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L1

SELECTED APPLICATIONS OF LABORATORY SMALL ANGLE X-RAY SCATTERING

Milan Dopita

Department of Condensed Matter Physics, Faculty of Mathematics and Physics, Charles University, Ke Karlovu 5, 121 16 Prague 2, Czech Republic
milan.dopita@matfyz.cuni.cz

The lecture will provide an introduction to the fundamental principles of small-angle X-ray scattering (SAXS), with emphasis on its theoretical background, experimental methodology, and data interpretation. Particular attention will be devoted to laboratory-based SAXS instrumentation available at our department, including its capabilities, limitations, and practical implementation in materials research.

Selected case studies will be presented to illustrate the versatility of SAXS in the characterization of nanostructured and nanocrystalline systems. These will include investigations of oriented hexaferrite thin films, highly disordered carbon nanomaterials, and three-dimensionally ordered micro–mesoporous carbons. Further examples will cover metal-organic pharmaceutical compounds as well as silicon- and metal-based materials relevant for energy storage and catalytic applications.

A dedicated part of the lecture will focus on advanced in situ and in operando SAXS experiments performed using laboratory equipment equipped with microfocus X-ray sources. The possibilities and inherent limitations of such setups for high-temperature and time-resolved studies will be critically discussed. Temperature-induced morphological and structural evolution will be demonstrated using tungsten nanoparticle layers prepared by gas aggregation cluster sources.

In addition, in operando SAXS studies will be presented to illustrate real-time monitoring of microstructural changes in functional devices. These examples will include microstructural evolution during charge-discharge cycling (lithiation/delithiation) in lithium-based coin cell batteries, as well as structural transformations in water electrolyzers and fuel cells under operating conditions.

Overall, the lecture aims to highlight the potential of laboratory SAXS as a powerful and accessible tool for probing nanoscale structure and its evolution in a wide range of advanced materials.

L2

Rh-Mn-Sb HEUSLER COMPOUNDS IN THIN FILMS

Esther de Prado[1], Artem Shamardin[1], Stanislav Cichoň[1], Jan Duchoň[1], Michal Rameš[1], Ladislav Fekete[1], Jaromír Kopeček[1], Šimon Šťastný[2], Aleš Melzer[2], Michal Hubert[2], Martin Veis[2], Ján Lančok[1], Oleg Heczko[1]

[1]FZU-Institute of Physics of the Czech Academy of Sciences, Na Slovance 1999/2, Prague 18000, Czech Republic
[2]Institute of Physics of Charles University, Ke Karlovu 2026/5, Prague 12116, Czech Republic

Heusler compounds constitute a broad family of intermetallic materials, with more than 1500 possible compositions reported to date. Among them, the Rh–Mn–Sb system has attracted scientific interest for nearly half a century [1]. However, although bulk Rh–Mn–Sb alloys were studied several decades ago, experimental work on this system has since remained very limited, and thin films have not been explored. In this context, Rh_2MnSb full-Heusler thin films were epitaxially grown for the first time on MgO(001) substrates by DC magnetron sputtering at deposition temperatures of 600, 700, and 800 °C [2]. Films with a nominal thickness of 200 nm were systematically characterized in terms of their structural, morphological, optical, magneto-optical, and magnetic properties. The sample deposited at 600 °C was close to stoichiometric and exhibited an almost perfectly ordered $L2_1$ structure. All films exhibited a tetragonal structure and a regular twinned microstructure, in which most twin domains were oriented with the c-axis perpendicular to the film surface, presumably as a result of the constraint imposed by the substrate. Magnetic measurements for the film grown at 600 °C yielded a Curie temperature of about 220–275 K and a saturation magnetization of approximately 55 emu/g at 10 K, both close to bulk values, while magneto-optical Kerr measurements indicated para-

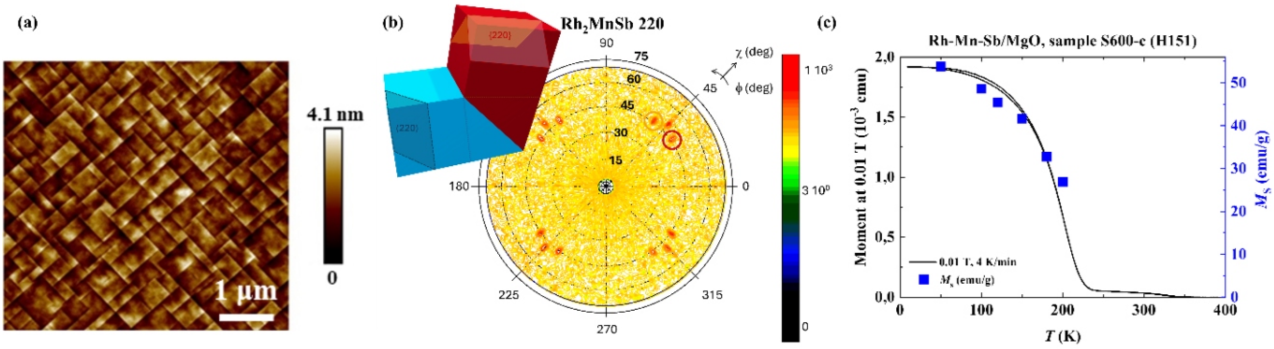

Figure 1: (a) AFM topography image of a Rh_2MnSb film deposited at 600 °C; (b) structural model of twin-related tetragonal Rh_2MnSb variants and corresponding {220} pole figure; and (c) thermomagnetic curves measured at 0.01 T.

magnetic behavior at room temperature. These results demonstrate the potential of Rh_2MnSb thin films and motivate further work aimed at improving compositional precision and structural control.

