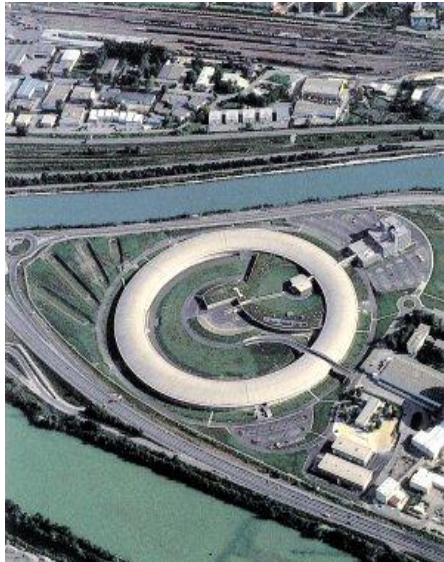


Depth [nm]	Y	Ba	Cu	O	C	Sr	Ti
100	1.1	1.8	3	6	0.4	-	-
26	1	1.8	2.8	6	-	-	-
14	-	0.6	1.8	2	1.6	1	0.2
8	-	0.8	1	3	1	-	-
10000	-	-	-	3	-	1	1

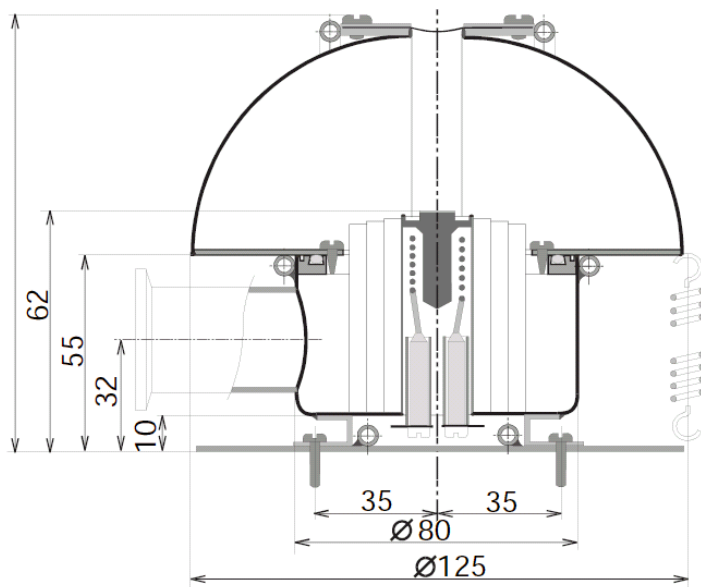
About 20 nm thick interface layer of BaCuO<sub>2</sub> is formed when the oxygen supply is not sufficient for the SC phase formation.

