Combined Analysis: structure-texture-microstructure-phase-stresses-reflectivity determination by x-ray and neutron scattering

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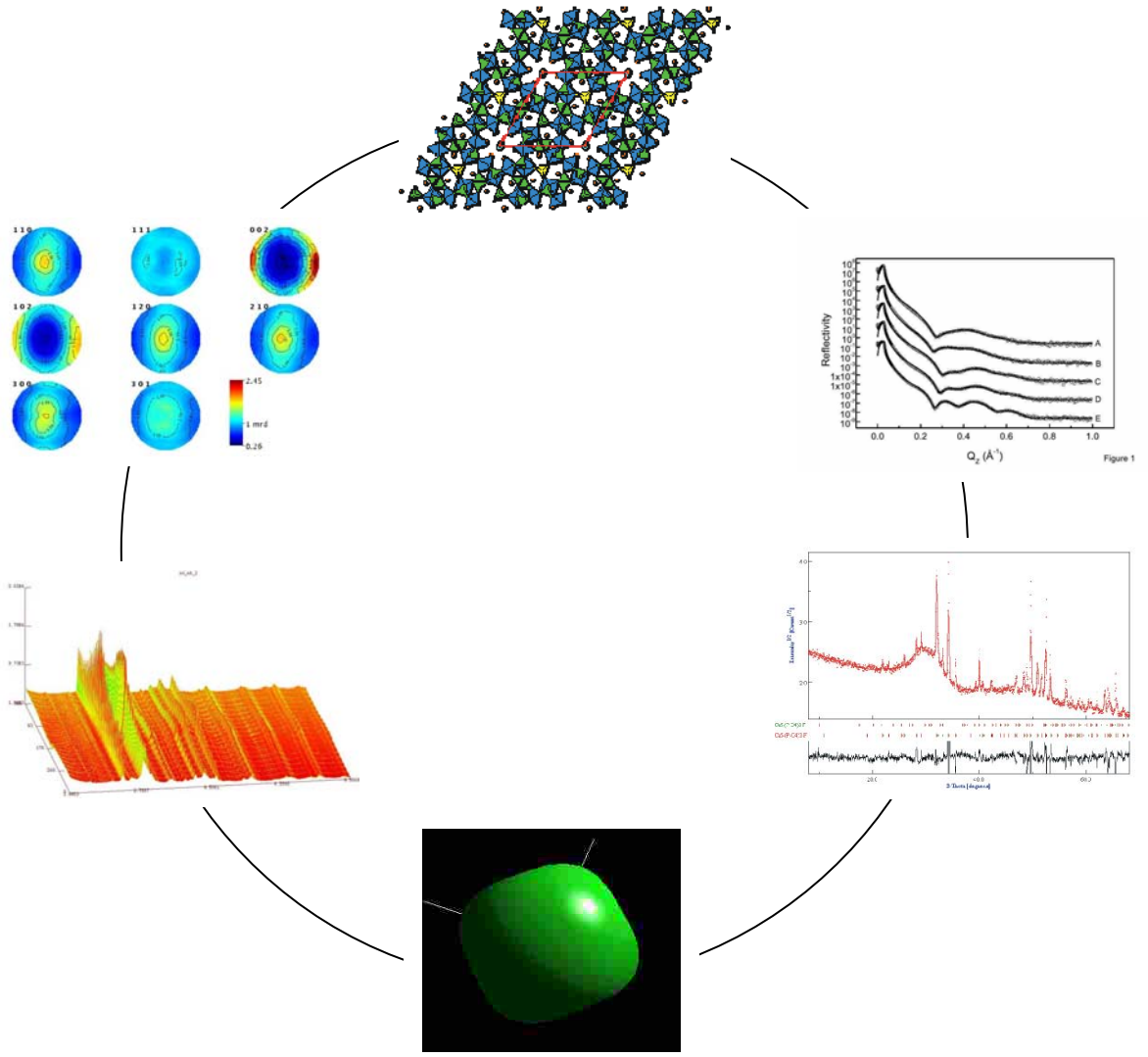

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Combined Analysis: structure-texture-microstructure-phase-stresses-reflectivity determination by x-ray and neutron scattering

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http://www.ecole.ensicaen.fr/~chateign/texture/combined.pdf

Some typographical mistakes may have been introduced throughout this document.
Suggestions and corrections are very welcome.

0 Introduction

Solid state chemistry and technology recent developments gave rise to the necessity of intensive structural analysis from single crystal diffraction. However for many solids, single crystal growth is not easy to manage and sometimes impossible. When this is the case, or when structural defects cannot be overcome, the corresponding phases have often been forsaken, due to the inherent difficulties to carry out crystallographic characterisations on polycrystals. But in the last decades powder diffraction techniques progressed significantly, notably due to the Rietveld approach (Rietveld, 1969) and computer science developments. Undoubtedly these developments are of prior importance in the study of solids that do not form large crystals, but also of all materials elaborated by classical solid state reactions, thin deposited structures, natural materials like clays and more recently nanomaterials in which the required properties are intimately linked to the stabilisation of small crystals.

