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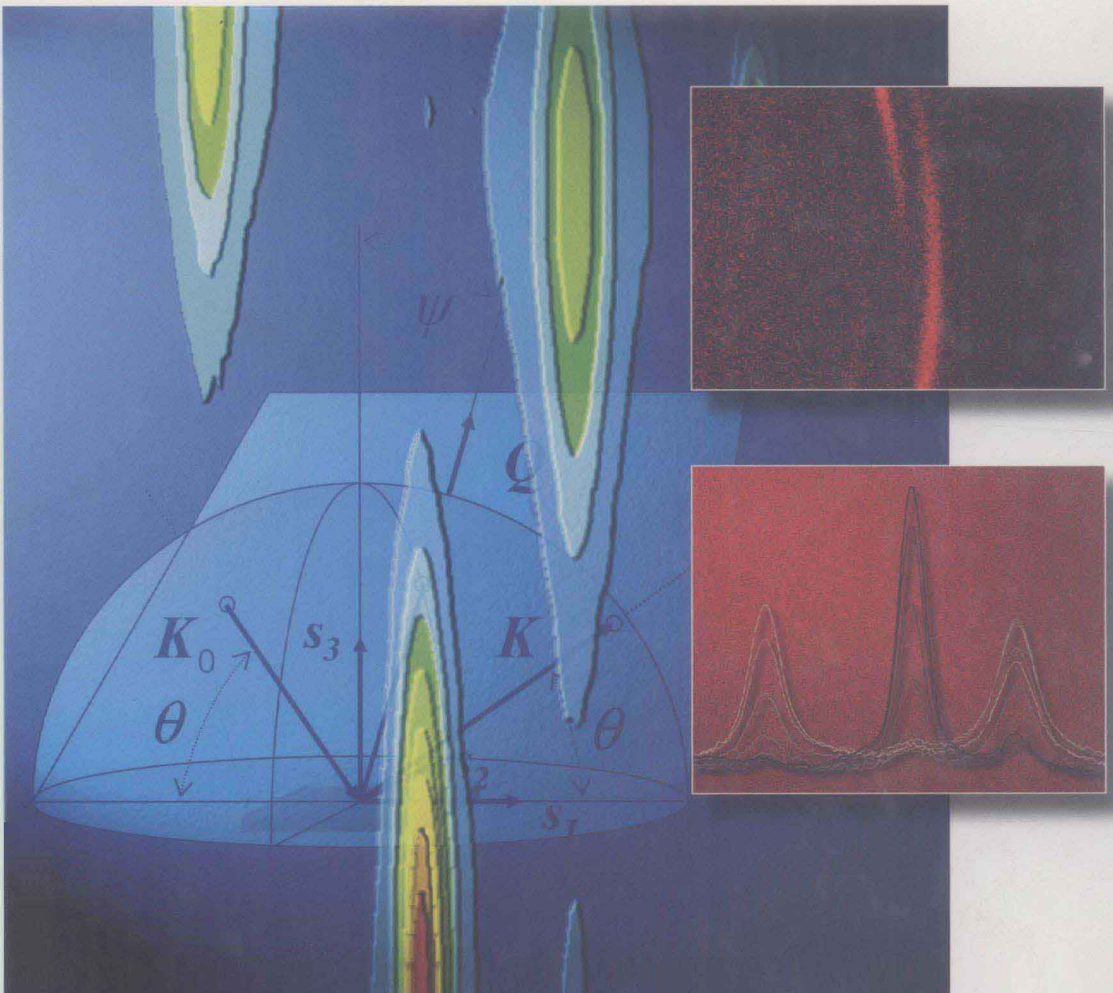
Thin Film Analysis by X-Ray Scattering



Thin Film Analysis by X-Ray Scattering

Mario Birkholz

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Preface

Mario Birkholz

Thin films have become an important branch of materials science and technology over the last few decades. A thin film is considered in this book as having a thickness between about 1 nm and some 10 μm. Their first application was probably in the field of decorative coatings, but in the last century many other applications in microelectronics, optics, data storage, sensorics, protection and other purposes have had a large impact on the development of thin films and related deposition techniques. Figure 1 displays a variety of thin-film applications from the areas mentioned. Depending on the intended application, thin films are made of metals, inorganic compounds, organic compounds or from biological molecules. The task of the thin-film developer is easily described by stating that the deposition process has to be optimized such that the arrangement of atoms enables the film to fulfill the intended functionality.

