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Structural, magnetic, and transport properties of $\text{Fe}_{1-x}\text{Rh}_x/\text{MgO}(001)$ films grown by molecular-beam epitaxy

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$\text{Fe}_{1-x}\text{Rh}_x$ layers are grown with varying rhodium fraction x on (001)-oriented MgO substrates by molecular-beam epitaxy. Film structural, morphological, magnetic, and transport properties are investigated. At room temperature, layers are ferromagnetic (FM) for $x < 0.48$ and antiferromagnetic (AF) for $x > 0.48$. Separating the two magnetically ordered phases at $x = 0.48$ is an abrupt change in the $\text{Fe}_{1-x}\text{Rh}_x$ lattice parameter of $\Delta a = 0.0028$ nm ($\Delta a/a = -0.9\%$). For AF layers, the FM state is recovered by heating across a first-order phase transition. The transition leads to a large resistivity modulation, $\Delta\rho/\rho = 80\%$, over a narrow temperature range, $\Delta T = 3$ K, in stoichiometric $\text{Fe}_{0.50}\text{Rh}_{0.50}/\text{MgO}(001)$. For samples with compositions deviating from $x = 0.50$, fluctuations broaden ΔT and defect scattering reduces $\Delta\rho/\rho$. Published by AIP Publishing.

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FeRh ($Pm\bar{3}m$, B2, CsCl structure) is a fundamental component in memory cells,^{1,2} magnetocaloric refrigerators,^{3,4} and logic devices.^{5,6} Its diverse functionality stems from an entropy-driven first-order transition⁷ between ferromagnetic (FM) and antiferromagnetic (AF) states which persists when deposited as films—a prerequisite for integration in device heterostructures. Accompanying the intrinsic magnetic transition is a large resistivity modulation which rivals giant magnetoresistance effects observed in magnetic multilayers.^{8,9} The rhodium fraction x is suspected to strongly affect $\text{Fe}_{1-x}\text{Rh}_x$ transport characteristics, but its role has not yet been systematically investigated in epitaxial films. Instead, work has been focused on understanding size effects,^{10,11} annealing treatments,^{12–15} and transition mechanics.^{16–20} The few compositional studies on $\text{Fe}_{1-x}\text{Rh}_x$ films omit transport properties entirely, emphasizing magnetic attributes,²¹ or are based on inhomogeneous polycrystalline layers containing secondary phases.²² Here, we systematically examine the structural, morphological, magnetic, and transport properties as a function of rhodium fraction x of phase-pure epitaxial $\text{Fe}_{1-x}\text{Rh}_x$ films with the CsCl structure deposited on (001)-oriented MgO substrates.

$\text{Fe}_{1-x}\text{Rh}_x/\text{MgO}(001)$ films are grown via molecular-beam epitaxy to a thickness of ~ 35 nm in a Veeco GEN10 system (base pressure: 1×10^{-8} Torr = 1.3×10^{-6} Pa) by simultaneously supplying iron (99.995% pure) and rhodium (99.95% pure) from independent effusion cells. Rhodium fractions x are controlled by adjusting iron and rhodium cell temperatures within 50°C of 1150 and 1600°C , respectively, while maintaining a total atomic flux of $\sim 4 \times 10^{13}$ atoms/cm² s, corresponding to a growth rate of ~ 0.3 nm/min. x values determined²⁴ from Rutherford backscattering spectra agree with x-ray reflectivity (XRR) deposition rate calibrations

based on pure iron and rhodium layers (linear correlation coefficient $r = 0.997$), demonstrating that atomic incorporation probabilities are unaltered by chemistry. From the calibrated atomic fluxes, deposition times are set to produce layers with a thickness of ~ 35 nm. A substrate temperature $T_s = 420^\circ\text{C}$ (estimated from a thermocouple in indirect contact with the growth surface and concealed from incident molecular fluxes) is employed for film growth and subsequent 30-min-long *in situ* anneals. High homologous growth temperatures ($T_s/T_m = 0.37$ for FeRh with melting temperature $T_m \approx 1600^\circ\text{C}$) are necessary²⁵ to order bcc $\text{Fe}_{1-x}\text{Rh}_x$ alloys into the B2 CsCl-structure intermetallic with iron and rhodium residing on distinct positions of the two-atom basis.