# Synchrotron radiation experiment

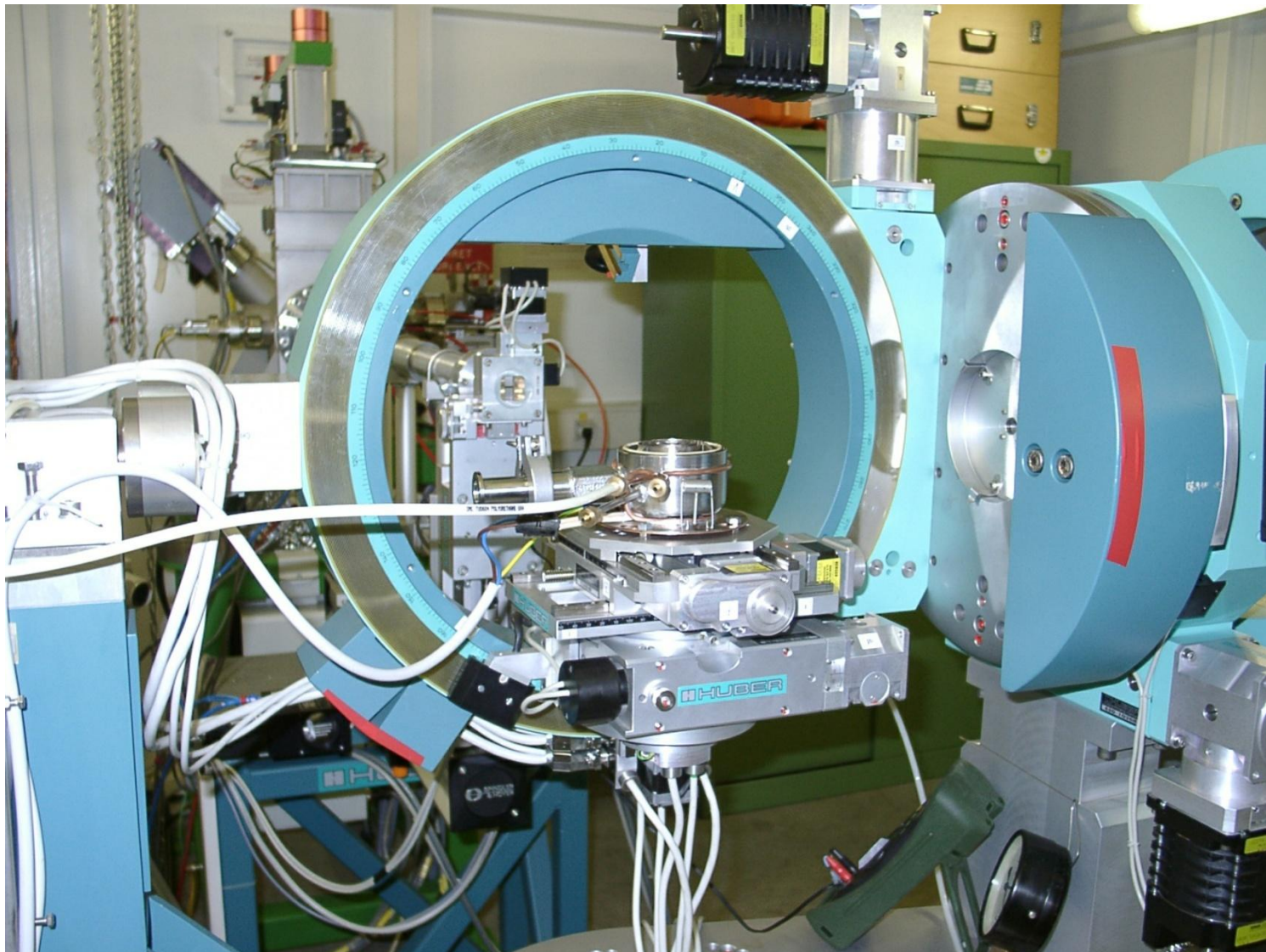
ROBL at ESRF - Grenoble



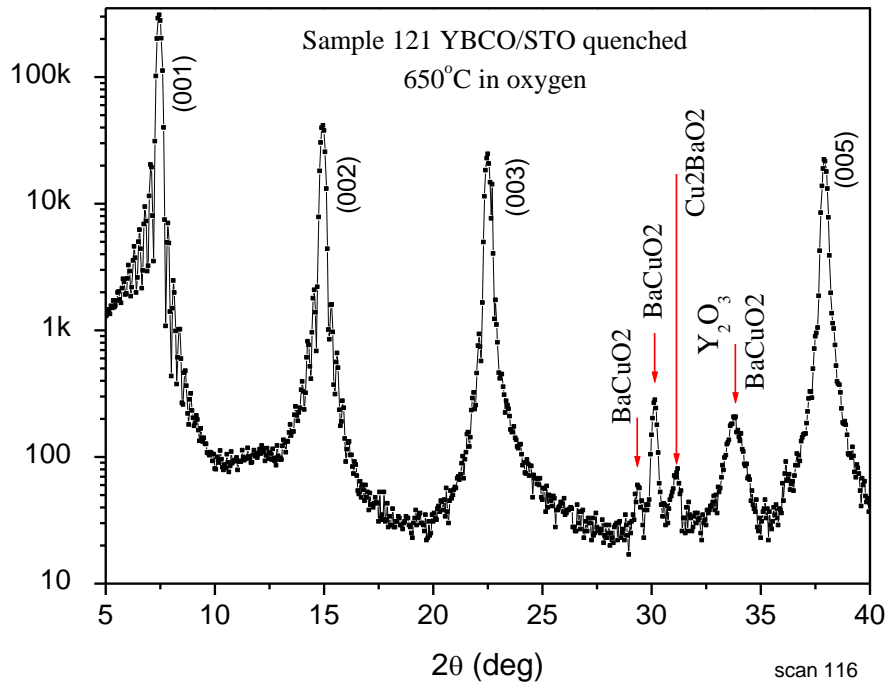
Measured structure --- 40 nm quenched in vacuum YBCO deposited on SrTiO<sub>3</sub> (STO) [100] substrate.