Since the Rietveld method's birth, several ten thousands of structures have been refined and some thousands have been resolved *ab-initio* from the only diffraction data of powder samples. The number of laboratories and industries using this technique, still fairly new when dealing with the incorporation of various formalisms like in the combined approach, does not stop increasing.

However, materials having specific properties are often elaborated from low symmetry phases, which are consequently anisotropic. Property's optimisation is then conditioned by the elaboration processes which have to keep the intrinsic microscopic anisotropy of the constituting crystals at the macroscopic level. These elaboration techniques are complex (alignment under uniaxial pressure, magnetic or electric fields, thermal gradients, flux or substrate growing ... and combinations) and often sample preparation is a hard, time consuming, matter. Naturally, non-destructive characterisations are then required. Unfortunately, when samples are oriented, which was not often the case until recently, most of the characterisation techniques (as the Rietveld analysis of concerns here) require samples grinding. Very often this grinding is not acceptable, for the previously described reasons, but also in the case of rare samples (fossils, comets ...) or simply when grinding modifies the physical behaviour of the samples themselves (thin films, residual stress materials ...). Sometimes grinding is simply not possible, imagine peeling off a 10 nm thick film on a substrate !

In all these cases, the combined analysis becomes essential.

The first part of this document is dedicated to some basic notions concerning diffraction by polycrystals. The various peak profiles used are described and some, most common combined analysis instrumental set-up detailed.

In the second part, powder diffraction data treatment is introduced. In particular, the Rietveld analysis is detailed, including treatment of all the information provided by diffraction diagrams, when texture is not present in the sample or simple to treat.

The third part deals with the automatic phase indexing, necessary step to solve a structure *ab-initio*.

Since its effect prevails on real samples where textures are often stabilised, quantitative texture analysis is detailed in the fourth part.

The fifth part is dedicated to microstructural aspects (isotropic and anisotropic crystal sizes and microdistortions) of the poder diffraction profiles.

In part six, quantitative phase analysis from Rietveld analysis is introduced.

Part seven describes residual stress analysis for isotropic and anisotropic materials.

The eighth part focuses on specular x-ray reflectivity and the various models associated.

Part nine introduces the combined analysis concept, showing all the dilemma that show up when one looks at only one part of the analyses, and case examples are shown as illustration of the methodology.

The 10^{th} part is dedicated to the anisotropic and tensorial macroscopic properties and their simulations to account for the distribution of crystallite orientations in samples.

This book is not intended to provide the reader a complete description of all the approaches dealt within it, though quantitative texture analysis is more deeply detailed than others since texture appears to be the largest signal biaser, but a red wire to follow the many concepts introduced through so many years and necessary to understand scattering patterns.

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Glossary

ρ	Material density
ρ_e	Material electron density
dS	Surface element of the Pole Sphere
a, b, c, α_c, β_c, γ_c	Unit-cell parameters
a, **b**, **c**	Unit vectors of the unit-cell
Δk	Scattering vector
n	Normal to the sample surface
$\mathcal{S}$	Spectrometer (Diffractometer) space
$\mathcal{S}$p	Physical 3D space
$\mathcal{S}$e	External (reciprocal) 3D space
$\mathcal{S}$i	Internal (reciprocal) superspace orthogonal to $\mathcal{S}$e
χ	Polar angle in the diffractometer space
φ	Azimuthal angle in the diffractometer space
$\mathcal{Y}$	Pole figure space
ϑ_y	Polar angle in the pole figure space
φ_y	Azimuth of pole figures
hkℓ	Miller indices
(hkℓ)	Crystallographic plane hkℓ
{hkℓ}	Crystallographic planes hkℓ and diffracting equivalents
[hkℓ]	Crystallographic direction hkℓ
[hkℓ]*	Crystallographic direction hkℓ of the reciprocal space
<hkℓ>	Crystallographic direction hkℓ and diffracting equivalents
<hkℓ>*	Crystallographic direction hkℓ and diffracting equivalents of the reciprocal space
$L_{hk\ell}$	Lotgering factor
p, p_0	ratio entering the Lotgering factor for a textured and a random sample respectively
h	<hkℓ>* directions
y	ϑ_y, φ_y direction in $\mathcal{Y}$
$I_{\mathbf{h}}(\mathbf{y})$	Direct pole figure
$P_{\mathbf{h}}(\mathbf{y})$	Normalised pole figure
K_A	Sample reference frame
($\mathbf{x_A}$, $\mathbf{y_A}$, $\mathbf{z_A}$)	Unit-vectors of the sample reference frame
$\mathbf{X_A}$, $\mathbf{Y_A}$, $\mathbf{Z_A}$	Sample axes aligned with $\mathbf{x_A}$, $\mathbf{y_A}$, $\mathbf{z_A}$ respectively
[XYZ]	Vector of the sample reference frame
K_B	Crystal reference frame
($\mathbf{x_B}$, $\mathbf{y_B}$, $\mathbf{z_B}$)	Unit-vectors of the crystal reference frame
$\mathbf{X_B}$, $\mathbf{Y_B}$, $\mathbf{Z_B}$	Crystal axes aligned with $\mathbf{x_B}$, $\mathbf{y_B}$, $\mathbf{z_B}$ respectively
$\mathcal{H}$	Orientation space
g	Set of three Euler angles defining one orientation
$\tilde{g}$	Orientation distance
dg	Orientation element in the $\mathcal{H}$-space
α, β, γ	Euler angles in the $\mathcal{H}$-space in the Roe-Matthies convention
φ_1,Φ,φ_2	Euler angles in the $\mathcal{H}$-space in the Bunge convention