X-ray diffraction (XRD) θ - 2θ scans, collected using Cu $K_{\alpha 1}$ radiation (wavelength $\lambda = 0.154056$ nm), establish a phase diagram consisting of four regions: single-phase bcc-Fe(001) ($x \leq 0.20$), single-phase B2 $\text{Fe}_{1-x}\text{Rh}_x$ ($0.20 \leq x \leq 0.60$), two-phase mixtures of (001)-textured B2 $\text{Fe}_{1-x}\text{Rh}_x$ and fcc-Rh ($0.60 \leq x \leq 0.80$), and single-phase fcc-Rh(001) ($x \geq 0.80$). The phase boundaries of our epitaxial films grown on MgO(001) are in close agreement with reports for bulk samples:^{23,26} the rhodium-deficient limit, for which the bcc solid solution orders into the CsCl structure, agrees exactly, while the rhodium-rich limit extends 0.08 rhodium fractions above the bulk boundary ($x = 0.52$) due to epitaxial stabilization.^{27–31}

A representative XRD θ - 2θ scan is presented in Fig. 1(a) for stoichiometric $\text{Fe}_{0.50}\text{Rh}_{0.50}/\text{MgO}(001)$. Five peaks are observed over the 2θ range 10 – 110° : the three reflections at $2\theta = 29.94$, 62.18 , and 101.6° are indexed as $\text{Fe}_{0.50}\text{Rh}_{0.50}$ 00 l ; the two peaks at 42.92 and 94.05° are identified as MgO 00 $2l$. Sharp mixed-integer film reflections (no systematic absences) indicate CsCl-type ordering. The lack of additional reflections together with pole figure and grazing-incidence scans (not shown) establish that films with $0.20 \leq x \leq 0.60$ are phase-pure untwinned epitaxial layers oriented with a 45° in-plane

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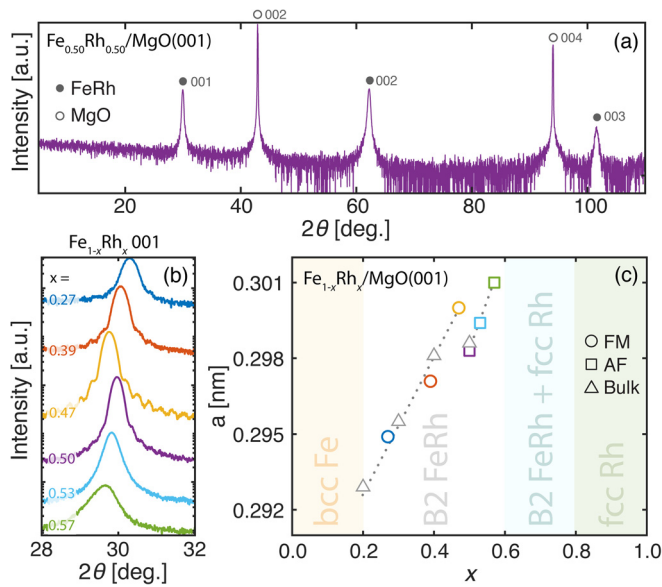


FIG. 1. (a) XRD θ - 2θ scan of an ~ 35 -nm-thick stoichiometric $\text{Fe}_{0.50}\text{Rh}_{0.50}$ film with the B2 CsCl-structure grown on $\text{MgO}(001)$ at 420°C by molecular-beam epitaxy. (b) θ - 2θ scans showing the $\text{Fe}_{1-x}\text{Rh}_x$ 001 peak for rhodium fractions $0.20 \leq x \leq 0.60$. (c) Film out-of-plane lattice parameters (circles and squares) as a function of composition together with bulk lattice parameters²³ (triangles) for reference. Circles indicate ferromagnetic ordering and squares indicate antiferromagnetic ordering at room temperature.

rotation with respect to their MgO substrates: $(001)_{\text{Fe}_{1-x}\text{Rh}_x} \parallel (001)_{\text{MgO}}$ and $[110]_{\text{Fe}_{1-x}\text{Rh}_x} \parallel [100]_{\text{MgO}}$.