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L3

STUDY OF Ni_2FeGa MICROWIRE BY CONVENTIONAL X-RAY DIFFRACTION

Petr Cejpek, Limpat Nulandaya, Ladislav Straka

Institute of Physics, Czech Academy of Sciences, Na Slovance 1999/2, 182 00 Praha 8, Czech Republic

Ni_2FeGa is a magnetic shape memory alloy with potential applications in microdevices such as actuators and micropumps. The preparation of bulk single crystals of this material is challenging; however, the fabrication of microwires containing highly oriented grains appears promising and economically feasible, as large quantities of such wires can be produced [1]. Still, the characterization of these microwires by conventional X-ray diffraction remains difficult due to their small diameter (typically 10–50 m), which results in a limited diffracting volume. An additional challenge is the manipulation and mounting of the sample.

The Ni_2FeGa microwires were placed and stretched inside own 3D-printed sample holder (Fig. 1) and initial measurements were performed using a SmartLab diffractometer with a 2D pole figure method and a Hypix3000 area detector positioned as close to the sample as possible. This setup significantly reduces the acquisition

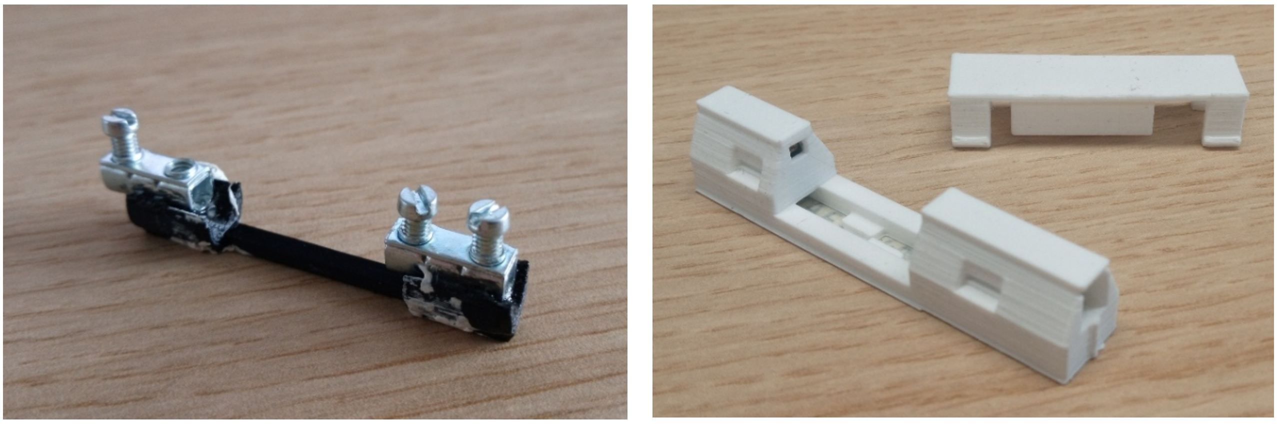

Figure 1. 3D-printed microwire holder. **a)** The first prototype, **b)** the second prototype – the metallic parts are placed upside down inside the plastic body to hide the screws.

time for a single pole figure. However, individual reconstruction of pole figures is required to avoid the integration of a large background contribution and diffraction peaks originating from the sample holder.

Several conclusions can be drawn from the pole figures: 1) the presence of both cubic austenite and tetragonal martensite phases can be identified; 2) Ni_2FeGa exhibits a strong preferential orientation with the direction aligned along the wire axis; and 3) the presence of single or multiple martensitic variants is clearly observable.

These initial results open the way for future in situ experiments, such as in situ Joule heating by passing an electric current through the microwire, or in situ mechanical straining using a dedicated sample holder with a geared mechanism.

[1] Frolova, L., et al. *Reversible structural transition in monocrystalline Ni2FeGa microwires for shape-memory applications*, Materials Science and Engineering B **263** (2021) 114891, https://doi.org/10.1016/j.mseb.2020.114891.