# The Goniometer



Scan 106



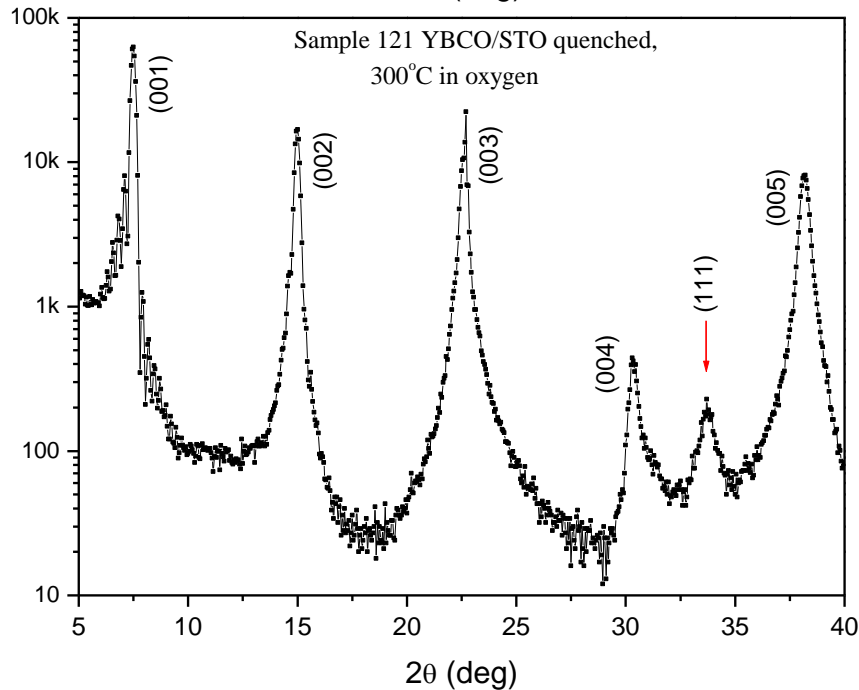
Other phases:

