



	Experiment title: Compositional tuning of charge-ordering spin-state transition in REBaFe ₂ O _{5+w}	Experiment number: 01-01-267
Beamline: BM01B	Date of experiment: from: 27.09.2001 to: 02.10.2001	Date of report: 10.02.2002
Shifts: 12	Local contact(s): Mr. Hermann Emmerich	<i>Received at ESRF:</i>
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Report:

Synchrotron x-ray powder diffraction data suitable for Rietveld refinements of the extremely weak charge-ordered superstructure of the double-cell perovskite superstructure were collected with the help of the newly installed sextuplet of crystal analyzers. Patterns for highly stoichiometric Fe^{2.5+} mixed-valence samples EuBaFe₂O_{4.992}, GdBaFe₂O_{4.999}, DyBaFe₂O_{4.996}, HoBaFe₂O_{5.000} and YBaFe₂O_{4.992} have been collected across a wide 2θ range at low temperatures, and the double-superstructure crystal structure parameters were refined. An example of the collected, refined and calculated pattern is shown in Fig. 1. In addition, isothermal scans were collected for these and one more sample in dense temperature intervals across both the main and premonitory Verwey transition and further, in order to establish the transition temperature, coexistence of the charge-ordered and mixed-valence phases under the transition, evolution of the unit-cell parameters and the ΔV value of the first-order main transition. The premonitory Verwey transition proceeds on the structural background of a very subtle orthorhombic distortion and it proved not possible to detect any consistently occurring volume change for the approximately ten times weaker premonitory transition despite the crystal analyzer detection, high angular resolution and the reasonably good stability of the diffraction geometry under temperature changes. The latter is the most critical part, but owing to the very much improved control of the sample position by the "x-ray eye", it was possible to come to a conclusion that there is probably no volume change under the premonitory transition, or at least that the change is less than what the resolution and stability of the diffractometer allows to detect. It goes without saying that none of the data could be collected in any other than synchrotron based powder diffraction instrument.

Fig. 1: Rietveld-refinement profile of the synchrotron x-ray powder diffraction data for the $\text{GdBaFe}_2\text{O}_{4.999}$ sample at 100 K. A detail that includes several of the double-superstructure reflections is in the inset.

