Experimental report

25/02/2026

Proposal: 1-05-165 **Council:** 4/2025

Title: INVESTIGATING THE MAGNETOELASTIC PHASE TRANSITION IN Fe-Rh ALLOYS BY POLARIZED NEUTRON IMAGING

Research area: Materials

This proposal is a new proposal

Main proposer: **Kenny PADRON ALEMAN**

Experimental team: Kenny PADRON ALEMAN
Ines PUENTE ORENCH

Local contacts: Jiazhou SHEN
Alessandro TENGATTINI

Samples: Fe50Rh50
Fe49Rh51

Instrument	Requested days	Allocated days	From	To
MOTO	4	3	16/09/2025	24/09/2025

Abstract:

Fe100-xRhx (48 ≤ x ≤ 54 at. %) alloys showcase thermally- and magnetically-driven magnetoelastic phase transition, featuring about 1 % of unit-cell volume expansion coupled with an antiferromagnetic-to-ferromagnetic transition when heating. Small differences in the Rh-content can induce different temperatures of transition and result in a broader or sharper phase transition. Consequently, the transition mechanisms vary depending on the alloy composition. The objective of this proposal is to investigate the nucleation process of magnetic domains along the magnetoelastic phase transition through Polarized Neutron Imaging of two Fe-Rh alloys with composition 50:50 and 49:51.

Report MoTo Beamtime: Investigating the Magnetoelastic Phase Transition in Fe-Rh Alloys by Polarized Neutron Imaging

Fe–Rh alloys with compositions close to equiatomic are widely recognized for their giant multicaloric effects—such as magnetocaloric, barocaloric, and elastocaloric responses—associated with a magnetoelastic first-order phase transition (FOPT) occurring around room temperature [1]. The cubic B2 crystal structure of these alloys remains unchanged through the phase transformation; however, the unit-cell volume changes by approximately 1%, and the magnetic order transforms from antiferromagnetic (AFM) at low temperatures (T) to ferromagnetic (FM) at higher T [1].

The characteristics of this phase transition—namely the transition temperature (T_t), hysteresis width (ΔT_{hyst}), and temperature span of the transformation, and consequently the multicaloric responses—can vary significantly from one sample to another, regardless of the synthesis route, composition or morphology [2]. In this beamtime, the transformation kinetics mechanism of three bulk $Fe_{100-x}Rh_x$ alloys with Rh concentrations in the range $50 \leq x \leq 52$ at.% are investigated using polarized neutron imaging (PNI), which enables direct visualization of the AFM-to-FM phase transition of the bulk Fe–Rh alloys.

The measurements were carried out at the MoTo beamline of the ILL in depolarization-contrast mode. The field of view (FOV) used in the setup is 117 × 117 mm^2, which corresponds to an effective pixel sizes of ~57 μm. For polarized neutron experiments, solid-state benders serve as polarizer and analyzer with around 90 % beam polarization across the region-of-interests while a Mezei spin flipper allowed for reversal of the beam polarization. The sample was placed in the free space without a guide field. Instead, the magnetic field generated from the spin polarizer and the analyzer act as effective guide fields. A home-built sample holder and temperature control setup were specifically developed for the experiment. The samples were mounted on an aluminum (Al) block, which served both as a mechanical holder and as a heat transfer element, and also sealed in a Teflon cylinder for less temperature gradient. A commercial hot plate Miniware MHP-50 implemented together with two Type-K thermal couples positioned near the samples, and a PID controller is used to heat up the sample from the bottom of the aluminum holder and maintain stable temperature during the measurement. The setup provided the capability to heat up from RT to 450 K and only passive cooling is implemented in the cooling process.

Depolarization contrast radiographs were acquired at various temperatures during both heating and cooling regimes, covering the full AFM↔FM phase transition in all three samples. Each radiograph consisted of 13 frames in the spin-up configuration and 27 frames in the spin-down configuration, with an acquisition time of 30 s per frame. In addition, tomography measurements were performed at selected temperatures. Each tomographic dataset consisted of 121 radiographs collected over a rotation range of 0–180o, with an angular step of 1.5^o. For each each angular position, 3 spin-up frames and 5 spin-down frames were recorded, also with 30 s exposure per frame. All radiographs were post-processed to reduce noise by subtracting the electronic dark-current noise from the CMOS camera and normalized to flat field images where only the neutron aperture and the detector are present. Depolarization contrast is normalized pixel-to-pixel by an

empty measurement where all the spin polarizer, analyzer, and flipper are in position while the sample setup is removed from the FOV.