L4

HEXAFERRITE THIN FILMS GROWN BY CHEMICAL SOLUTION DEPOSITION ON DIFFERENTLY ORIENTED SAPPHIRE SUBSTRATES

Radomír Kužel[1], Lukáš Horák[1], Milan Dopita[1], Darina Smržová[2], Josef Buršík[2]

[1]Charles University, Faculty of Mathematics and Physics, Prague, Czech Republic
[2]Institute of Inorganic Chemistry of the Czech Academy of Sciences, v.v.i., 250 68 Husinec-Řež 1001, Czech Republic
radomir.kuzel@matfyz.cuni.cz

Strongly oriented ferrite films were deposited on several substrates by chemical solution deposition, and different substrate/seeding layer/ferrite layer architectures were designed [e.g.1, 2]. In previous experiments, the hexaferrite films were deposited on STO ($SrTiO_3$) substrates with orientations (111), (110), (100), and ceramics. In all cases, (00l) textures of Y-phases of different degrees were observed, but only for the (111) substrate strong in-plane texture was found. For other used substrates, usually some intermediate seeding layer was necessary to obtain good orientation of Y-films. The most successful were $LaAlO_3$ and MgO substrates.

In this work [3], highly oriented M-phase films with different orientations were grown on Al_2O_3 ((11$\bar{2}$0) - *a*-cut, (0001) – *c*-cut, ((10$\bar{1}$0) - *m*-cut and ((1$\bar{1}$02)- r-cut single crystal substrates. A procedure involving thin and the difference in lattice parameters between the seed and bulk layers. $SrFe_6Ga_6O_{12}$ seed layers was adopted to modify the lattice mismatched interface between film and substrate and allow the growth of oriented films. Usual thickness of the seeding layer was about 100 nm obtained after several deposition cycles. M-phase, $SrFe_{12}O_{19}$, film samples with 1600 nm thickness were used for different analyses. The deposition of the bulk M layer always reduces the in-plane orientation (while maintaining the epitaxial relationship) of the originally well-oriented M seed layer. The deposition of the bulk layer M usually results in a broadening of the and scan peaks by about a factor of two in most cases. The increase in FWHM value for the scan observed in the case of the M-bulk layer suggests a slight increase in misorientation of the layer and deterioration of the OP orientation of the M phase due to the increasing total layer thickness the difference in lattice parameters between the seed and bulk layers

The pseudo-epitaxial growth and orientation relation were verified by X-ray diffraction texture analysis and reciprocal space mapping. and scans as well as selected pole figures were measured in low-resolution parallel beam setup with polycapillary in primary beam and parallel plate collimator (Panalytical MRD), monochromator and point detector in the secondary beam. Significant out-of-plane textures of (*h*00), (00*l*), and (*hh*0) were found on the *a*-, *c*- and *m*-cuts of substrates, respectively. The FWHM of -scans were 0.8-1.2°. Similarly, significant in-plane orientations were found for the *a*- and *m*-cuts of the substrates, respectively while for the *c*-cut they were less good indicating a quite large fraction of unoriented crystallites. The surface morphology at room temperature using AFM in tapping mode. Except for the *c*-cut substrates, the surface structure is formed by parallel aligned grains with high aspect ratio and the shortest dimension along *c*-crystallographic axis. The FWHM of the -scans depends strongly on angle being the highest along this smallest crystallite dimension which could be caused by the size effect. However, to decide this, the Williamson-Hall plots for -scans were measured for more diffraction orders. They were also constructed in classical form, i.e. for the profiles measured along the diffraction vector ( ). These measurements were performed in higher-resolution setup with hybride K$_1$ monochromator in the primary beam and Pixcel 3D detector in the secondary beam.

For -scans, it is clear, that crystallite misorientations (slope) is still the dominating effect for broadening and it is more significant in the direction perpendicular to crystallite rod, i.e. along [0001]. The intercepts would give crystallite size of 30 to 50 nm for *m*- and *c*-cut of subtrates, respectively (Figure 1). This corresponds quite well to AFM pictures. Classical WH plots along the diffraction vector gives small microstrains of about 0.06 to 0.1 % and crystallite size above 100 nm.

For M layers on *r*-cuts of substrates, the orientation is more complex and in original measurements no intensity in symmetrical scans could be observed. However, pole figure 114 showed that actually there is an inclined texture with inclination 3-5 ° depending on the film (Figure 2) and the case was studied in detail [4].