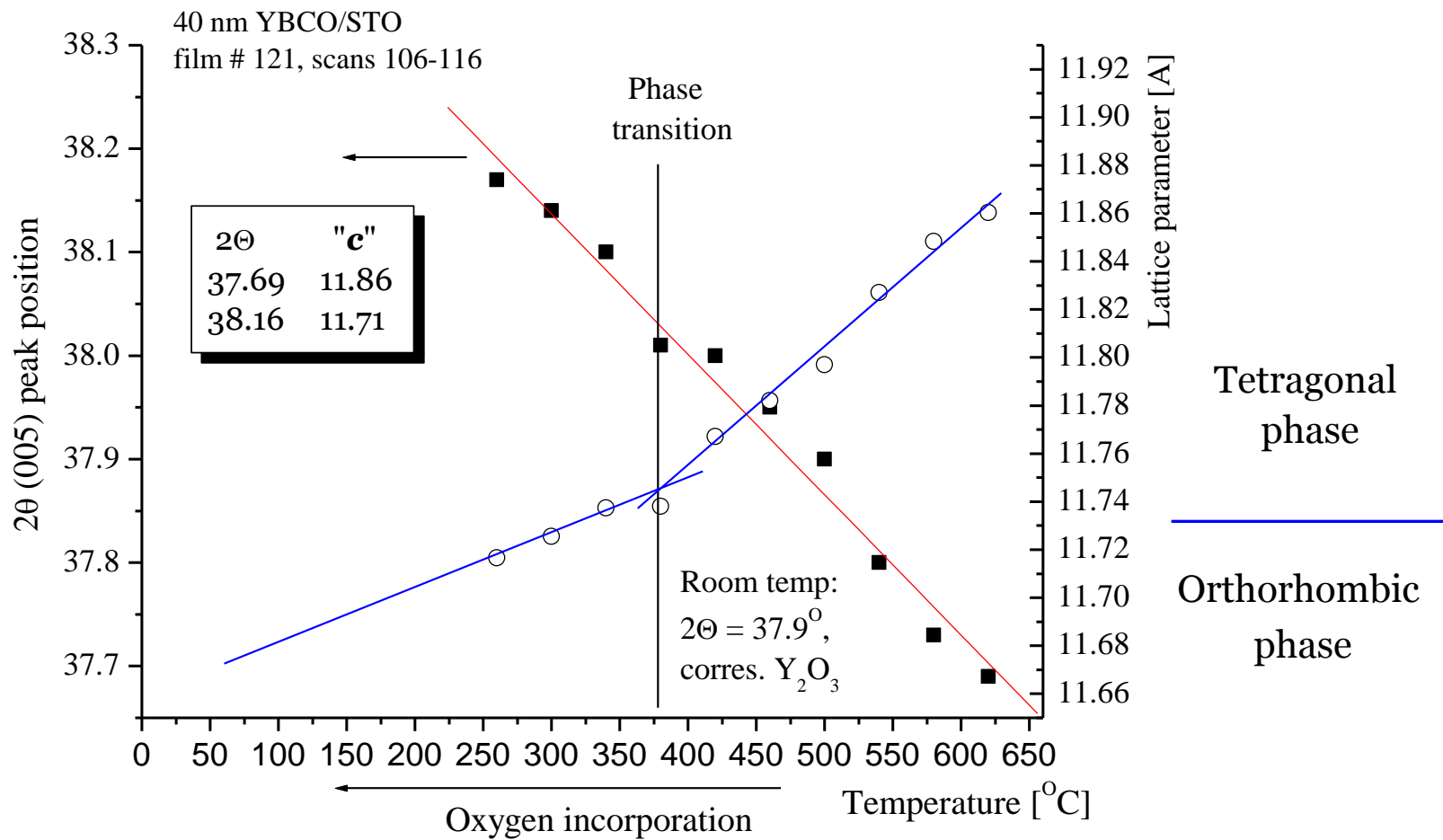
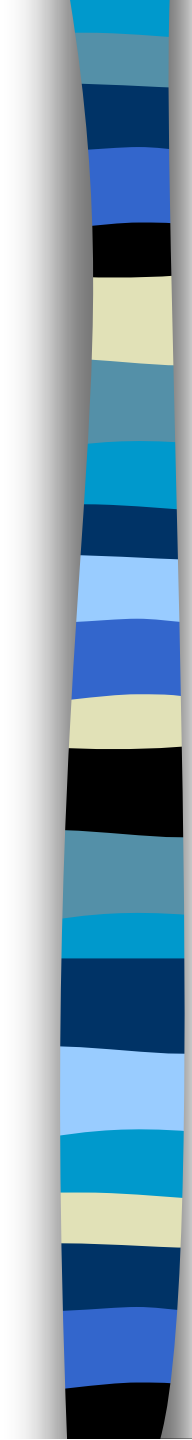
Y<sub>2</sub>O<sub>3</sub> - bcc;

