ESRF	**Experiment title: X ray holography on MBE grown FePd alloys**	**Experiment number:** MI-174
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Report:

The aim of the experiment was twofold:

- start developing the X-ray Fluorescence Holography (XFH) method at ESRF
- find experimental conditions allowing to obtain a local holographic image of MBE-grown FePd thin films

Since hard x-ray holography is not by far a routine technique, in the first part of the beamtime we worked on the optimisation of the experimental arrangement. For this purpose we used a NiO[l 1 l] single crystal as the reference sample. This crystal with unusual orientation, just starting to be used for metal-insulator interface studies, was primarily chosen here because the Ni-K emission line is close to the Fe one (resp. 7.4 and 6.4 keV); in addition, the large mosaic spread (- 1 deg.), similar to the MBE-FePd one, is very well matched to the XFH geometry. This allowed us to check the energy resolution and the detected spectrum.

Since the detection system is a key part of the XFH method, we tried 3 types of detection :

- APD detector alone (with its energy discrimination electronics)
- a sagittally bent crystal analyser (XTA), with 1 $^{\circ}$mosaic pyrolitic graphite (PG) + the APD
- 3 identical XTAs in a doubly focusing arrangement [l] + the APD

The experiments were done both in the normal and inverse XFH modes. Continuous 2D-scans were performed by rotating the detector arm over nearly 2π, the sample being fixed in XFH, and rotating together with the detection systems around the same axes (elevation θ, azimuth ϕ).

<u>Results</u> Direct APD detection gave high fluorescence count rates (a few MHz), however high energy background including several pronounced Bragg peaks appeared in the fluorescent channel. Further a slight systematic variation of the intensity appeared in the ϕ-scans (i.e. rotations around the sample normal). These

[l] the triple XTA was designed at ESRF for the XFH experiment with the Optics Group and ID32 beamline.

Background features showed up in both type XFH modes. The second detection (i.e. interposing one XTA) suppressed most of the background, so that the Bragg peaks almost completely disappeared; however, a more pronounced intensity variation in the +-scans was observed. The third arrangement added a factor 2-3 in intensity; the ϕ-scans variations contained however a higher spatial frequency than in the previous case. These results are summarised in fig. 1 showing the solid angle scans (a. direct APD, b. one XTA), a sort of stepped spiral: ϕ-scans of 180 degrees, with positive and negative θ with θ approaching 0.

In the case of the NiO bulk sample, the fluorescent intensity was high enough to record the 2D-scans in 3-5 hours with sufficient statistical accuracy. However, the systematic ϕ-scans intensity variations (2-15%) prevent a straightforward deconvolution of holographic information - whose level is in the 0.3 % range - moreover because of the presence of a spatial frequency component close to that of the holographic information. The likely cause of this intensity variation is a slight misalignment of the optics on the detector arm. Therefore subsequent realignments of the system were done during the experimental run, resulting in a decreased systematic error. However, we could not go below a 2 % intensity change in the ϕ-scans. We are developing new evaluation procedures which might allow to disentangle these systematic errors from the holographic information. Nevertheless the 3-fold symmetry in the low frequency range, together with the Standing waves/Kossel lines structure shows that the holographic information is indeed present in the data, implying that the detection system is precise enough (i.e. I,, monitor + XTA + detector gave a resolution in the intensity better than 0.3%); the problem of the holographic reconstruction is thus given by the theta direction (elevation angle), which leads to wrong interatomic distances (not the symmetries); this is due to the fact that misalignment and beam motion after refills produces different systematic errors at different θ values.

Although the hologram itself is not available at this level of data treatment, new interesting results have been already obtained on NiO:

1) In the XFH experiments, the Kossel-lines pattern is deduced (c.f. fig.2a). Up to now X-ray experiments in which such pattern was detected are scarce [2], and only on nearly perfect single crystals.

2) In the inverse XFH experiment, a full standing wave line pattern (fig. 2b).

To our knowledge, our data represent the first observation of both patterns on the same crystal. They result from the diffraction of the fluorescent (Kossel) and incident photons (SW) and are related through the optical reciprocity theorem.

In the second part of the experimental run we concentrated on the $Fe_{.5}Pd_{.5}$ thin films. From the experience gained on NiO, an analyser crystal should be used to avoid Bragg peaks, but also the ϕ-scans intensity variation minimised; hence we choose a single XTA. In spite of an Helium path around the XTA, the fluorescent intensity was quite low (15-20 KHz) due to the small quantity of matter involved (100 nm). In order to collect statistically meaningful data, only the more ordered film was measured in the remaining 4 days.

Summary: Various experimental conditions for holographic measurements at ESRF have been tried. Moving the detector on an hemisphere resulted in large intensity variations in ϕ-scans attributed to misalignment in the detection optics. Once solved, NiO holograms can be obtained rather straightforwardly, as our laboratory measurements demonstrate (fig. 3). It is quite promising that even so the Kossel and standing wave patterns of NiO[111] were obtained. The measurement time on the FePd film could be reduced to a few days using the triple XTA and a slightly modified setup.