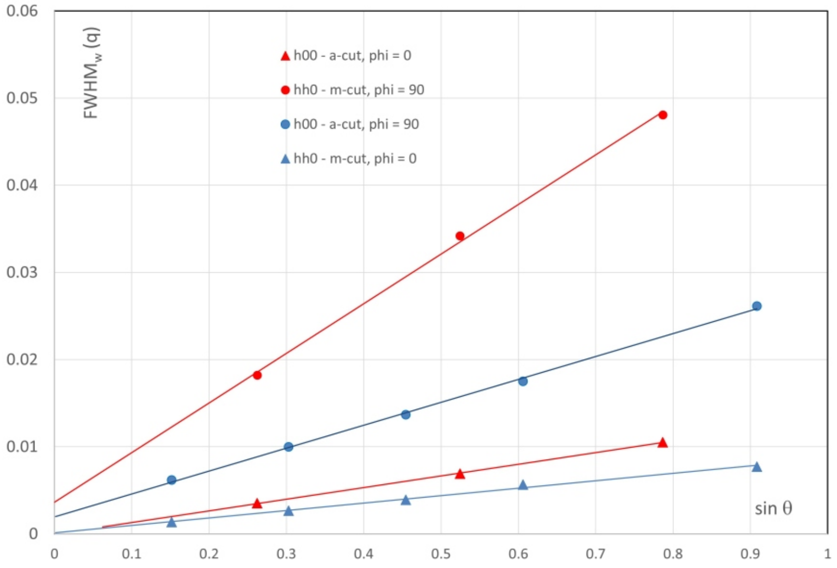

Figure 1. Williamson-Hall plot of the width of -scans of *hh*0 and *h*00 diffractions for *m*-cut and *a*-cut substrates, respectively and for two orientations of the film, along (= 0°) and perpendicular (= 90°) to crystallite rods.

A comparison of texture analysis results obtained on sapphire substrates of different cuts shows that the mutual orientations of the crystal structure of the M film and the sapphire substrates in epitaxial structures are always the same (except for a small angle deviation for an *r*-cut). As a result, there is a high probability that thin films of the M hexaferrite film with an easy axis of magnetization at any angle with respect to the substrate can be attained by making an appropriate choice of the orientation of the Al_2O_3 substrate.

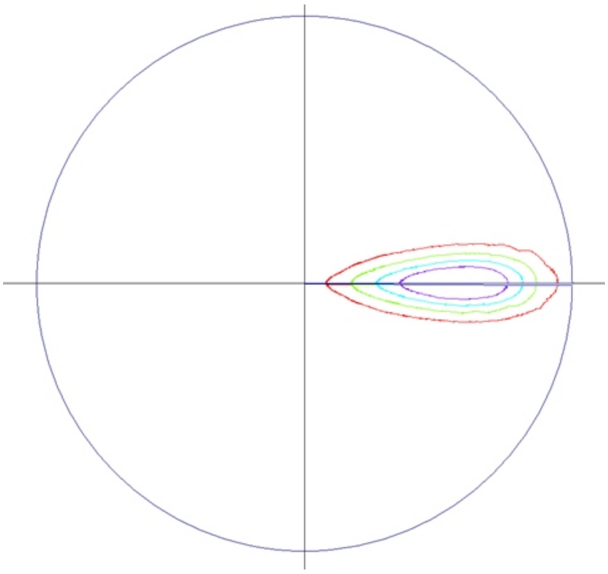

Figure 2. Pole figure 114 of the M-film on *r*-cut substrate. The circle corresponds to the angle of 10°.

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L5

PROBING NON-TRIVIAL LAYER-SUBSTRATE ORIENTATIONAL RELATIONSHIPS USING WIDE RECIPROCAL SPACE MAPPING

Lukáš Horák[1], Radomír Kužel[1], Milan Dopita[1], Josef Buršík[2]

[1]*Charles University, Faculty of Mathematics and Physics, Prague, Czech Republic*
[2]*Institute of Inorganic Chemistry of the Czech Academy of Sciences, v.v.i., 250 68 Husinec-Řež 1001, Czech Republic*
lukas.horak@matfyz.cuni.cz

Introduction

Strongly oriented polycrystalline thin films and epitaxial layers typically exhibit a well-defined crystallographic relationship with their substrates. Describing and understanding this relationship is essential for interpreting the structural, electronic, and functional properties of thin-film systems.

For epitaxial layers, the orientation relationship is most commonly investigated using high-resolution reciprocal space mapping (HR-RSM), which relies on a limited set of predicted unit-cell orientations derived from lattice matching considerations. In this case, the experiment primarily serves to verify one of the anticipated configurations. In contrast, strongly textured polycrystalline films are usually characterized by pole-figure measurements, which probe all crystallographic orientations present in the film. This approach, however, requires reasonably well-known lattice parameters in order to select appropriate diffraction conditions, which are not always available.

In some systems, the film texture or epitaxial relationship is neither obvious nor intuitive. In such non-trivial cases, no diffraction peaks from the layer appear at the expected positions, and no layer reflections are observed in symmetric scans, indicating the absence of any low-index crystallographic plane parallel to the surface. Pole-figure analysis may also become impractical when lattice parameters are uncertain or when the number of diffracted peaks is large and overlaps significantly with substrate reflections, making reliable interpretation extremely difficult.

We define non-trivial layer-substrate orientation relationships as configurations in which the layer is both in-plane and out-of-plane oriented, yet no low-Miller-index plane of the layer is parallel to the substrate surface (or to a near-surface crystallographic plane of the substrate), and where only a minimal number of planes or directions are aligned in an unintuitive manner relative to the substrate lattice.

In such situations, conventional techniques-high-resolution reciprocal space mapping, symmetric scans, and pole-figure measurements are either insufficient or require laborious experimental effort. We therefore propose the use of low-resolution wide reciprocal space mapping as a fast, robust, and largely foolproof experimental approach. This method rapidly collects comprehensive reciprocal-space information, limited primarily by instrumental resolution, without requiring prior assumptions about lattice parameters or orientation relationships.

