Experimental report

10/06/2025

Proposal: CRG-3051 **Council:** 4/2023

Title: Elucidating the magnetic anisotropy in cobalt ferrite nanoparticles by polarized powder neutron diffraction

Research area:

This proposal is a new proposal

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Samples: CoFe2O4

Instrument	Requested days	Allocated days	From	To
D1B	5	3	28/05/2024	31/05/2024

Abstract:

Experimental title: Elucidating the magnetic anisotropy in cobalt ferrite nanoparticles by polarized powder neutron diffraction
Proposal: Nº CRG-D1B-23-514
Proposer: López Ortega, Alberto
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In this study, non-stoichiometric cobalt ferrite nanoparticles ($Co_xFe_{3-x}O_4$) were investigated using polarized neutron powder diffraction (PNPD) to uncover the origin of their pronounced magnetocrystalline anisotropy. Two samples with different cobalt-to-iron ratios were measured on the D1B diffractometer at temperatures of 2 K and 300 K under a 2 T magnetic field.

High-quality diffraction patterns enabled precise determination of cobalt and iron occupancies in tetrahedral and octahedral sites, and, more importantly, allowed extraction of the local magnetic susceptibility tensor for each crystallographic void. Deviations of these tensors from spherical symmetry were interpreted as local magnetic anisotropy.

Refinement of the PNPD data, in combination with compositional ratios obtained by inductively coupled plasma analysis, revealed that the first sample had nearly equal cobalt and iron fractions in both tetrahedral and octahedral positions, while the second sample was markedly iron-rich in both sites. Refinements conducted in the cubic space group yielded isotropic magnetic tensors, in contradiction with independent magnetometry results. Upon lowering the symmetry to tetragonal, a clear axial anisotropy emerged in the octahedral sites, which weakened when the temperature increased from 2 K to 300 K. This model, with an easy axis lying in the basal plane, provided a substantially better fit and accounted for the temperature dependence of the magnetic anisotropy observed experimentally (Figure 1).

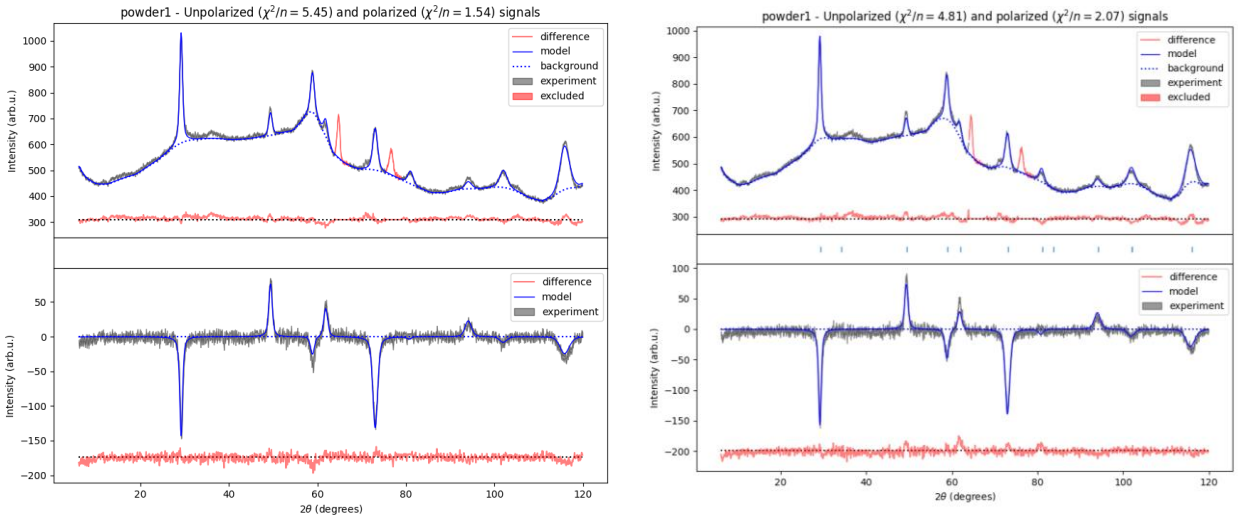

Figure 1. Refinement of the neutron diffraction patterns of sample 1. Sum and difference patterns, measured at 2 K (a,b) and at 300 K (b,d), respectively, Tetragonal SG $I4_1/amd$.

The combination of polarized neutron powder diffraction with prior characterization by inductively coupled plasma and X-ray diffraction enabled precise determination of cobalt and iron site occupancies in both tetrahedral and octahedral voids. Moreover, the local magnetic anisotropy at the octahedral sites was found to be axial and to decrease with

increasing temperature, in agreement with independent magnetometry measurements. The refinement performed in a tetragonal space group suggests the presence of Co^{2+}-induced tetragonal distortions, proposing this symmetry as a working model for future studies. Finally, this work demonstrates that PNPD is a highly effective technique for establishing the easy-axis direction even in nanostructured powder systems.