Since structure and function are intimately related properties in any material, the characterization of structural properties is a very relevant issue in thin-film development. This book is concerned with the structural analysis of thin films by x-ray scattering procedures. There exist various other characterization techniques like electron microscopy, scanning tunneling methods, ion beam scattering, magnetic resonance, optical spectroscopy and others by which important structure properties may be elucidated. Here, however, the focus is on x-ray scattering. The suitability of this technique for thin-film analysis is mainly motivated by two reasons:

1. The wavelengths of x-rays are of the order of atomic distances in condensed matter, which especially qualifies their use as structural probes.
2. X-ray scattering techniques are nondestructive and leave the investigated sample or – more importantly – the produced device intact.

Electron microscopy might be considered of comparative importance for the characterization of structure and morphology. This technique is a complementary one to x-ray scattering, since it probes a rather confined volume of the sample, whereas x-ray scattering yields information from a much larger volume. Therefore, some micrographs from electron microscopy will appear in the text, but the reader is referred to the special literature for an introduction to the subject.

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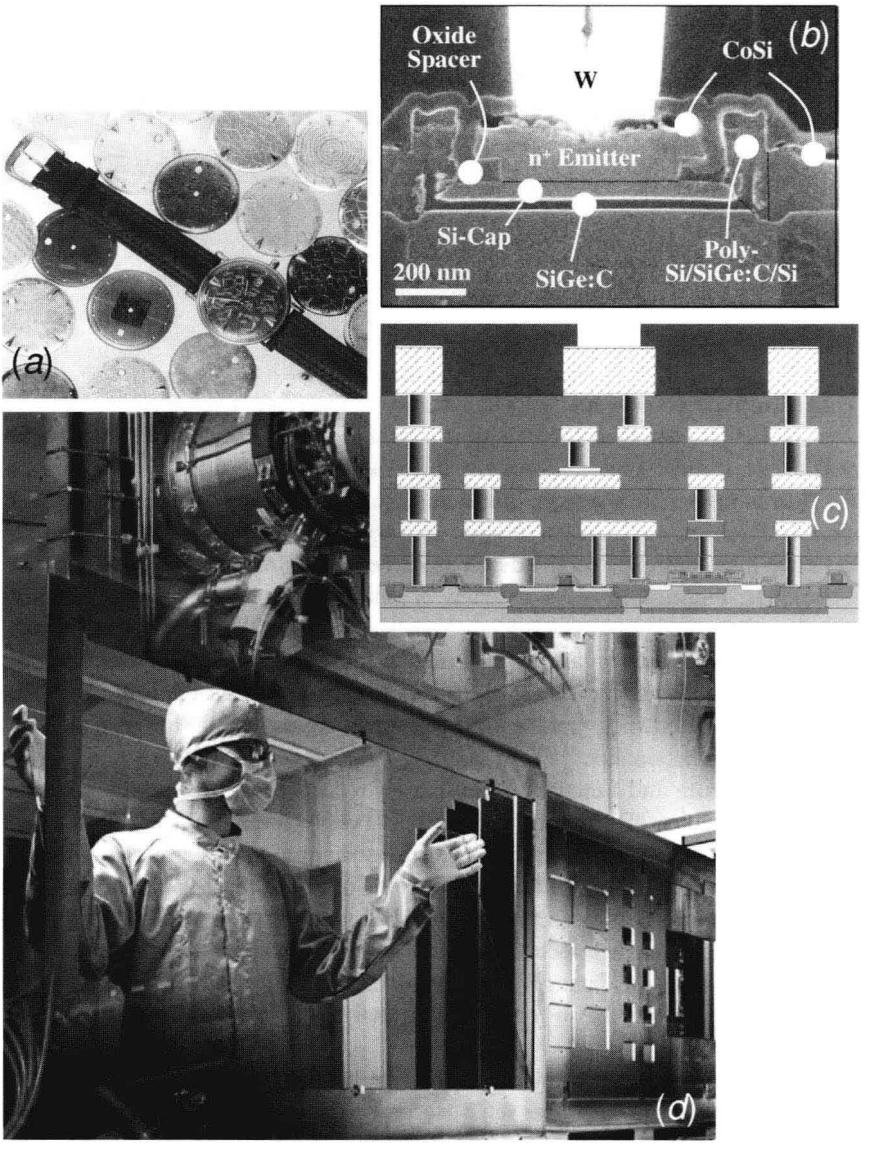
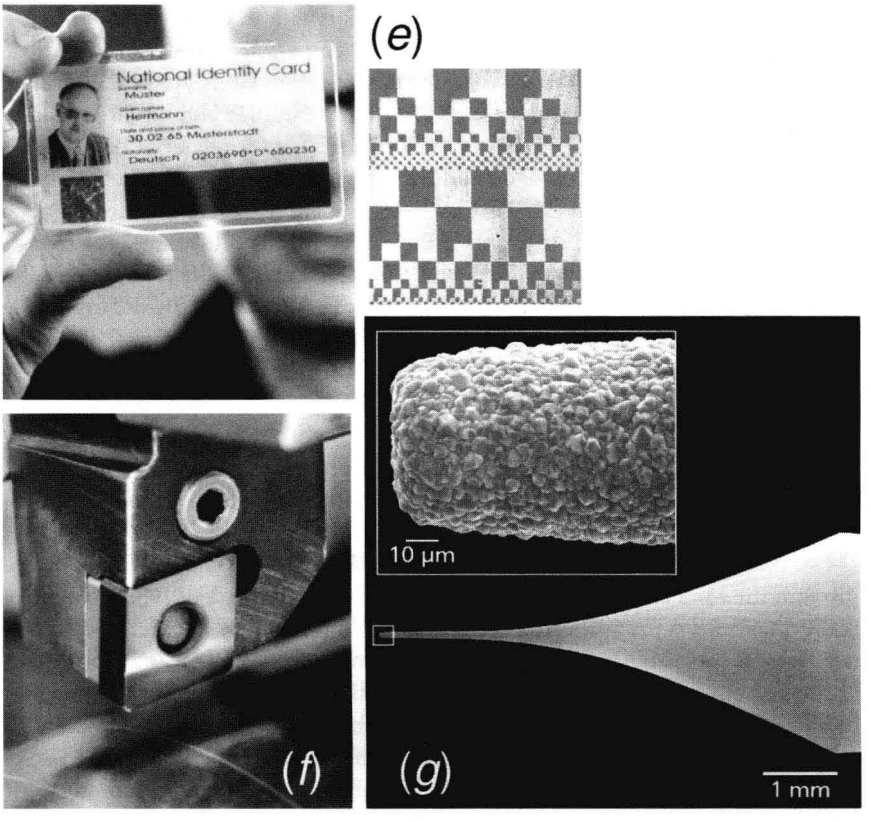


Various examples from thin film applications: (a) decorative coatings on metal dials, (b) scanning electron micrograph of cross-section from SiGe:C heterojunction bipolar transistor, (c) schematic of SiGe:C BiCMOS architecture with four metal layers, (d) processing of window glass for optical coating (Fotograf: Rainer Maier, BFF, Wittmar), (e) identity card with optical data storage made from bacterial purple membrane containing the protein bacteriorhodopsin and example of stored pixel patterns, (f) cemented carbide cutting insert with c-BN protective coating and (g) tip of a diamond-coated abrasive pencil of 60 µm diameter (figures kindly provided by (a, d, f and g) Fraunhofer IST, Braunschweig [1], (b, c) IHP, Frankfurt (Oder) [2], (e) Prof. N. Hampp, University of Marburg [3]).