In the following sections, we briefly describe the wide reciprocal space mapping methodology and present results from one representative systems: strongly textured polycrystalline M-type hexaferrite thin film grown on R-cut sapphire (ALO) substrate. Previous studies have shown that M-type hexaferrites grown on Al_2O_3 or MgO substrates exhibit substrate-dependent crystallographic orientations, with well-defined low-index alignments on C-cut sapphire and MgO(111), but increasingly complex and tilted configurations on *A*-, *M*-, and especially *R*-cut sapphire [1-4]. However, across all these substrate cuts, the approximate crystallographic relationship appears to be universal. Thus, even though there existed well-justified expectations for the layer orientation based on crystallographic considerations, we demonstrate that the actual growth results in non-trivial orientation relationships, with no low-index planes parallel to the substrate surface, arising from a small tilt of the substrate lattice along a specific in-plane direction.

Methods

The crystallographic orientation relationships between the thin films and their substrates were investigated using wide reciprocal space mapping (WRSM), a low-resolution X-ray diffraction technique currently being developed in our laboratory. WRSM is designed to rapidly probe a large volume of reciprocal space without requiring any prior knowledge of lattice parameters, expected texture components, or preferred orientations. The method serves as a complementary approach to conventional pole-figure measurements and powder diffraction, enabling simultaneous phase identification and texture analysis in strongly oriented thin films.

WRSM measurements were performed using a laboratory X-ray diffractometer equipped with a rotating Cu anode source. The primary X-ray beam was vertically parallelized using a parabolic mirror and further defined by a vertical slit limited to 0.5 mm. Horizontal collimation was achieved using two sequential slits, each with a width of 0.5 mm. The diffracted intensity was collected using an area detector with an active area of 38.5 × 77.5 mm^2 (vertical × horizontal) and a pixel size of 0.1 × 0.1 mm^2. The detector was positioned at a distance of approximately 15 cm from the sample and operated without secondary optics, allowing the collection of a broad angular range of diffracted signals. Both the X-ray source and detector arms moved in the vertical diffraction plane.

Reciprocal space maps were acquired in symmetric skew geometry using extended scans. In this configu-

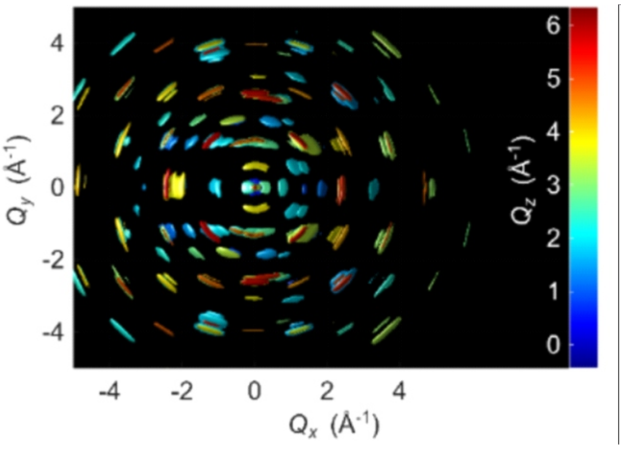

Figure 1. Top view of the diffracted intensity distribution in three-dimensional reciprocal space acquired by wide reciprocal space mapping (wRSM). The out-of-plane reciprocal-space coordinate Qz is encoded by the color scale. Each wide RSM represents a vertical reciprocal-space section containing the Qz axis. By repeating the mapping for different azimuthal angles with a fine azimuthal step, the complete three-dimensional reciprocal space can be reconstructed.

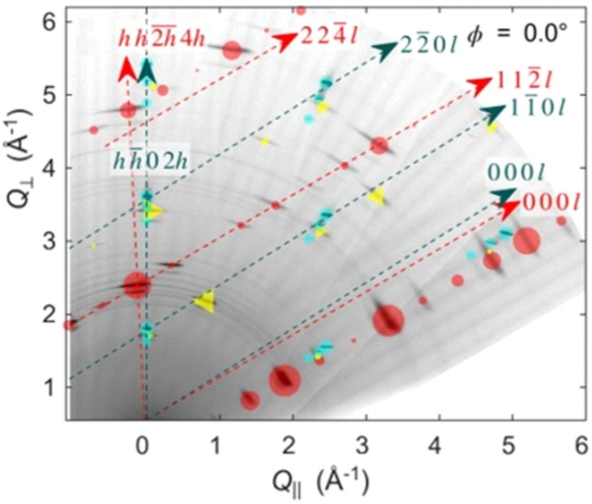

Figure 2. Representative wRSM (vertical cut through reciprocal space). Experimental data (grey scale) are overlaid with numerical simulations for the Al_2O_3 substrate (cyan spots), the M-type layer (red), and a minor hematite phase (yellow). Concentric Debye rings indicate the presence of randomly oriented crystallites within the film, whereas sharp, intense diffraction spots correspond to the dominant texture component.

ration, each scan corresponds to a straight radial trajectory in reciprocal space. By tilting the sample by an angle ÷, this trajectory becomes tilted accordingly within reciprocal space, enabling access to a wide range of orientations. During each scan, the diffracted intensity collected by the area detector was projected onto a two-dimensional stripe, effectively reducing the dimensionality of the data.

