ESRF	**Experiment title:** **Anomalous Diffraction Interest in Texture Analysis**	**Experiment number:** MI 122
Beamline: BM 16	**Date of experiment:** from: 20/11/96 to: 24/11/96	**Date of report:** 28/2/97
Shifts: 15	**Local contact(s):** Eric DOORYHEE*	*Received at ESRF:* 28 FEV. 1997

Names and affiliations of applicants (* indicates experimentalists):

Daniel CHATEIGNER*, Jean-Louis HODEAU*, Pierre BORDET*, Michel PERNET*: *Lab. de Cristallographie, BP1 66 38042 Grenoble, France*

Michael THOMS: *ESRF, BP220 38042 Grenoble, France*

Jitka VACINOVA: *Fac. of Nuclear Science and Pfys. Eng., Brehova 7,115 19 Praha, Czech Republic*

Over many years calculations of the Orientation Distribution Function (ODF) has been achieved from experimental pole figures using the generalized spherical harmonic description developped by Bunge [1]. If particularly adapted to centrosymmetric cubic crystal systems, this description however becomes of high complexity for lower symmetries and has never been extended so far except for the hexagonal case. Furthermore, the Fourier coefficient development of the harmonic terms is restricted to the even orders [2], which makes them unable to deal with non-centric crystal systems. More recently Matthies [3] proposed an iterative approach, now called the WIMV method, with which the OD (no more function is used to fit the data) of all the crystal symmetries can be refined. The problem of solving the OD in the case of non-centric structure still remains, but could be solved using anomalous scattering [4]. However it has never been tested up to now, the main reason being that not enough powerful1 X-ray sources existed.

The aim of this experiment was to verify the feasibility of such anomalous texture analysis. We then chosed non-centric materials, so-called PZT ferroelectric films, 1 μm thick, deposited on Pt/Ti/(100)-Si, with gold dots used to polarize the films for orienting the ferroelectric domains. We worked in transmission mode, with a beam of 0.5x0.5 mm2, and an on-line image plate reader developped by one of us (M.T.). We decided first to reconstruct a 'normal' pole figure, by taking 15 patterns at different orientations of the sample, which gave enough orientation space coverage to calculate the OD. The employed technique here is the equivalent for image plates of the one developped for CCD acquisition recently [5]. Figure 1 shows an original 2D pattern with phases indexing (left), the 111 pole figure of PZT calculated from 15 patterns

(center), and the recalculated pole figure (right) which shows a satisfying recalculation (good OD refinement) and the completed experimental blind area (because of geometry) where the main 111 pole is located (normal to the film surface). We have to note that only one ring, the 111, was used to calculate the OD, but the other rings will be used in a deeper data treatment.

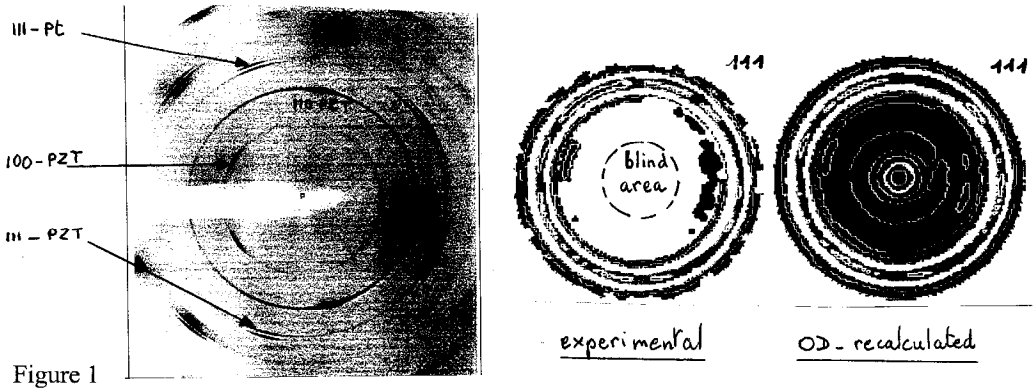

Figure 1

In a second approach we concentrated on observing a possible anomalous signal. Around the Zr edge, the maximum anomalous effect was calculated to be on the 100 and 110 rings, enlarged because of a tetragonal deformation. The difference of ring intensities between two energies, just below and just after the Zr edge, is due to volume/absorption/fluorescence variation on one hand, and to anomalous scattering variation on the other hand. Figure 2 shows the developped 110 ring of PZT, for 18.1 and 17.9 keV. The predicted overall intensity variations are observed, but are not homothetic along all the rings as it should have been observed (on a log scale only) if only volume/absorption/fluorescence variations have occured. We think the slight non homothetic variations are then due to anomalous scattering. This would be of considerable importance for measuring the full non-centric OD, thought the orientation of non centric domains, and we will consequently propose other experiments in order to combine the two here used procedures.

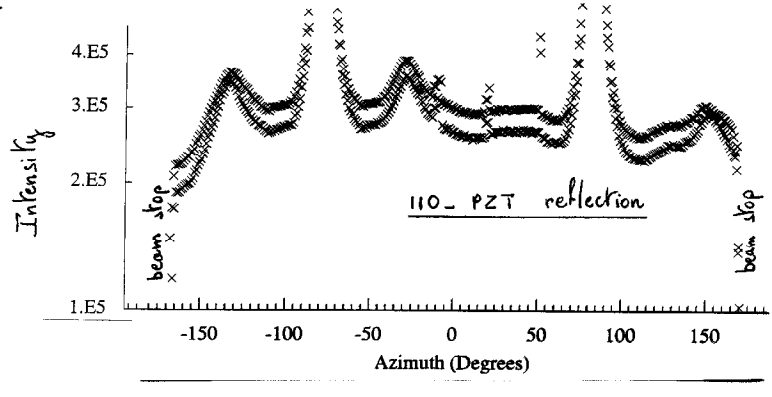

Figure 2

[1]: Bunge HJ, Z. Metall. 56, 872 (1965); [2]: Matthies S & Helming, Phys. Stat. Sol. B 113, 569 (1982);[3]: Matthies S & Vinel G, Phys. Stat. Sol. B 112, K111 (1981); [4]: Bunge HJ et al., J Appl. Cryst. 13, 544 (1980); [5]: F Heidelbach & C Riekel, ESRF news 1997.