This book is intended to give overviews of the relevant x-ray scattering techniques for thin-film work and to equip scientists and engineers with the basic understanding to apply them. It has to be emphasized that for each x-ray technique presented in one of the following chapters there are authoritative and comprehensive textbooks available; these are listed at the end of each chapter and the reader is referred to them for further consultation. It seems, however, that there exists a gap between the highly developed and complex structural sciences on the one hand and the daily needs of materials scientists on the other. Many of the conclusive, effective and powerful techniques that have been developed for structural investigations appear to be not as extensively used in thin-film technology as they would deserve. It is the aim of this book to bridge this gap by introducing the concepts of x-ray techniques that appear most interesting to elucidate the close relations between structure, function and growth of thin films.

Chapter 1 introduces the basic phenomenon of x-ray diffraction by a crystalline lattice. In Chapter 2 methods for the identification of chemical phases are presented. Chapter 3 is related to the line profile analysis of diffraction peaks with respect to film microstructure. Measurement geometries characterized by a grazing incident x-ray beam are introduced in Chapter 4. The preferred orientation of crys-

tallites and residual stresses in thin films are dealt with in Chapters 5 and 6, respectively. Up to this point mostly polycrystalline films will be considered and use will be made of the kinematic theory only. Epitaxial thin films are in the focus of Chapter 7, where high-resolution x-ray diffraction is outlined and the first grounding of dynamical theory is introduced. The majority of the material presented is based on the physical phenomenon of diffraction, but some parts – as for instance the presentation of reflectometry in chapter four – are related to the more general phenomenon of x-ray scattering. This is the reason for the title of the book.

It is recommended to start reading with Chapters 1 and 2, which might be helpful even for those readers to whom the basics are already known in order to become familiar with the conventions and notation used. After this introductory training the reader may consult any other chapter presenting the method that might be expected of relevance for his or her actual work. The emphasis of the book is on x-ray scattering with laboratory setups in contrast to synchrotron radiation beam lines. However, many of the measurement concepts presented are equally realized at synchrotron facilities and may also be applied in experiments with the much higher intensity available at synchrotron sources.

Two concepts or quantities meander through the following chapters like a thread through the tows of the former British royal navy ("roter Faden" [4]). The first of these quantities is the scattering vector that is abbreviated by $\boldsymbol{Q}$ here. The scattering vector is met in almost every chapter since interatomic distances are probed by diffraction only along the direction of $\boldsymbol{Q}$. Diffraction or scattering experiments may be considered as intensity mappings under complex rotations of the sample with respect to the scattering vector. These reorientations are dealt with by the use of three reference frames $\{\boldsymbol{l}_i\}$, $\{\boldsymbol{s}_i\}$ and $\{\boldsymbol{c}_i\}$, one for each frame of the laboratory, the sample and the crystallographic unit cell. The different frames are sometimes confusing for the newcomer to the field. It is recommended that when one seems of having lost the "roten Faden" it might be taken up again by answering the question "what are the coordinates of the scattering vector $\boldsymbol{Q}$ within the respective reference frame?"

The second recurring quantity is that of the x-ray attenuation coefficient μ. In condensed matter x-rays are attenuated on a length scale of some 10 to some 100 µm. These penetration depths are accordingly often larger than the film thickness t and special methods have been developed to restrict the probing beam to the sample volume. In almost all of the forthcoming chapters we have to derive how the μt product affects the measured scattering intensities. It may even be stated that the μt product can be regarded as the central physical quantity in thin-film analysis by x-ray scattering.

The chapters end with an application section, where studies and works related to the issue of the chapter are presented. The selection of these examples and those mentioned in the main body of the text is probably highly selective and reflects the interest and working areas of the author(s). Since each chapter covers a large field of research activities it was hardly possible to overview fully the many interesting x-ray scattering investigations that have been carried out in the appropriate areas. A collection of exercises is given at the end of each chapter, by the solution of which

the reader may verify the understanding of the text. Solutions to the exercises can be found on the internet [5].

Two further issues are considered in parallel with the main text. These are related, firstly, to the instrumentation in x-ray scattering experiments and, secondly, to the structure of selected material classes. In the instrumentation boxes, the instrumentation required by the experiments described in the chapter is detailed. The structure boxes present crystallographic structures, structural parameters and selected physical properties of relevant materials. The material systems have been chosen in accordance with their relevance to illustrate the x-ray scattering technique in the chapter. The selected material systems are in order of increasing chapter number metals, semiconductors, nanocomposites, optical thin films, dielectric and superconducting materials, hard coatings and finally semiconductors for micro- and optoelectronics. Other combinations would have equally been possible.