Diffracted intensities near the $\text{Fe}_{1-x}\text{Rh}_x$ 001 reflection are plotted as a function of x in Fig. 1(b). As x increases across the single-phase field, $\text{Fe}_{1-x}\text{Rh}_x$ peaks shift—with one exception—to lower 2θ angles. Figure 1(c) shows out-of-plane lattice parameter a values obtained³² from θ - 2θ peak positions. a increases approximately linearly from 0.2950 ($x=0.27$) to 0.3000 nm ($x=0.47$), contracts sharply to 0.2983 nm ($x=0.50$), and then continues increasing to 0.3010 ($x=0.57$). Film lattice parameters values $a(x)$ are in excellent agreement with reports for bulk polycrystals [also shown in Fig. 1(c)].²³ Regression analyses yield a slope of 0.04 ± 0.01 nm per rhodium fraction, in close agreement with 0.06 expected based on the larger metallic radius³³ of rhodium (134 pm) versus iron (126 pm), suggesting that rhodium substitutes for iron across the $\text{Fe}_{1-x}\text{Rh}_x$ single-phase field. The lattice parameter discontinuity of $\Delta a = 0.0028$ nm ($\Delta a/a = -0.9\%$) at $x = 0.48$ occurs as $\text{Fe}_{1-x}\text{Rh}_x$ undergoes a first-order transition⁷ from a FM ($x < 0.48$) to an AF ($x > 0.48$) state.³⁴ The contracted AF cell corresponds to the new equilibrium geometry³⁵ after spins ferromagnetically aligned on iron ($3.2 \mu_B$) and rhodium ($0.9 \mu_B$) leave rhodium ($0.0 \mu_B$) magnetically inactive and reorganize antiferromagnetically along $\{001\}$ on iron ($3.3 \mu_B$).^{26,36,37}

The structural quality of the films is assessed from ω -rocking curves of $\text{Fe}_{1-x}\text{Rh}_x$ 001 reflections and atomic force microscopy (AFM) elevation maps. Rocking curve scans and corresponding peak full-width-at-half-maxima (FWHM) are plotted in Figs. 2(a) and 2(b). Reflections are broad at $x = 0.27$ and 0.57 due to crystalline mosaicity, but sharpen as x approaches 0.50. FWHM values decrease from 0.65° ($x = 0.27$) and 1.28° ($x = 0.57$) to 0.23° ($x = 0.47$) and 0.32° ($x = 0.50$) indicating increasing crystalline perfection. MgO

002 rocking curves, measured for reference, are found to consist of split peaks with individual peak FWHM values of $\sim 0.005^\circ$ (18 arcsec) and an ensemble FWHM of $\sim 0.06^\circ$ (216 arcsec); the splitting results from the formation of subgrains (separated by small-angle grain boundaries) and are commonly observed in commercial MgO substrates.³⁸

Figure 2(c) depicts representative AFM height images. Root-mean-square surface roughness values ρ_{rms} determined independently from AFM and XRR (not shown) are plotted as a function of x in Fig. 2(d). At $x = 0.27$, the surface morphology ($\rho_{rms} = 3.0$ nm) is comprised of 150-nm-wide mesas separated by 1.5-nm-deep trenches preferentially aligned along $\text{Fe}_{1-x}\text{Rh}_x \langle 100 \rangle$. Such features are the hallmark of unfavorable substrate wetting and three-dimensional island growth.³⁹ For $x = 0.47$, the mesas fuse leaving a smooth surface with sub-monolayer height fluctuations ($\rho_{rms} = 0.1$ nm). Further increasing x to 0.57 is accompanied by the appearance of mounds faceted along $\text{Fe}_{1-x}\text{Rh}_x \langle 100 \rangle$ due to the combination of high surface energies and high diffusivities.^{40,41} Thus, the smoothest films with the highest structural perfection are obtained near $x = 0.50$.

