ESRF	**Experiment title:** Oxide Growth on Single Crystals	**Experiment number**: ME 816
Beamline: BM20	**Date of experiment**: from: 13.07.2004 to: 19.07.2004	**Date of report**: 01.09.2005
Shifts: 15	**Local contact(s)**: Dr. Nobert Schell	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Prof. Dr. Walter Reimers, TU Berlin

Michael Skornia*, TU Berlin

Dr. Sascha Dieter*, TU Berlin

Prof. Dr. Anke Rita Pyzalla*, TU Wien

Report:

The aim of the experiment were phase and internal stress analyses on growing oxide layers on iron single and also fine and coarse grained polycrystals.
We performed phase and internal stress analyses both ex-situ on samples oxidised prior to the experiment in the home lab and in-situ during oxidation in a furnace. Starting with the samples oxidised ex-situ we tested both the θ-2θ-geometry and grazing incidence measurements for phase analyses and internal stress analyses.

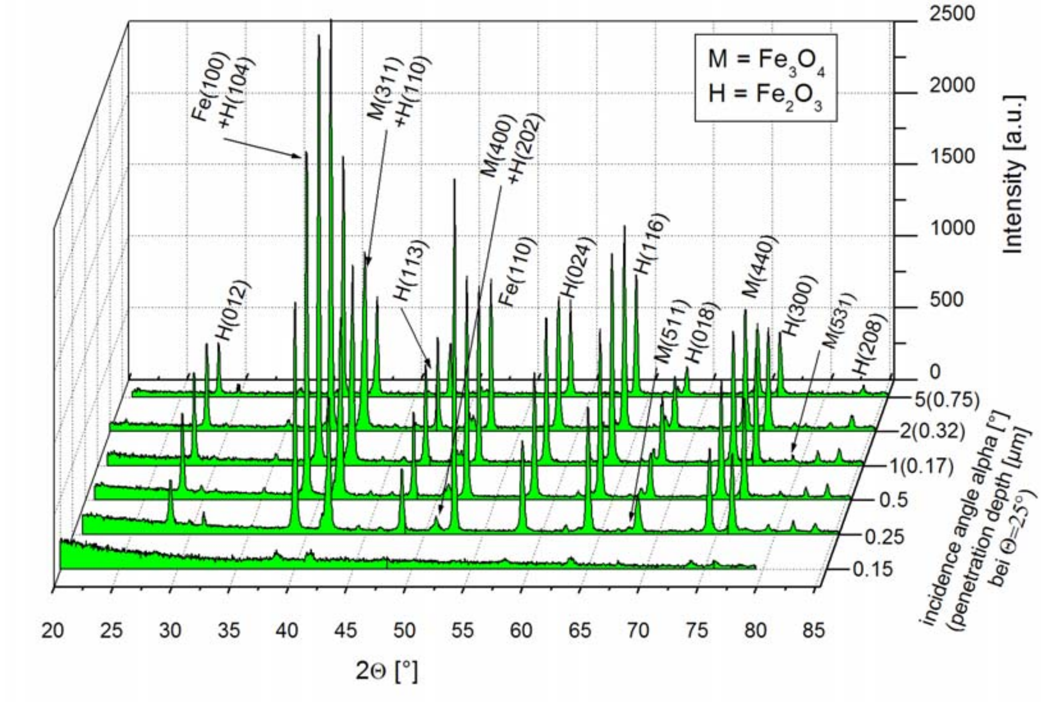

Fig.1: Diffractograms obtained for various incidence angles in an oxide layer on a 100 oriented Fe single crystal