To reconstruct planar cuts of reciprocal space, scans were repeated for a sequence of sample tilt angles . The tilt step was chosen to ensure minimal overlap between adjacent stripes based on the solid angle covered by the detector; in our setup, an optimal step of 15° was used. Individual stripes obtained at different tilt angles were combined to form a two-dimensional reciprocal space map corresponding to a single sample azimuth.

To assess in-plane orientation, the planar reciprocal space reconstruction was repeated for multiple sample azimuthal rotations . An azimuthal step of 15° was sufficient to capture symmetry-related features for common substrate orientations, while smaller steps could be employed when smoother intensity reconstruction was required. The complete dataset thus provided a series of reciprocal-space sections containing the out-of-plane direction Q_z, collectively enabling qualitative and quantitative assessment of both out-of-plane and in-plane texture.

Experimental WRSM data were analyzed by direct comparison with numerical simulations of reciprocal-space intensity distributions for the substrate and possible film phases. Simulations included contributions from single-crystalline substrate reflections, textured film components, and randomly oriented crystallites, which manifest as Debye-Scherrer rings. Sharp and intense diffraction spots were associated with dominant texture components, while continuous rings indicated polycrystalline fractions.

The texture models were iteratively adjusted until good agreement between experimental maps and simulations was achieved, yielding angular resolution of the determined orientations on the order of 1°. This approach enabled reliable determination of complex and non-trivial layer-substrate orientation relationships that could not be resolved using conventional scans, high-resolution reciprocal space mapping, or pole-figure analysis.

Results

The crystallographic texture and orientation relationship of M-type hexaferrite films grown on R-cut sapphire substrates were primarily investigated using wide reciprocal space mapping (wRSM). The blindly collected full 3D diffraction data shown in Figure 1 allowed determining the phase and the texture. In contrast, conventional symmetric scans did not reveal any intense film diffraction peaks for this system, indicating only the absence of any low-index hexaferrite plane parallel to the substrate surface.

Representative wide reciprocal space map for the M-seed and M-bulk hexaferrite layers deposited on Al_2O_3 (R-cut) substrates are shown in Fig. 3. The experimentally measured diffracted intensity distributions (grey scale) were successfully reproduced by numerical simulations including contributions from the sapphire substrate, the hexaferrite layer, and a minor hematite phase. The agreement confirms that wRSM provides reliable access to the full three-dimensional reciprocal-space information in the presence of complex textures.

In the reconstructed reciprocal-space sections, diffraction-spot chains corresponding to the sapphire substrate ($h\bar{h}0l$) and the hexaferrite layer ($hh\overline{2h}l$) are clearly visible. The directions of these chains directly reflect the orientations of the crystallographic *c*-axes of the substrate and the layer. From their relative inclination, a mutual misalignment of approximately 2° between the substrate and layer c-axes was determined. This misorientation is not observable in conventional geometry but is unambiguously resolved in wRSM.

Importantly, the tilt between the nominally corresponding planes $(1\bar{1}02)$ ALO and $(11\bar{2}4)$M is directly accessible