Figure 3(a) shows the in-plane room-temperature magnetization M of $\text{Fe}_{1-x}\text{Rh}_x/\text{MgO}(001)$ films measured as a function of applied magnetic field H using a vibrating sample magnetometer. Films with $x \leq 0.48$ display hysteretic behavior characteristic of FM ordering with saturation magnetizations of $\sim 4 \mu_B/\text{f.u.}$, consistent with prior reports.³⁶ Coercive fields H_c , defined as the value of H where M changes maximally, decrease with increasing x from 235 ($x = 0.27$) to 129 ($x = 0.39$) and 59 Oe ($x = 0.47$). Fitting $H_c(x)$ with a

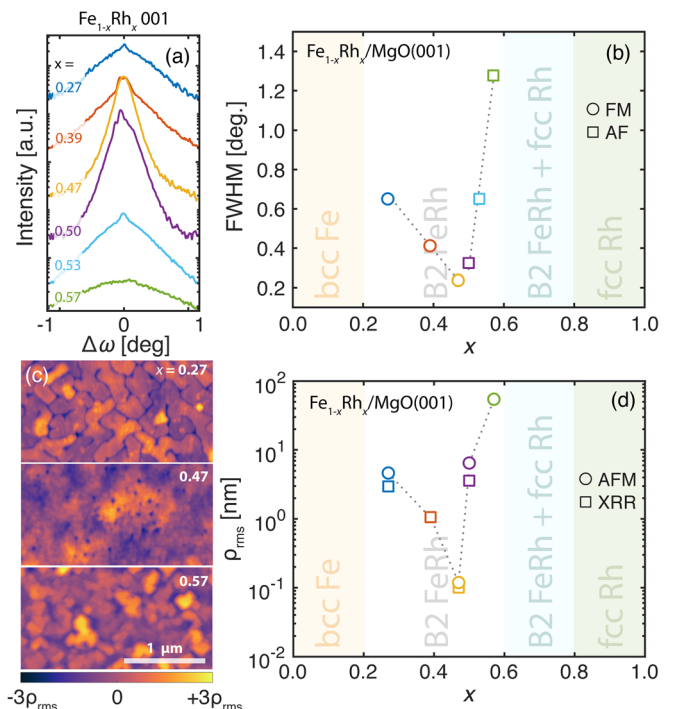


FIG. 2. (a) XRD ω -rocking curve scans of $\text{Fe}_{1-x}\text{Rh}_x$ 001 reflections and (b) corresponding FWHM values as a function of rhodium fraction x . (c) Representative AFM height images of $\text{Fe}_{1-x}\text{Rh}_x/\text{MgO}(001)$ layers as a function of composition x across the B2 single-phase field. $\text{MgO}[100]$ and $\text{Fe}_{1-x}\text{Rh}_x[110]$ are aligned with the horizontal image axis. (d) Root-mean-square surface roughness values determined as a function of x independently using XRR and AFM.