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[2] H.J. Ulrich et al., NIM A349 (1994) 269

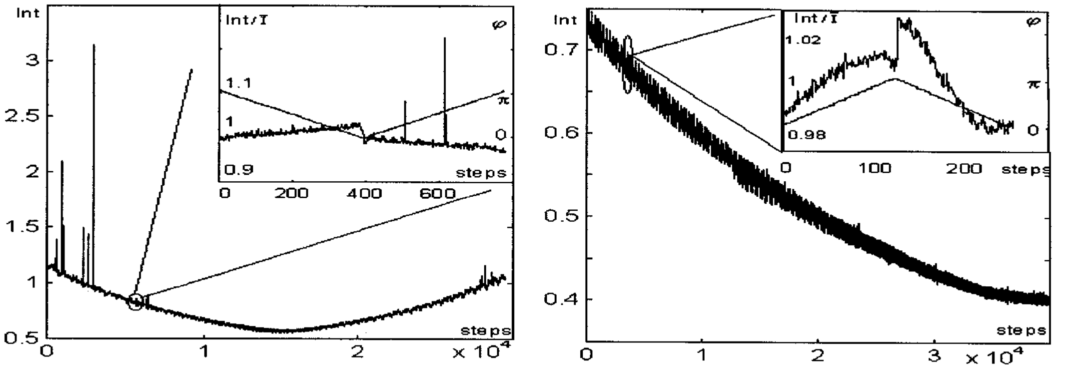

Figure 1. ***Intensity variation in a holographic scan, a sort of stepped spiral: phi (azimuth) scan of*** 180 ***degrees, with positive and negative theta (elevation) with absolute value of theta approaching 0. In the insert it is shown the normalised intensity and phi. a) without crystal analyser a lot of Bragg peaks alter the low energy channel, b) with crystal analyser, the variation in a phi scan is bigger but Bragg peaks are removed.***

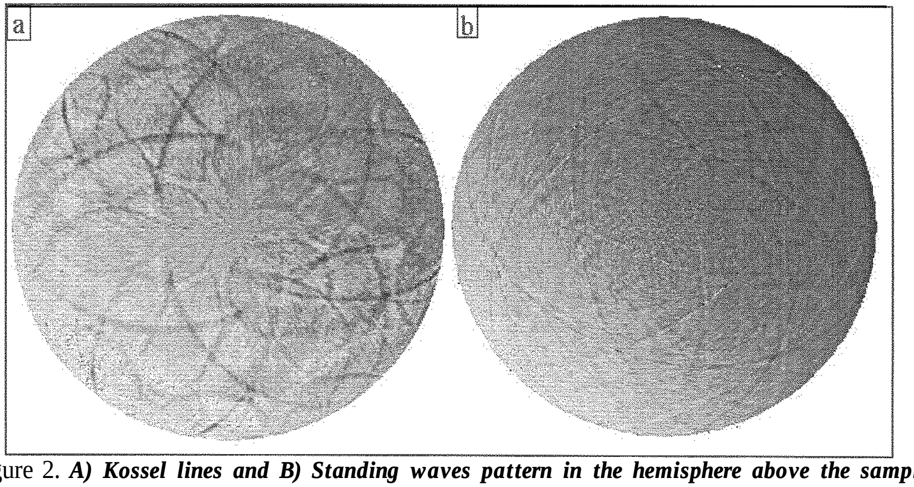

Figure 2. ***A) Kossel lines and B) Standing waves pattern in the hemisphere above the sample***

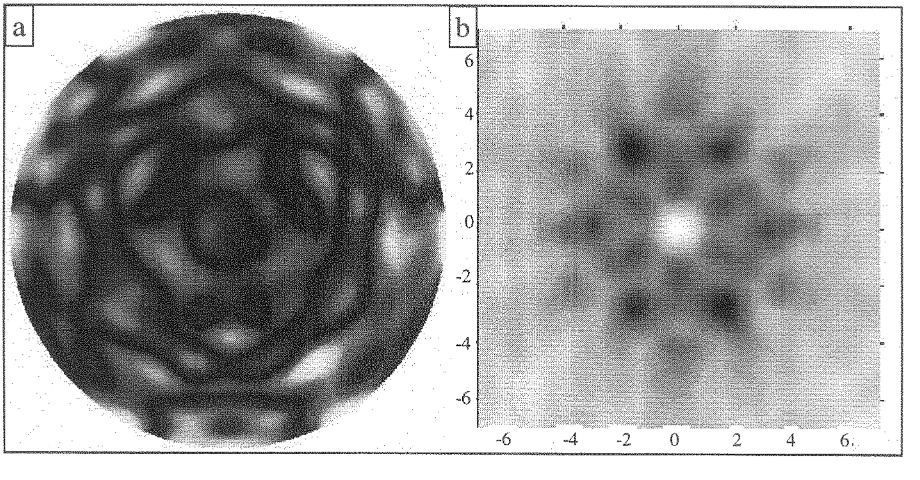

Figure ***3 Hologram (a) and holographic reconstruction (b) in the z=O plane obtained on the same NiO crystal at the laboratory. The dark spots in (b) represent the atoms or their twin images.***