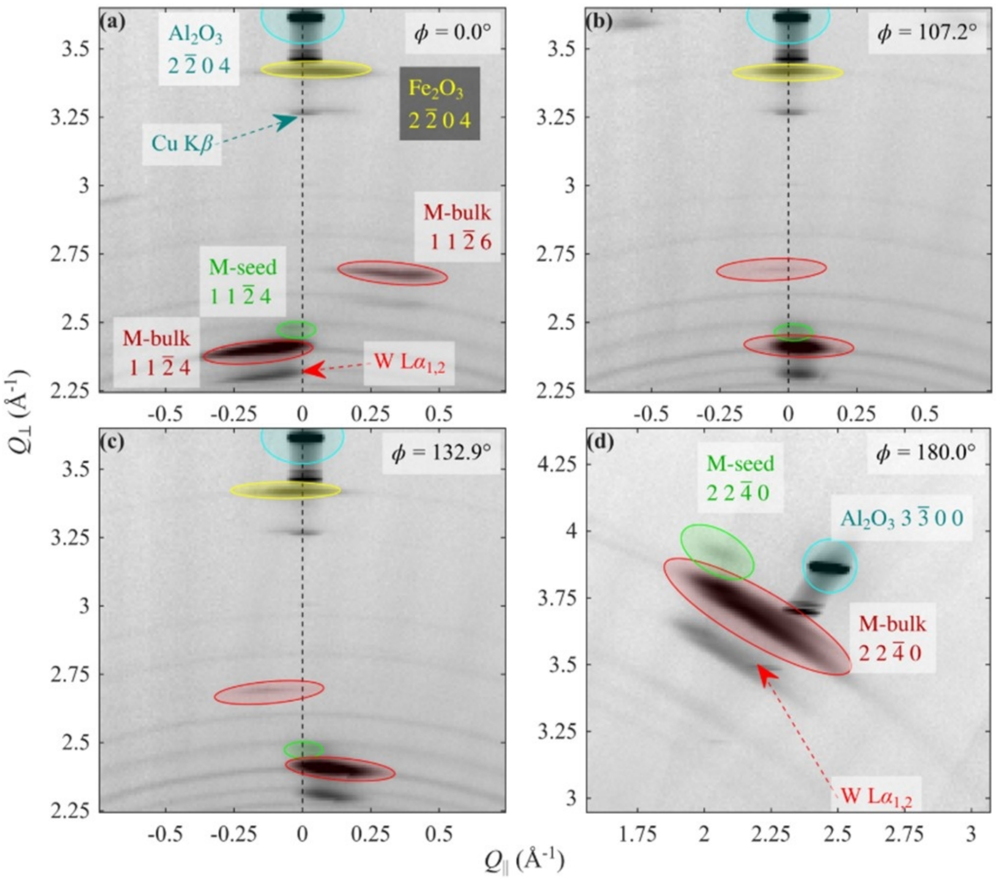

Figure 3. X-ray diffraction wide reciprocal space maps for M-seed and M-bulk layers grown on a sapphire (R-cut) single crystal substrate. The experimental data in gray color scheme are overlayed by numerical calculations for the ALO substrate (cyan spots), M-bulk layer (red), M-seed layer (green), and minor phase of hematite (yellow).

in a reciprocal-space section chosen perpendicular to the in-plane directions [11$\bar{2}$0]ALO||[1$\bar{1}$00]M, which were identified as exactly parallel from the complete wRSM dataset. The observed angular separation between the respective diffraction-spot chains corresponds to the true out-of-plane tilt of the hexaferrite lattice with respect to the substrate.

Analysis of maps recorded at different sample azimuths demonstrates that this tilt is azimuthally dependent and follows a well-defined in-plane crystallographic direction of the substrate. Magnified regions around selected layer and substrate reflections (see Figure 3) further reveal that the tilt distribution is relatively broad, with an angular spread on the order of several degrees, consistent with strong but non-ideal texture.

Pole-figure measurements carried out for the hexaferrite 11$\bar{2}$4 reflection qualitatively support the conclusions drawn from wRSM. The pole figure exhibits well-defined intensity maxima distributed along a circle corresponding to a tilt angle of approximately 5° with respect to the surface normal. This result confirms that the hexaferrite layer is simultaneously oriented both in-plane and out-of-plane, yet without any low-index plane parallel to the substrate surface.

However, due to the high number of accessible reflections and partial overlap with sapphire peaks, the pole-figure data alone are insufficient to uniquely determine the full orientation relationship. In contrast, wRSM directly provides the mutual alignment of crystallographic directions and planes without requiring prior assumptions about lattice parameters or expected texture components.

The combined wRSM and pole-figure results demonstrate that, for *R*-cut sapphire substrates, the hexaferrite layer adopts a non-trivial layer-substrate orientation. Unlike growth on *A*-, *C*-, or *M*-cut sapphire, the hexaferrite *c*-axis does not align parallel to the substrate *c*-axis, nor does the layer grow with any low-Miller-index plane strictly parallel to the substrate surface. These two requirements are geometrically incompatible in this system because of lattice-parameter mismatch and differing axial ratios, leading to competing tendencies.

Instead, the system adopts a compromise configuration characterized by a distribution of tilts ranging from 0° to 6°, where all orientation possibilities shown in Figure4a-c are populated. This tilt is rotation of the hexaferrite lattice about a specific in-plane axis ([$\bar{1}$100] direction) that remains parallel to a corresponding substrate direction ALO [11$\bar{2}$0], see Figure 4d. On the other hand, this relationship od the inplane direction seems to be fixed with misorientation bellow sensitivity limit.

Conclusion

We demonstrate that non-trivial layer–substrate orientational relationships, in which neither low-Miller-index planes nor principal crystallographic axes are parallel to