Phase analyses in the θ-2θ-geometry revealed averages of the phase volume fractions of the different oxides (magnetite Fe_3O_4, hematite Fe_2O_3 and wuestite FeO [only for T = 650°C]) in the oxide layers.
Measurements in grazing incidence in contrast allowed us to measure phase volume fractions in different information depths (fig.1). Using the LIBAD method we could also determine the internal stresses in the different oxides and the stress gradients versus the layer depth.
Comparisons of the oxide layers on single crystals with different substrate orientations (fig.2) revealed that the phase volume fraction of magnetite is significantly higher on the 111 – oriented substrate compared to the oxide layers on the 100 and 110 oriented samples and also compared to both fine and coarse grained pure iron substrates. Oxide layers on the iron single crystals contained in general higher compressive residual stresses in the hematite than oxide layers on the polycrystals, which is presumably due to internal oxidation along the grain boundaries of the coarse and fine grained polycrystals. We currently try to link the stress gradients observed to microstructure details observed in transmission electron microscopy. We also found distinct differences in the sharpness of the fibre texture on substrates with different orientations.

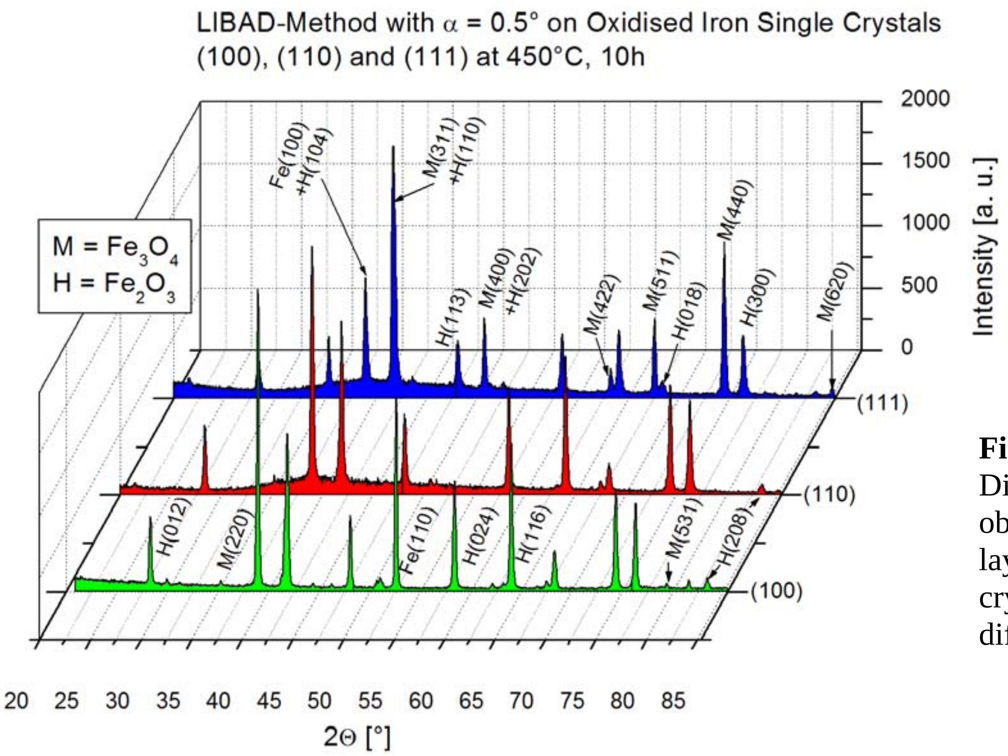

Fig. 2: Diffractograms obtained in the oxide layers on Fe single crystals with different orientations

Phase and especially internal stress analyses in-situ during oxide growth unfortunately were not as successful. This is mainly due to the fact that the times necessary for each measurement in the θ-2θ-geometry and the grazing incidence geometry in the single bunch mode for several samples was more than the remaining beam time and that in one case the data acquisition time was too high to determine significant changes in the internal stress state during the oxidation process. We initially preferred the $\sin^2\psi$ – method for internal stress analyses over the LIBAD method, since we assumed that it might be easier to obtain average values of the phase specific internal stresses that way. The results of the experiments however showed, that the diffractograms obtained during did not allow a reliable determination of the internal stresses and that the LIBAD method should be preferred. A publication is in preparation.