Fig. 1(a) display the depolarization-ratio radiographs at selected temperatures recorded during heating across the AFM→FM phase transition corresponding to some preliminary results of this experiment. As *T* increases the depolarization contrast at the samples is increasing as result of the new formed FM regions. Notice that the depolarization pattern evolution is different among the samples indicating the distinct transformation pathways. Fig. 1(b) quantify the average depolarization ratio over the volume of each alloy as the function of the temperature in heating and cooling regimes. At lower temperature the depolarization is close to zero indicating that the material is in the AFM state, at higher temperature, where the sample is fully FM, the beam is completely depolarized. The resulting curves exhibit a broad transformation for the $Fe_{50}Rh_{50}$ alloy and sharp transitions for the near-equiatomic compositions, together with the characteristic thermal hysteresis of the FOPT. Additionally, the uniformity of the phase transition across different regions of each sample was analyzed by averaging the depolarization ration over circular areas of approximately 570 μm in diameter.

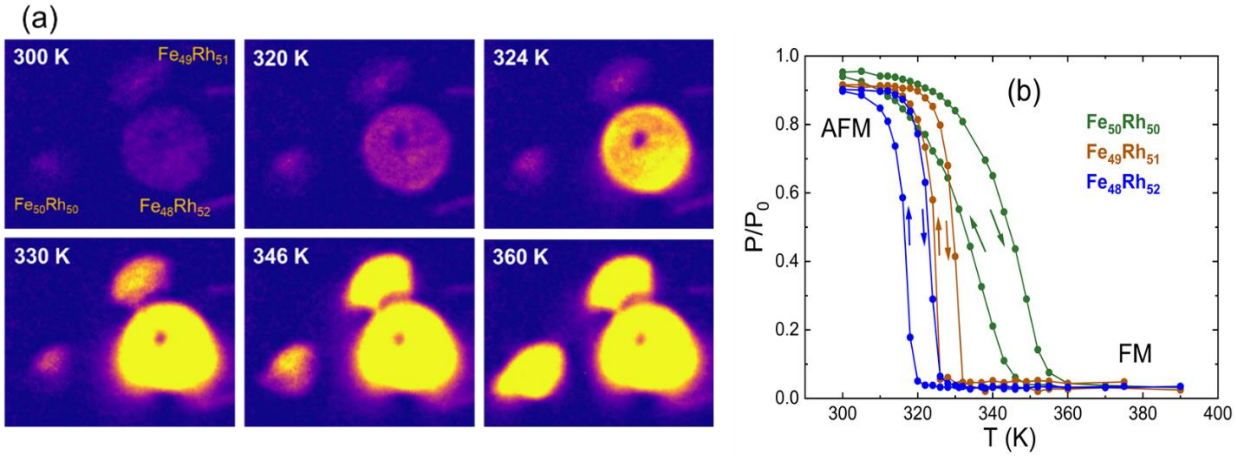


Fig. 1. (a) Depolarization-contrast radiographs mapping the AFM→FM phase transition in the equiatomic and near-equiatomic Fe–Rh alloys at selected temperatures. (b) Temperature dependence of the averaged depolarization ratio over the whole field of view of each sample area in heating and cooling.

This experiment enabled, for the first time, the direct visualization of the FOPT in bulk Fe–Rh alloys. The result indicates a strong dependence of the alloy composition and the homogeneity of the phase transformation over the volume of the samples. Further analysis of the phase transition though the depolarization ratio tomography is underway.

References

[1] K. Padron-Aleman et al., J. Mater. Chem. C, 2025,13, 7017-7026
[2] M. L. Arreguín-Hernández et al., J. Alloys Compd., 2021, 871,159586