Two remarks have to be made on notation: (a) SI units have been used throughout the text and (b) it has been endeavored to use a consistent notation throughout the text. However, this turned out a difficult task, since every chapter covers a highly developed subfield of x-ray scattering with its own nomenclature. It could thus not be completely avoided to make use of the same symbol with different meaning in different chapters. These cases will explicitly be pointed out. In case of doubt, the appropriate meaning of a symbol can be identified by consulting the symbol list.

In some cases, the names of inventors or scientific pioneers are mentioned. It should be borne in mind, however, that scientific achievements always rely on the communication among different researchers exchanging their ideas and imaginations. This statement is illustrated by the famous discussion between P.P. Ewald and M. Laue that laid the basis for the first x-ray diffraction experiments by Friedrich, Knipping and Laue [6]. Scientific progress has always been based on teamwork, even if the protagonists did not know each other personally. This fact is explicitly stated here, since we cannot be sure in every case that all the researchers that should be credited were adequately indicated when one or more of them are mentioned. Since x-ray diffraction is about 100 years old, it may be possible that future research in the history of science will reveal personal contributions of which we were not fully aware at the time of writing. The reader interested in the early history of x-ray diffraction is referred to a paper collection published for the International Union of Crystallography [7].

This book project would not have become reality without the help of some friends and colleagues. Firstly, I would like to thank Paul Fewster and Christoph Genzel for their co-authorship of Chapters 6 and 7. They both agreed on being co-authors to chapters they would be much more qualified of writing themselves and let me assume the role of the first author in order to maintain consistency with the rest of the book. Their great expertise in the respective fields helped enormously to formulate these two state-of-the-art chapters. I enjoyed the work with both of them very much. I am indebted to Daniel Chateigner, Carl Krill, Paolo Scardi, Thomas Schröder, Antonella Tagliente, Mark Vaudin, Thomas Wieder, Don Williamson, Joachim Woitok and Peter Zaumseil, who carefully red draft versions of single chapters, gave valuable recommendations and pointed me to some examples from

the work of their own and others. Moreover, I like to thank those colleagues, who permitted the reprint of figures from their publications, which are (in addition to those already mentioned) J. Almer, U. Balachandran, M. Deutsch, J. Driscoll, J. Gubicza, N. Hampp, K. Helming, Y. Iijima, D. Knoll, S. Kondruweit, L. Lutterotti, M. A. Meyer, C. Michaelsen, M. Morales, A. Navarro-Quezada, S. Peist, A. J. Perry, M. Li, R. P. Reade, Á. Révész, J. Ricote, A. Saerens, K. Saito, K. Schiffmann, S. Sun, H. Suo, T. Ungár, J. J. Wells and E. Zschech. Their work on thin film analysis will probably be very helpful to the reader by illustrating the theoretical derivations with real-world examples. Thanks are also to Jürgen Altmann and Wulff Pfeiffer for their support of this project, to the companies AMD, Bruker AXS, IfG, PANalytical, Seifert for providing pictures from their thin film or x-ray equipment products, to Cyrille Boudias and Daniel Monceau, who draw the crystallographic figures in the structure boxes by virtue of their software package CaRIne Crystallography [8] and to the Wiley-VCH Verlag, who followed all my suggestions in preparing an editing the manuscript. Most of all, I would like to thank my wife, Johanna, for her patience and continuous support of this project.

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Symbols

Symbols consistently used throughout the book are listed in the sequence of appearance in the chapters.