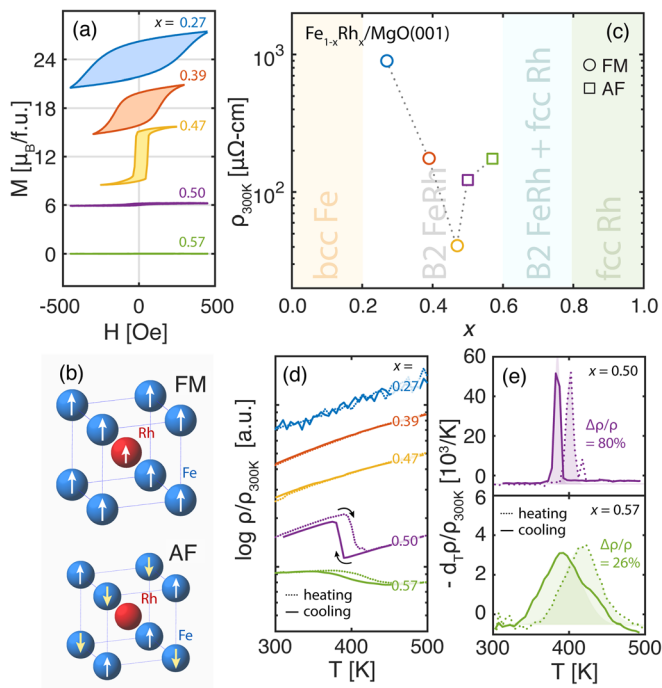


FIG. 3. (a) Magnetization M of $\text{Fe}_{1-x}\text{Rh}_x/\text{MgO}(001)$ films versus applied magnetic field H as a function of rhodium fraction x . Curves are offset by $6 \mu_B/f.u.$ for clarity. (b) The crystal structure and spin configurations of ferromagnetic (FM: $x < 0.48$) and antiferromagnetic (AF: $x > 0.48$) $\text{Fe}_{1-x}\text{Rh}_x$. (c) Room-temperature $\text{Fe}_{1-x}\text{Rh}_x$ resistivities $\rho_{300K}(x)$ for $x = 0.20$ through 0.80 , spanning the B2 single-phase field. (d) Temperature-dependent resistivities $\rho(T)$ as a function of x ; curves are vertically offset for clarity. (e) Negative temperature-derivative of $\rho(T)$ for samples exhibiting AF-FM transitions ($0.48 < x \leq 0.60$).

mean-field behavior, $H_c \propto \sqrt{x - x_c}$, yields a critical rhodium fraction of $x_c = 0.48$ below which $\text{Fe}_{1-x}\text{Rh}_x$ is FM. For x above x_c , $\text{Fe}_{1-x}\text{Rh}_x$ films are macroscopically demagnetized at room temperature, but recover their magnetization when heated above ~ 400 K. Since symmetries are necessarily restored by heating across any phase transition,⁴² the loss of magnetization in films with $x > 0.48$ implies AF ordering, for which heating leads to the recovery of additional symmetry operations and the emergence of a FM state. These conclusions are in agreement with Mössbauer spectroscopy²⁶ and neutron scattering³⁶ results. The crystal structure and spin configurations of ferromagnetic and antiferromagnetic $\text{Fe}_{1-x}\text{Rh}_x$ are illustrated in Fig. 3(b).

Room-temperature resistivities $\rho_{300K}(x)$ of ~ 35 -nm-thick $\text{Fe}_{1-x}\text{Rh}_x/\text{MgO}(001)$ films are shown in Fig. 3(c). As x is varied across the single-phase field, ρ_{300K} decreases from $898.3 \mu\Omega\text{ cm}$ ($x = 0.27$) to $40.9 \mu\Omega\text{ cm}$ ($x = 0.47$), rises rapidly to $122.3 \mu\Omega\text{ cm}$ ($x = 0.50$), and continues increasing slowly to $174.9 \mu\Omega\text{ cm}$ ($x = 0.57$). The resistivity obtained here for stoichiometric $\text{Fe}_{0.50}\text{Rh}_{0.50}$, which represents the lowest value reported in the literature,⁴³ reflects the structural perfection and chemical purity of the layer. The large $\rho_{300K}(x)$ values near the $\text{Fe}_{1-x}\text{Rh}_x$ phase field boundaries stem predominately from increased structural disorder.

