ESRF	**Experiment title:** Crystal structure of charge-ordered $RBaFe_2O_5$ phases	**Experiment number:** CH-1324
Beamline: BM01B	**Date of experiment:** from: 29.11.2002 to: 02.12.2002	**Date of report:** 20.01.2003
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Report:

Diffraction data for $DyBaFe_2O_{5.013}$ were collected at 100 K, for Rietveld refinements (Fig. 1) of the charge-ordered superstructure. This superstructure appears solely due to ordering of oxygen-atoms, unlike the main, double-cell-perovskite-type superstructure that arises owing to ordering of Ba and Dy atoms and oxygen vacancies. Isothermal scans were collected subsequently across $400, 020, 004$ Bragg peaks upon warming through the Verwey transition, in order to calibrate temperature against the more precise DSC value. The sequence was concluded by a data collection at 330 K for the valence-mixed (Robin–Day class-III) phase (Fig. 2).

Due to mechanical problems at the cryostat, a substitute program was adopted, on a sample originally intended for the next round: $GdBaFe_2O_{5+\overline{w}}$ ($\overline{w} = 0.013$), annealed over a period of several months at an elevated temperature in order to narrow the distribution of the oxygen non-stoichiometry w across the bulk. The $400, 020, 004$ Bragg peaks were scanned in dense temperature pace across the interval of the main Verwey transition, up and down, in order to: 1) Get a second point for the temperature calibration. 2) Obtain sharpnes and width of the hysteresis curve of this first-order transition. 3) Deconvolute some peak profiles into a composition distribution by least-squares fitting with the instrumental width (established on a specially collected pattern of Si) via the composition dependence of the unit-cell parameters. 4) Convolute the composition distribution (valence-mixed versus charge-ordered phase) with the instrumental width into a profile, via the composition dependence of the Verwey transition temperature.

Fig. 1: Detail of the refined pattern (0.616 of angular and 1/20 of intensity ranges) for charge-ordered $DyBaFe_2O_{5.013}$ at 100 K. The vertical bars denote Bragg reflections for the present traces of Dy_2O_3 (upper set) and the charge-ordered *Pmma* $DyBaFe_2O_{5.013}$ (lower set). Superstructure Bragg reflections are marked with their *hkl*s where visually resolved on this scale.

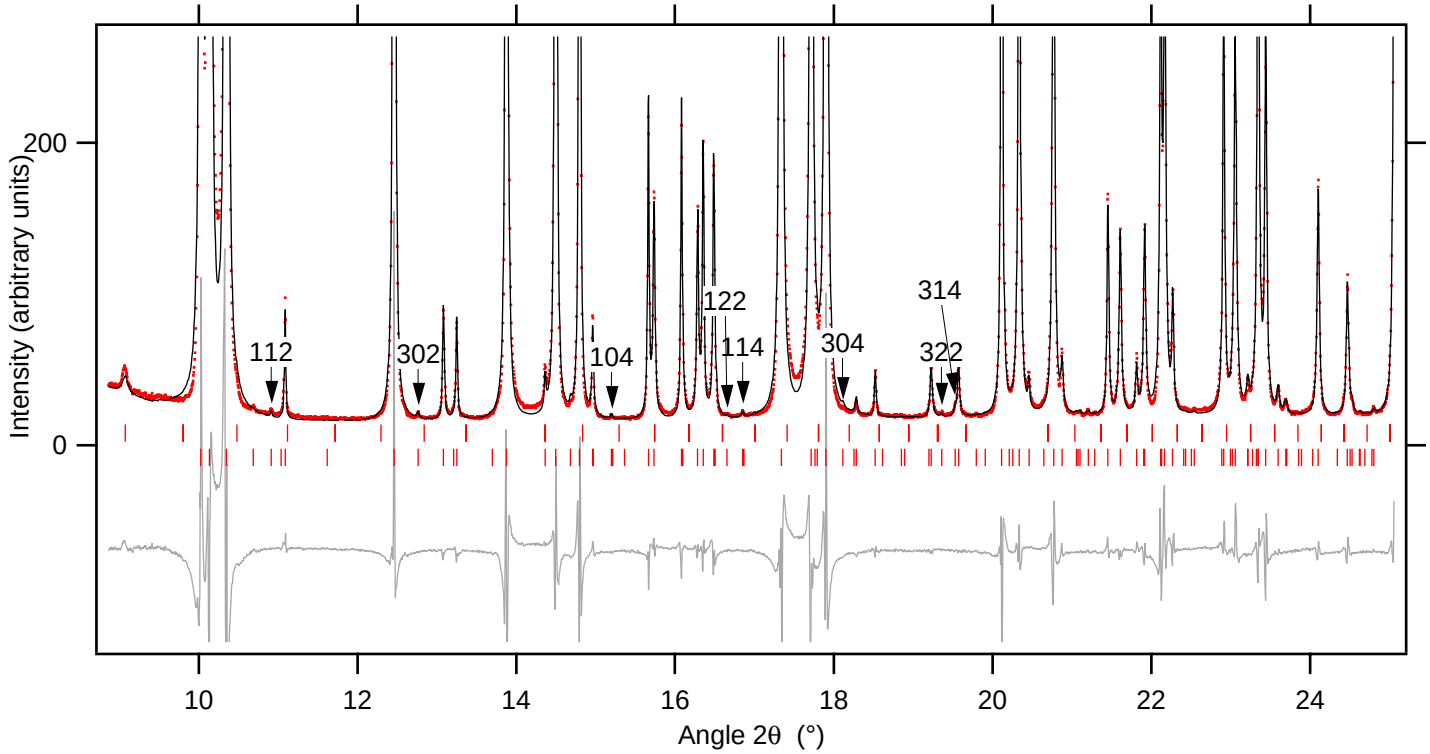

Fig. 2: Detail of the refined pattern (1/2 of angular and 1/20 of intensity ranges) for valence-mixed $DyBaFe_2O_{5.013}$ at 330 K. The vertical bars denote Bragg reflections for the present traces of Fe (upper set), Dy_2O_3 (middle set) and the main phase *Pmmm* $DyBaFe_2O_{5.013}$ (lower set).