Chapter 1

2θ	scattering angle
α, β, γ	angles between edges of crystallographic unit cell
δ	divergence of x-ray beam
ε_0	permittivity of a vacuum
$\phi_{\boldsymbol{h}}$	phase of diffracted plane wave
λ	x-ray wavelength
μ	linear x-ray attenuation or absorption coefficient
μ_{m}	mass attenuation coefficient
μt	product of attenuation coefficient and film thickness
σ, π	indices to denote perpendicular and parallel polarized beam components
ρ	mass density
ρ_{e}	electron density
$\tau_{1/e}$	$1/e$ penetration depth
θ	Bragg angle
$\theta_{\mathrm{B}}, 2\theta_{\mathrm{B}}$	Bragg angle and scattering angle of a Bragg reflection
$\{\boldsymbol{c}_i\}$	crystal reference frame of individual crystallites (superscript C)
$\{\boldsymbol{l}_i\}$	laboratory reference frame (superscript L)
$\{\boldsymbol{s}_i\}$	specimen reference frame (superscript S)
a, b, c	lattice parameters or cell edges of crystallographic unit cell
a_{ij}^{XY}	transformation matrices from reference frame X to Y (with $X, Y =$ C, L, S)
$\boldsymbol{b}_i$	unit cell vectors of reciprocal lattice
A	absorption factor in XRS experiment
$A(2\theta)$, $A_{\theta/2\theta}$	absorption factor for symmetric $\theta/2\theta$ experiment
B	isotropic temperature damping factor

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c	velocity of light in vacuum
C	polarization factor
$\overline{C^2}$	average polarization factor
Cu Kα	x-ray radiation from copper source (Kα line)
e	electron charge
d	interplanar spacing between crystallographic lattice planes
E	energy of radiation
$\boldsymbol{E_0}$, $\boldsymbol{E}$	electrical field vector of incoming and scattered beam
f	atomic form factor
f'	real part of anomalous scattering factor
f''	imaginary part of anomalous scattering factor
$\boldsymbol{F}$, $\boldsymbol{F^*}$	structure factor and its complex conjugate
G	geometry factor
$\boldsymbol{h}$	abbreviated form of Miller indices *hkl*, also used as subscript
hkl	Miller indices of Bragg reflection from (*hkl*) lattice planes
(hkl), $\{hkl\}$	Miller indices of crystallographic lattice plane
$[hkl]$, $\langle hkl \rangle$	Miller indices of crystallographic direction
i	imaginary unit
$\Im$	interference function
I	x-ray intensity
I_0	intensity of the incident beam
I_B	maximum intensity of x-ray reflection at Bragg angle θ_B
$I_{\boldsymbol{h}}$	integrated intensity of a Bragg reflection from (*hkl*) lattice planes
k	geometry factor for XRS experiment
$k(2\theta)$, $k_{\theta/2\theta}$	geometry factor for symmetric $\theta/2\theta$ scan
$\boldsymbol{K_0}$	wave vector of incoming x-ray beam outside the sample
$\boldsymbol{K}$	wave vector of scattered x-ray beam outside the sample
ℓ	path length of x-ray in the sample
L	Lorentz factor
Lp	Lorentz polarization factor
m_{hkl}, $m_{\boldsymbol{h}}$	multiplicity of a Bragg reflection *hkl* in a powder pattern
m_0	rest mass of an electron
n	order of reflection in Bragg equation
N	number of unit cells in a crystallite
n_1, n_2, n_3	integers to denote the position of a unit cell in a crystallite
N_1, N_2, N_3	number of unit cells of a single crystallite along its orthogonal directions
N_L	Loschmidt's number
P	power of scattered x-ray beam
$\boldsymbol{Q}$	scattering vector
r	atomic distance or bond length
$\boldsymbol{r_{n_1 n_2 n_3}}$	distance vector from the origin of a crystallite to unit cell of number (n_1, n_2, n_3)
r_e	classical radius of the electron
R	goniometer radius

R_{FC}	radius of the focusing circle
SCF	scaling factor for x-ray intensity received by the detector
S	surface
$\boldsymbol{s}_3$	substrate normal vector
t	thickness of thin film
t'	time
T	temperature
$T_{\boldsymbol{h}}$	texture factor of $\boldsymbol{h}$th reflection
$\overline{u^2}$	mean-square oscillation amplitude due to thermal vibrations of the atom
V	irradiated sample volume
V_{uc}	unit cell volume
x_n, y_n, z_n	fractional coordinates of nth atom in the unit cell
z	thin film depth coordinate
Z	number of electrons of an atom