Temperature-dependent $\text{Fe}_{1-x}\text{Rh}_x$ resistivities $\rho(T)$ between 300 and 500 K are plotted in Fig. 3(d). For rhodium-deficient films ($0.27 \leq x \leq 0.47$), $\rho(T)$ increase linearly with T demonstrating metallic phonon-limited conduction. The superposition of resistivity curves measured during

heating and cooling reflect the stability of these layers in air. At $x = 0.50$, a drop in resistivity is observed near $T_c \approx 392$ K, associated with a transition⁷ between AF ($T < T_c$) and FM ($T > T_c$) states. The negative temperature-derivative of $\rho(T)/\rho_{300K}$, plotted in Fig. 3(e), shows that the transition is sharp, hysteretic, and symmetric—attributes consistent with first-order transitions—and occurs at 385 ± 3 and 401 ± 3 K during heating and cooling, respectively. The pronounced modulation in resistivity observed, $\Delta\rho/\rho \equiv (\rho_{AF} - \rho_{FM})/\rho_{FM} = 80\%$, represents the highest thermally induced value reported^{6,10,22,43–45} and is consistent with the $85 \pm 6\%$ theoretical maximum realizable for well-ordered films,²² the narrow transition widths, $\Delta T = 3$ K, are the smallest observed to date.^{6,10,11,22,43–45} For bulk stoichiometric samples, a comparable resistivity change was observed at room temperature by driving the AF-FM transition with pulsed magnetic fields exceeding 15 T; thermally induced resistivity changes were not investigated, but a T_c of 405 K, in close agreement with our measured values, was deduced from temperature-dependent heat capacity measurements.⁴⁶

Rhodium-rich films with $x = 0.57$ also exhibit a similar transition. In this case, the resistivity changes by only 26% (versus 80% for $x = 0.50$) as AF regions slowly transform into FM domains at 418 ± 32 K and back at 393 ± 40 K [Figs. 3(d) and 3(e)]. The smaller $\Delta\rho/\rho$ values results from defect scattering, which simultaneously raises ρ_{AF} and ρ_{FM} . The broader transition stems from fluctuations, as expected for a film characterized by chemical disorder, crystalline mosaicity, and high surface roughness.

In summary, ~ 35 -nm-thick epitaxial $\text{Fe}_{1-x}\text{Rh}_x/\text{MgO}(001)$ films are grown at 420°C by molecular-beam epitaxy and systematically investigated as a function of rhodium fraction x . Within the CsCl-structure $\text{Fe}_{1-x}\text{Rh}_x$ single-phase field ($0.20 \leq x \leq 0.60$), rhodium replaces iron producing a linearly increasing lattice parameter due to its larger metallic radius (134 versus 126 pm).³³ B2 CsCl-type ordering is evidenced by pronounced x-ray diffraction from mixed-integer film reflections. A lattice parameter discontinuity of $\Delta a = 0.0028$ nm ($\Delta a/a = -0.9\%$) is observed at $x_c = 0.48$, below (above) which films are FM (AF). The perfection and surface smoothness of the layers are optimized near $x = 0.50$. Room-temperature resistivities $\rho_{300K}(x)$ exhibit a minimum of $40.9 \mu\Omega\text{ cm}$ at $x = 0.47$. For AF layers ($x \geq 0.48$), FM ordering can be recovered by heating across the first-order phase transition. Temperature-dependent resistivity measurements demonstrate sharp, hysteretic, and symmetric transitions at 385 ± 3 K and 401 ± 3 K during heating and cooling of stoichiometric $\text{Fe}_{0.50}\text{Rh}_{0.50}/\text{MgO}(001)$ films. The large resistivity modulation achieved, $\Delta\rho/\rho = 80\%$, represents the largest thermally induced value observed to date for $\text{Fe}_{1-x}\text{Rh}_x$ films. In rhodium-rich layers, the transition is broadened by fluctuations and the percent resistivity change is reduced due to defect scattering.

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