BaCuO<sub>2</sub> - cubic

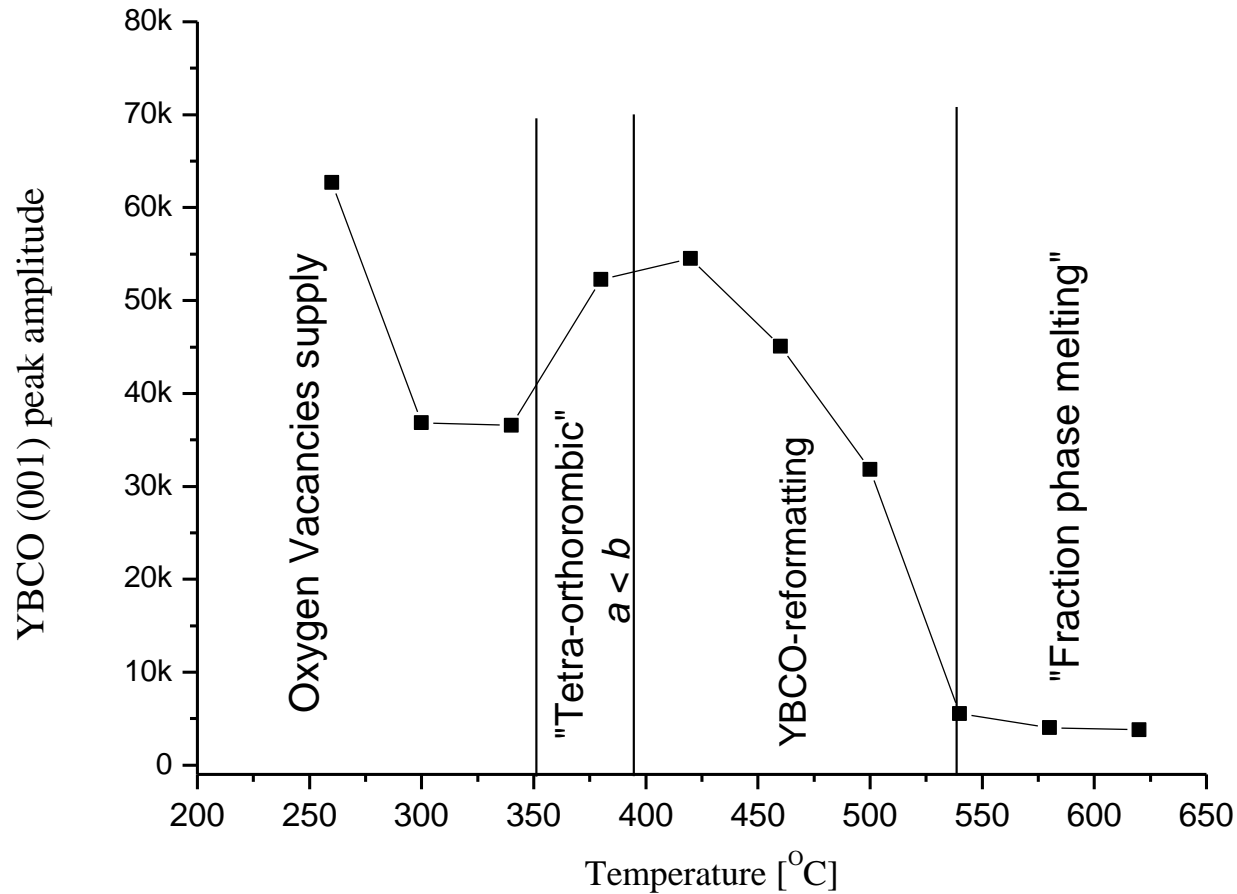
Cu<sub>2</sub>BaO<sub>2</sub> - tetragonal