Chapter 2

α_1, α_2	subcomponents of α x-ray emission line
δ_a	x-ray beam divergence along the θ and 2θ axis (axial divergence)
δ_p	x-ray beam divergence in the scattering plane (equatorial divergence)
δ_{ds}	opening angle of divergence slit
δ_{rec}	opening angle of receiving slit
δ_{Sol}	opening angle of Soller slits
γ	angle between scattering vector and bond vector
ϕ	azimuth, i.e. angle of in-plane sample rotation
σ	cross-section for x-ray scattering
θ_0	centroid of Bragg reflection
$(\Delta 2\theta)_i$	instrumental shifts of centroid from Bragg peak position
a_0, a_1	coefficients in the linear expansion of unit cell edge a
b	height misalignment of sample surface
B_a, B_p	extension of x-ray foot step on the sample along axial and in-plane directions
j	numeration of layers in a multilayer stack
J	number of layers in a multilayer stack
M	atomic mass
S	subscript to denote quantities related to a substrate
T_{dep}	deposition temperature
T_{hom}	homologous temperature
T_{mel}	melting temperature
TF	subscript to denote quantities related to a thin film
w_j	weight of the jth phase in a thin-film sample
W	total weight of a thin film
x	stoichiometry coordinate of a binary compound $A_{1-x}B_x$

Chapter 3

β	integral breadth of Bragg reflection
$\beta_{2\theta}$	integral breadth on scattering angle scale
β_Q	integral breadth on the scale of momentum transfer
γ	variance of crystallite sizes in lognormal distribution function
Γ_{hkl}	ratio of the permutation invariants of fourth order polynomials of Miller indices
ε	lattice strain
$\varepsilon_{\mathrm{rms}}$	root-mean square strain
$\widetilde{\varepsilon}$	weighted average strain
λ_1, λ_2	wavelengths of subcomponents of α x-ray emission line
θ_S, θ_E	start and end value of scattering angle range
ρ_d	dislocation density
η	weight parameter of pV profile function indicating the Cauchy fraction
$2w$	full width at half maximum
A_n, $A(n)$	coefficients of Fourier expansion
A^S, A^{size}	coefficients of Fourier expansion accounting for size broadening
A^D, A^{dis}	Fourier expansion coefficients accounting for broadening by lattice distortions
$\boldsymbol{b}$	Burger's vector to describe geometrical structure of dislocation
$B(2\theta)$	background function in whole pattern fitting
C	Cauchy profile function
C_{hkl}	dislocation contrast factor
$\bar{C}_{hkl}$	average dislocation contrast factor
d_0	position of the centroid of a Bragg peak
D	crystallite size parameter
D_0	crystallite size median in lognormal distribution function
D_{cub}	length of a cube-shaped crystallite
D_{sph}	diameter of a spherical crystallite
$\langle D^n \rangle$	nth statistical moment of crystallite size distribution
$\langle D \rangle_A$	area-weighted average crystallite size
$\langle D \rangle_V$	volume-weighted average crystallite size
f	sample line profile free from instrumental effects
$\mathcal{F}(y(x))$	Fourier transform of function $y(x)$
g	instrumental line profile
$g(D)$	crystallite size distribution function
$g_{\mathrm{LN}}(D)$	logarithmic normal distribution function of crystallite size
G	Gauss profile function
G_n, $G(n)$	coefficients of Fourier expansion of standard peak
h	convolution of instrumental and sample-broadened line profile
H_n, $H(n)$	coefficients of Fourier expansion of measured sample peak
I_0	maximum peak intensity

k	parameter in Voigt profile function
K_S	Scherrer constant
K_D	scaling factor in Williamson–Hall relation and derivatives
L	correlation length
$\langle L^n \rangle$	nth statistical moment of column height distribution
m	parameter in Pearson profile function
M	dislocation configuration parameter
N	number of data points subjected to a Fourier transformation
N_3	number of unit cells in a unit cell column
pV	pseudo Voigt profile function
P	Pearson (VII) profile function
$p(L)$	column height distribution function of the sample
$P(L)$	column height distribution function of one crystallite
R	radius of spherical crystallite
R_c	core radius of dislocation
R_e	effective outer cut-off radius of dislocations
R_w	weighted R factor
R_{21}	intensity ratio of α_2 over α_1 intensity
V	Voigt profile function
$\boldsymbol{u}(\boldsymbol{r})$	displacement field or distortion field
$y(2\theta)$	model function of intensity in whole pattern fitting

