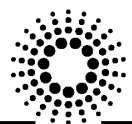


## Experiment Report Form

 <b>ESRF</b>	<b>Experiment title:</b> Structural study of new phases of Bismuth-layered perovskites doped with rare earth.	<b>Experiment number:</b> 25-1-717
<b>Beamline:</b> BM25A	<b>Date of experiment:</b> <u>CRG-beam time</u> from: 12/03/2009 at 8:00                      to: 16/03/2009 at 8:00	<b>Date of report:</b>
<b>Shifts:</b> 12	Local contact(s): <b>Dr. Pilar FERRER</b>	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants</b> (* indicates experimentalists): - *Pilar Ferrer, ferreres@esrf.fr - *Alicia Castro, acastro@icmm.csic.es - *Teresa Hungría, thungria@icmm.csic.es - *Inmaculada Martínez, inma@icmm.csic.es		

### **PRELIMINARY REPORT:** (*supporting request of a new proposal*)

*Further results and figures will be included in the final report (once results have been submitted for publication)*

The aim of this experiment was to solve the atomic structure of several solid solutions with  $\text{Bi}_{4-x}\text{R}_x\text{SrTi}_4\text{O}_{15}$  formula, where R is a rare earth (La, Pr, Nd, Sm, Gd, Dy, Er and Yb). These materials belong to the laminar structure family called Aurivillius. The R(III) cation substitutes the Bi(III) cation which could be in two different chemical environments. From all the samples prepared, the La and Pr doped solid solutions were measured.

These structures have an interesting reflections family at  $d \sim 2.72$  and  $1.363 \text{ \AA}$  (the (200) - (020) and (400) - (040) reflections, respectively), which show the tetragonal/orthorhombic grade. Previous works had provided evidences of the orthorhombic character thus the split exists at low x values of doping.

The  $\text{Bi}_{4-x}\text{La}_x\text{SrTi}_4\text{O}_{15}$  family was measured at 15 KeV, providing information from reflections at angles not allowed by using a conventional X-ray diffractometer, which will help us in the structure solution procedure. However, the difference in  $2\theta$  between the reflections mentioned before was very small, so the tetragonal-orthorhombic problem was not solved clearly (see Fig. 1). Also, other reflections observed at high  $2\theta$  angle could show this symmetric relation, but the presence of another reflections in those zones made undistinguished the ones looked for.

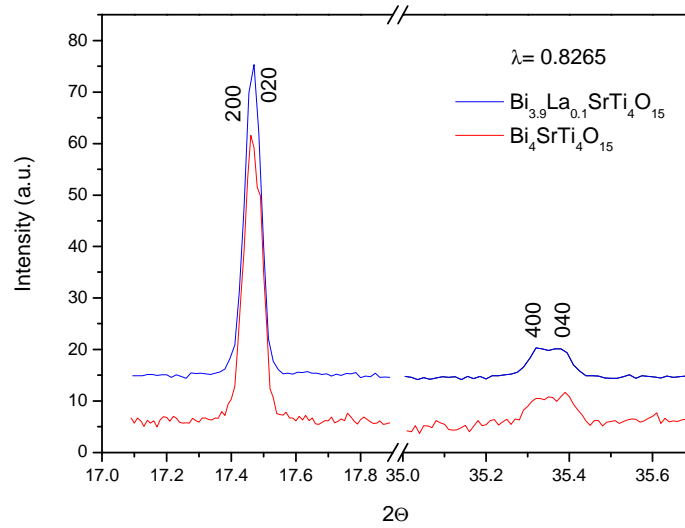


Figure 1. X-ray diffraction pattern of  $\text{Bi}_4\text{SrTi}_4\text{O}_{15}$  and  $\text{Bi}_{3.9}\text{La}_{0.1}\text{SrTi}_4\text{O}_{15}$  phases measured at  $\lambda = 0.8265 \text{ \AA}$ .

To improve the resolution we decreased the energy at 12 KeV, obtaining better results. The reflexions (400) and (040) can not be well distinguished when  $x$  increase. However the FWHM shows a logical decrease due to the lack of orthorhombicity character when  $x$  value increases (see Figure 2 and table 1 for  $x_{\text{La}} = 0, 0.2, 0.5$  and  $0.8$ ).

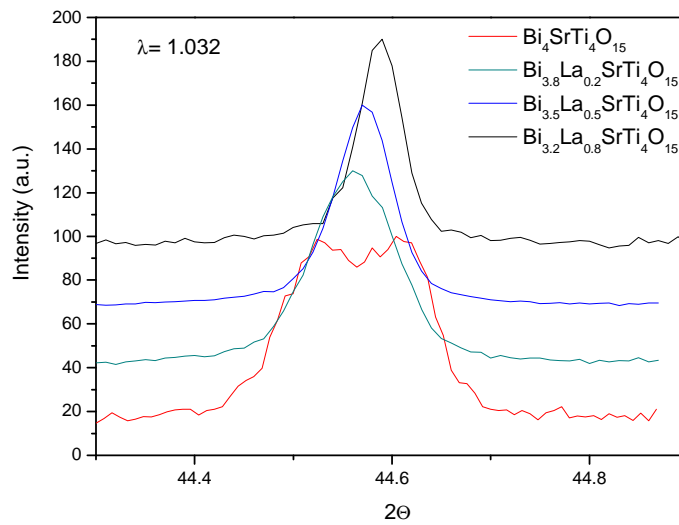


Figure 2. X-ray diffraction pattern of the phases  $\text{Bi}_{4-x}\text{La}_x\text{SrTi}_4\text{O}_{15}$  with  $x = 0, 0.2, 0.5$  and  $0.8$  at  $\lambda = 1.0323 \text{ \AA}$ .

Table 1. FWHM of the phases  $\text{Bi}_{4-x}\text{R}_x\text{SrTi}_4\text{O}_{15}$  with  $x = 0, 0.2, 0.5$  and  $0.8$  (at  $\lambda = 1.0323 \text{ \AA}$ ).

x value ( $\text{Bi}_{4-x}\text{La}_x\text{SrTi}_4\text{O}_{15}$ )	FWHM
0	(two peaks) $\sim 0.06$
0.2	0.10
0.5	0.07
0.8	0.05

Another way to know the structural environment of the different rare earth in the solid solutions is the *Extended X-Ray Absorption Fine Structure* technique. Thus, next round we are going to apply for beamtime at the XAS station in BM25A in order to complete the study.