	Experiment title:	Experiment number:
ESRF	In-situ direct observation of phase and texture evolution in Ag-sheathed Bi,Pb(2223 tapes, by high energy synchrotron radiation diffraction.	HS-829
Beamline: ID15	**Date of experiment**: from: 23-avr-99 to: 27-avr-99	**Date of report**:
Shifts: 12	**Local contact(s)**: Veijo Honkimäki (PLUO C)	*Received at ESRF:*

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Report:

The large efforts made to optimize the processing procedures of Ag-sheathed $(Bi,Pb)_2Sr_2Ca_2Cu_3O_\delta$ high temperature superconducting tapes have lead to critical current density values approaching the level needed for technological applications and established this compound as the most suitable one for use in superconducting devices at liquid nitrogen temperature. To procede beyond the present status, it is necessary to better understand the mechanisms responsible for the $(Bi,Pb)_2Sr_2Ca_2Cu_3O_\delta$ phase formation inside the Ag sheath and for the texture development. I*n-situ* diffraction experiments [1, 2, 3] turned out to be a powerful technique to investigate this topic and to achieve further improvement of Bi,Pb(2223) tapes performances. Neutron powder diffraction [2] and synchrotron X-rays diffraction, at intermediate- [3] as well as at high-energy [1], have been successfully used to study the phase formation process, to quantify the secondary phase composition and their role, to investigate the development of the preferred orientation of the superconducting platelets inside the Ag-sheath.
The present experiment allowed for an *in-situ* high-temperature study of the texture inside the Ag-sheath. The grain alignment was observed to take place already in the precursor $(Bi,Pb)_2Sr_2Ca_1Cu_2O_x$ phase during the first heat treatment. The effect of the secondary phases on the texture development was investigated. As the phenomena occurring in the precursor $(Bi,Pb)_2Sr_2Ca_1Cu_2O_x$ can strongly affect the final properties of the Bi,Pb(2223)-Ag tapes, the experiment focussed mainly on the behaviour of the precursor phases prior to react to form the $(Bi,Pb)_2Sr_2Ca_2Cu_3O_\delta$ phase.

In a second experiment, the effect of the intermediate mechanical deformation of the tapes was investigated: the re-crystallisation of the $(Bi,Pb)_2Sr_2Ca_2Cu_3O_8$ grains and the texture recovering were measured as a function of the temperature and time.
Several samples, which experienced different mechanical deformation processes were compared.
Two kinds of 2D-detectors were used, namely a CCD (Charge-coupled Device) Camera coupled to a fluorescent screen and an Imaging Plate. The advantages of the latter are a better resolution, a more feeble distortion of the diffraction patterns and the absence of large pixel area saturation close to the strong Ag reflections. Therefore, the Image Plate revealed to be more suitable to reduce the background and to better study the diffraction arcs profile for the texture analysis. On the contrary, owing to the longer exposition time needed for the Image Plates, for a real time acquisitions the use of the CCD Camera is recommended. By using the FIT2D software 2θ profiles at fixed ϕ, ϕ-profiles at fixed θ and θ–2θ patterns integrated over ϕ were analysed.
A dedicated X-ray compatible furnace was fabricated for the present experiment [4].
The secondary phase Ca_2PbO_4, has been found not to have any preferred orientation during the Bi,Pb(2212) texturing. The Bi,Pb(2212) texturing has been found to sharply occur at 750°C.
The different final texture due to the different intermediate mechanical deformation does not seem to affect the final critical current of the tapes, being the grain link rather than their relative orientation the most important parameter affecting the transport properties.

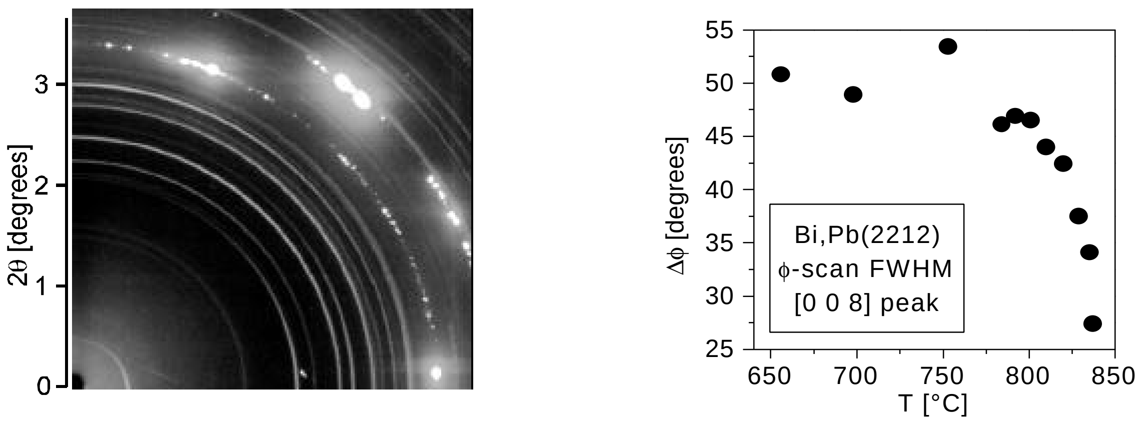

Figure 1. **(left):** 2D diffraction pattern from an Image Plate, in the range $0<\phi<\pi/2$. This picture shows the very good image quality provided by this detector. **(right):** Bi,Pb(2212) texture development during heating from *in-situ* diffraction using an IP detector. The [008] reflection was chosen because it is far from the large Ag spots.

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[2] E. Giannini, E. Bellingeri, R. Passerini and R. Flükiger, *J. Appl. Phys.*, submitted.
[3] L.-J. Wu, Y.L. Wang, W. Bian, Y. Zhu, T.R. Thurston, R.L. Sabatini, P. Haldar and M. Suenaga, *J. Mater. Res.*, 12 (11) 1997, 3055.
[4] E.Giannini, E.Bellingeri, F.Marti, M.Dhallé, V.Honkimäki, R.Flükiger, *Int. J. Mod. Phys. B*, at press.