scan 116





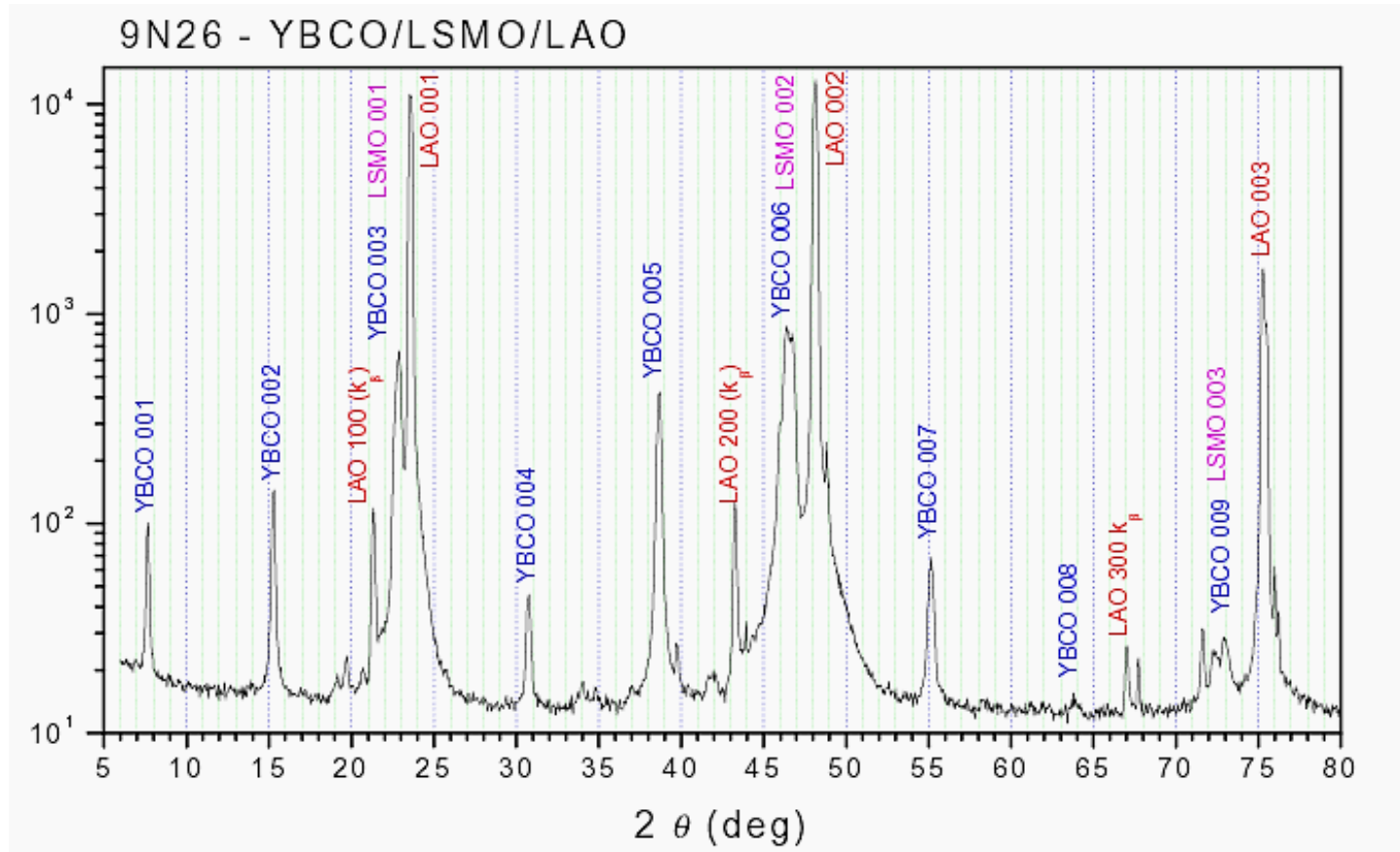
## Phase dynamics



If X-ray scans are acquired at very grazing angle at specular mode a regular undulating spectrum appears known as **Laue fringes**. The repetition frequency is directly related with the film thickness and it is typical for high crystalline film quality. Such Laue fringes are observed in the studied sample # 121 YBCO/STO and they begin to clearly appear after scan # 113, or below 380°C.