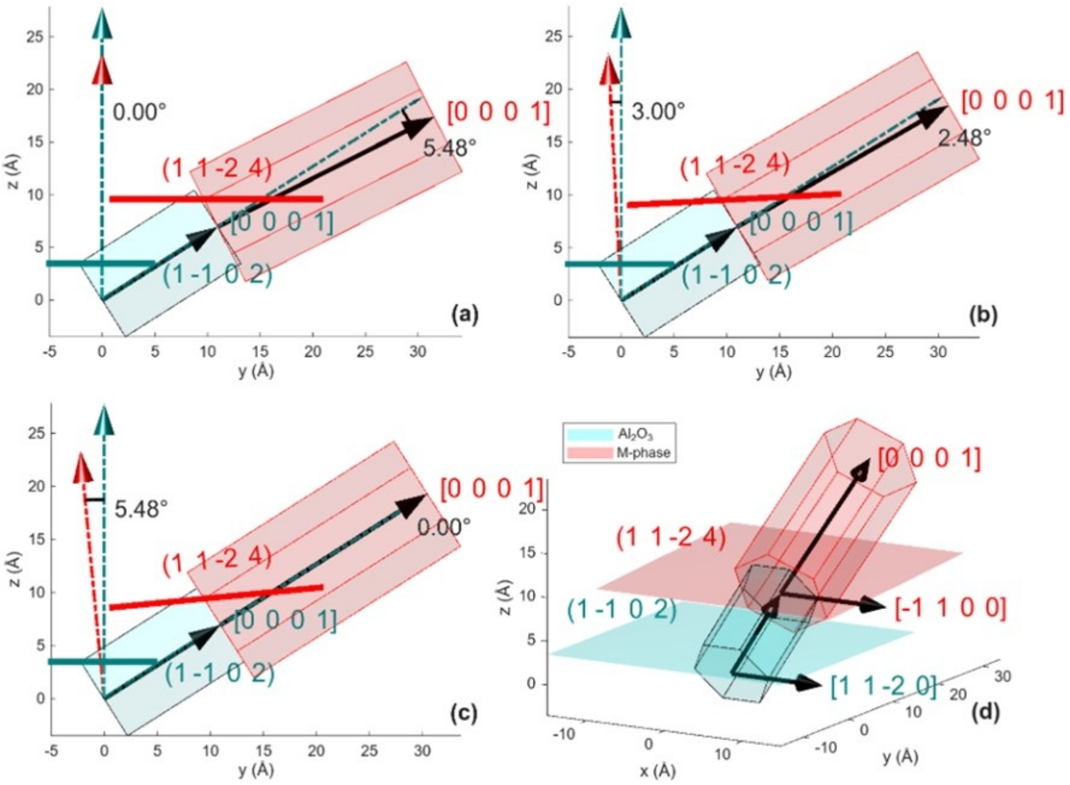

Figure 4. Visualization of the crystallographic relationship between the M-bulk layer and the substrate for different tilts of the M-phase $(11\bar{2}4)$ planes: (a) zero tilt, with M-layer M-layer $(11\bar{2}4)$ || ALO $(1\bar{1}02)$; (b) intermediate tilt, where neither the surface planes nor the basal planes are parallel; and (c) large tilt, with M-layer (0001) ||

the substrate surface, can be reliably resolved using wide reciprocal space mapping (wRSM). Strongly textured M-type hexaferrite thin films grown on R-cut sapphire serve as a representative example.

The wRSM analysis reveals that the hexaferrite layer adopts a compromise orientation characterized by a small, well-defined lattice tilt about a specific in-plane direction that remains parallel to the substrate lattice. This configuration satisfies competing geometric constraints imposed by lattice mismatch and axial-ratio differences between film and substrate, resulting in a non-trivial orientational relationship that is neither obvious nor predictable from conventional epitaxial considerations. While pole-figure measurements qualitatively confirm the presence and magnitude of the tilt, only wide reciprocal space mapping provides an unambiguous and comprehensive description of the full crystallographic relationship.

The method is particularly well suited for strongly textured or pseudo-epitaxial thin films with complex, counter-intuitive orientation relationships. As such, wRSM represents a powerful and broadly applicable tool for structural analysis of advanced thin-film materials.

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P1

MAGNETIC PROPERTIES OF Sc-DOPED M-TYPE HEXAFERRITE THIN FILMS

Eliška Spurná[1], Jakub Vít[2], Darina Smržová[3], Josef Buršík[3]

[1]Faculty of Mathematics and Physics, Charles University, Prague
[2]Institute of Physics, Czech Academy of Sciences, Praha
[3]Institute of Inorganic Chemistry, Czech Academy of Sciences, Rez

Hexaferrites, magnetic iron-containing oxides, are superior materials with respect to the large magnetoelectric effect at elevated temperatures, coming from their frustrated noncollinear magnetic structures. Common in forms of ceramics, hexaferrites are only rarely developed in forms of crystals or thin layers. Latter successfully grown predominantly with *c*-axis oriented along the film normal, which is inconvenient for applications. Their magnetoelectric properties can be stilted or further enhanced by substitutions within their crystallographic structure, subsequently altering their magnetic structure and anisotropy. In the presented study this enhancement of magnetic structure was explored by preparation of potentially magnetoelectric M-type hexaferrite in thin film form with various Sc doping and different orientations and the ensuing measurements of magnetic properties. The Fe positions in the M-type $SrFe_{12}O_{19}$ are partially substituted by Sc atoms causing a noncollinear conical magnetic structure to form in place of the original collinear magnetic structure. The thin film samples of $SrFe_{12-x}Sc_xO_{19}$ were prepared by chemical solution deposition method on Al_2O_3 single-crystal substrate. The measured temperature-dependent magnetic anisotropy is similar to that of magnetoelectric ceramics with $x(Sc) = 1.5$, which suggests a possibility to observe a magnetoelectric effect in the prepared films.