Chapter 4

α, α_i	incidence angle of incident x-ray with respect to sample surface
α_f	exit angle of diffracted x-ray with respect to sample surface
α_c, θ_c	critical angle for total external reflection
α_t	angle of transmitted x-ray with respect to surface plane
β	negative imaginary part of x-ray refractive index
δ	negative deviation of real part of x-ray refractive index from unity
$\Delta(2\theta)$	shift of Bragg peak position due to refraction
k_α	geometry factor for GIXRD experiment
θ_m	maximum of Kiessig fringes in reflectivity curve
σ	root mean square roughness of film surface or interface
ρ_a	density of atoms
$\tau_{1/e}$	penetration depth
τ_{63}	penetration depth due to scattered intensity
$\overline{\tau}$	average information depth in thin film of finite thickness
$\overline{\tau}_\alpha$	average information depth in GIXRD
A_α	absorption factor for GIXRD experiment
B_+, B_-	real and negative imaginary part of α_t
E_t	amplitude of transmitted electrical field
I_R	intensity of the reflected beam
$\boldsymbol{k}$	wave vector of x-rays within the sample

$k_{z,j}$	z component of wave vector in the jth layer of a multilayer stack
$\mathfrak{L}$	Laplace transform
m	order of Kiessig fringe
$\boldsymbol{M}$	transfer matrix in XRR
n	real part of index of refraction
$\hat{n}$	complex index of refraction
$P(z)$	depth-dependent property
$\overline{P}$	property averaged over depth
$\overline{P}_{\alpha}$	depth-averaged property in GIXRD
$\boldsymbol{R}_{j,j+1}$	refraction matrix in XRR
$r_{j,j+1}$	reflection coefficient between the jth and $(j+1)$th layer in a multilayer stack
t_{per}	superlattice period
$\boldsymbol{T}_{j,j+1}$	translation matrix in XRR
$z(x, y)$	height function of sample surface
$\overline{z}$	average sample height

Chapter 5

$\Delta\psi$	tilt in crystallite orientation distribution, i.e. FWHM of $I_{\boldsymbol{h}}(\psi)$
$\Delta\phi$	twist in crystallite orientation distribution, i.e. FWHM of $I_{\boldsymbol{h}}(\phi)$
$\varphi_1, \Phi, \varphi_2$	Euler angles for the S $\rightarrow$ C transformation
ω	sample rotation angle on Θ axis
k_{ψ}	geometry factor for pole figure measurement
k_{ω}	geometry factor in Ω mode
$\psi_{1/2}$	FWHM of volume share of fiber fraction
ψ	tilt angle or polar angle
$\overline{\tau}_{\psi}$	average information depth in ψ scan
A_{ψ}	absorption factor in Ψ mode
A_{ω}	absorption factor in Ω mode
C_l^{μ}	coefficients in linear expansion of inverse pole figure
C_l^{mn}	coefficients in linear expansion of harmonic ODF
$C_l^{\mu\nu}$	linear coefficients in symmetry-adapted ODF expansion
$\boldsymbol{d}$	direction vector
$f(g)$	orientation distribution function
$f(\varphi_1, \Phi, \varphi_2)$	orientation distribution function
$F_l(\boldsymbol{h})$	weight factors in $I_{\boldsymbol{h}}(\psi)$ expansion
$F_e^{\nu}(\boldsymbol{h})$	pole figure coefficients of $\boldsymbol{h}$th reflection
g	orientation of a crystallite with respect to the sample reference frame
G	volume fraction of the textured phase (March model)
$I_{\boldsymbol{h}}(\psi)$	intensity distribution of Bragg reflection $\boldsymbol{h}$ for varying tilt angles
$I_{\boldsymbol{h}}(\phi)$	course of intensity of Bragg reflection $\boldsymbol{h}$ for varying azimuths
$I_{rc}(\omega)$	intensity distribution in a rocking curve scan
J	texture index

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