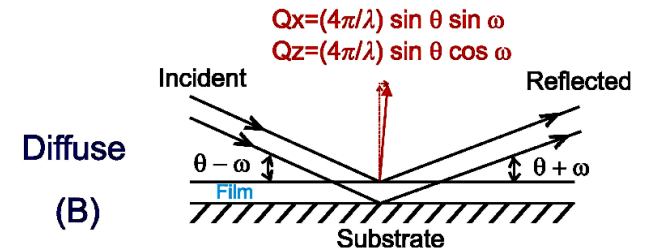
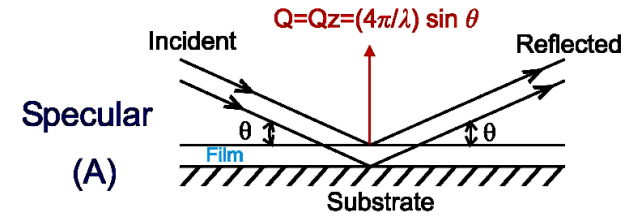
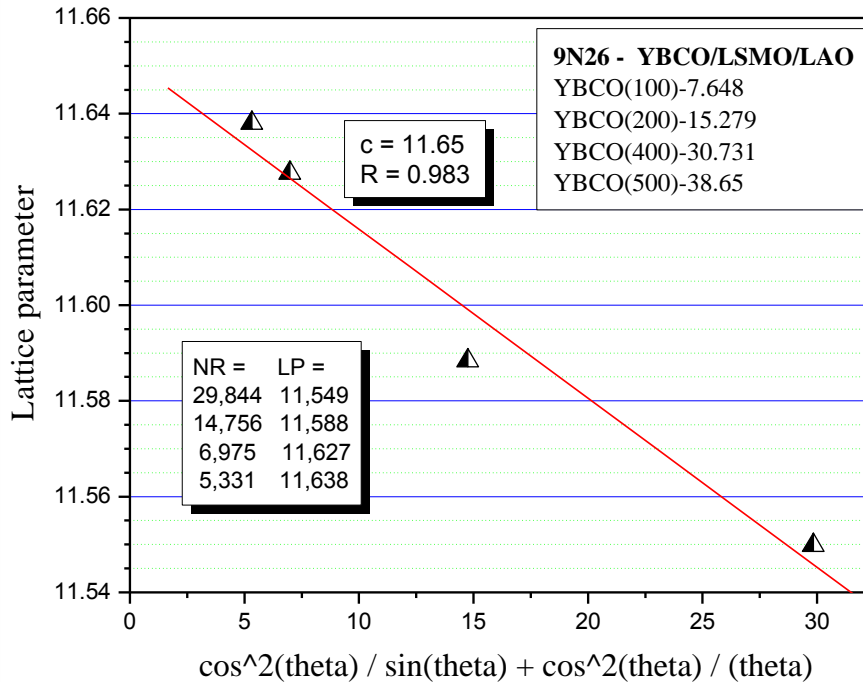
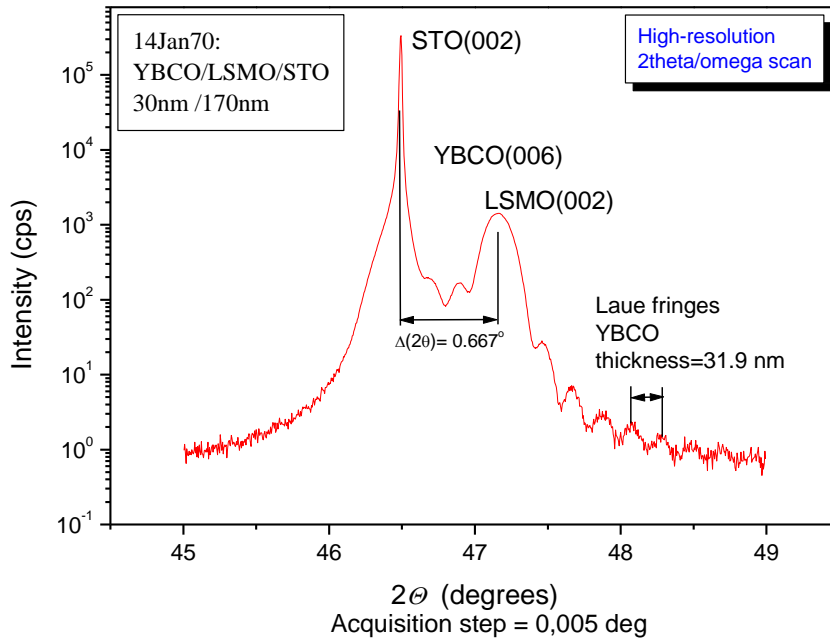
## Measured structure - YBCO/LSMO/LAO

The LAO (002) at R.T. peak is visible at  $2\theta = 47, 86^\circ$ . If LAO lattice is  $a = 3.778 \text{ \AA}$ , the synchrotron radiation is  $\lambda_{R0} = 3.778 * \sin(47.86/2) = 1.5324 \text{ \AA}$



*YBCO- slowly cooled in oxygen - superconducting*

The reverse deposition order (LSMO on top of YBCO) renders YBCO non-superconducting. During LSMO sputtering the oxygen partial pressure is not high enough and the YBCO film deoxygenates.



## X-ray Reflectivity



Specular reflectivity data are analyzed to determine the depth dependence of the electron density of the material of interest.

In diffuse or off-specular reflectivity measurements of the intensity as a function of  $Q_x$  allow a determination of the of surface or interface roughness.



## Conclusions

The process of the phase transitions in HTSC YBCO thin film were studied “*ex-situ*” by means of Real Time Synchrotron Radiation.

A special set-up was constructed for this purpose to fit the goniometer of the SR-XRD experiment and to operate at high temperature in oxygen atmosphere.

The analyzed dates show by two independent ways the exact ranges of phase existing and phase transitions.

High-precision RBS spectra taken in channeling and random geometry confirm also the existence of these phases.