$f(g)$	Orientation Distribution of crystallites
$f_f(g)$	OD of for ferroelectric domains
$d_{hk\ell}$,	Inter-reticular distance between (hkℓ) planes
ω	Angle between the incident beam and the sample surface: incidence angle
θ	Angle between the incident beam and the scattering planes {hkℓ}: Bragg angle
δ	Angle running along the Debye ring on a 2D detector
V	Irradiated volume of the sample
$dV(\mathbf{y})$	Volume of crystallites having **h** between **y** and **y** + **dy**
$dV(g)$	Volume of crystallites which orientation is between g and g + dg
Jc	Superconducting transport critical current density
Fd	Damaged (amorphous) fraction of an irradiated sample
Fc	Crystalline fraction of a sample
$\overline{\mathcal{T}}$	Microscopic tensor for a property
$\overline{\mathcal{T}}^M$	Macroscopic tensor
$<\overline{\mathcal{T}}>$	Arithmetic average of the tensor $\overline{\mathcal{T}}$
$\mathbf{p_h}$	electric polarisation vector of a ferroelectric domain
ε_{ij}	strain tensor
ε_{ij}^M	macroscopic strain tensor
σ_{ij}	stress tensor
σ_{ij}^M	macroscopic stress tensor
$S_{ijk\ell}$	elastic compliance tensor
$S_{ijk\ell}^M$	macroscopic elastic compliance tensor
$S_{ijk\ell}^{V,R,H}$	macroscopic elastic compliance tensor calculated using the Voigt, Reuss, Hill models
$C_{ijk\ell}$	elastic stiffness tensor
$C_{ijk\ell}^M$	macroscopic elastic stiffness tensor
$C_{ijk\ell}^{V,R,H}$	macroscopic elastic stiffness tensor calculated using the Voigt, Reuss, Hill models
ξ	mixing parameter of the Hill model
$\chi_{m,ij}$	Magnetic susceptibility tensor
$\mu_{r,ij}$	Magnetic relative permeability tensor
$\varepsilon_{r,ij}$	Dielectric relative permittivity tensor
ε_0	Dielectric permittivity of vacuum
L	Atomic orbital angular momentum
S	Atomic spin angular momentum
J	Atomic total magnetic momentum
g_{ij}	Anisotropic Landé factor
μ_B	Bohr magneton
k_B	Boltzman constant
c	light speed
$\mathbb{N}$	Avogadro number

Abbreviations

14:24	$(Sr,Ca)_{14}Cu_{24}O_{41}$
BAW	Bulk Acoustic Waves
Bi2223	$(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10+x}$
Bi2212	$(Bi,Pb)_2Sr_2Ca_1Cu_2O_{8+x}$
CAPS	Curved-Area Position Sensitive detector
CCL	Comarginal Crossed Lamellar layer
CPS	Curved Position Sensitive detector
CSL	Coincidence Site Lattices
EBSD	Electron Back-Scattering Diffraction
EDX	Energy Dispersive X-ray
ESR	Electron Spin Resonance
FAp	$Ca_{10}(PO_4)_6F_2$
FWHM	Full Width at Half Maximum
HAp	$Ca_{10}(PO_4)_6(OH)_2$
HRTEM	High-Resolution TEM
HWHM	Half Width at Half Maximum
HWHD	Half Width at Half maximum of the distribution Density
ILL	*Institut Laue-Langevin*
LN	$LiNbO_3$
MPB	Morphotropic Phase Boundary
MQTA	Magnetic Quantitative Texture Analysis
m.r.d.	multiple of a random distribution
MTG	Melt Texture Growth
NMR	Nuclear Magnetic Resonance
ODF	Orientation Distribution Function
PSD	Position Sensitive Detector
PZT	$Pb(Zr,Ti)O_3$
PL	PhotoLuminescence
PLE	PhotoLuminescence Excitation
QMA	Quantitative Microstructure Analysis
QPA	Quantitative Phase Analysis
QTA	Quantitative Texture Analysis
RCL	Radial Crossed Lamellar layer
RSA	Residual Strain-stress Analysis
RTGG	Reactive Templated Grain-Growth
SBN	$SrBi_2Nb_2O_9$
SEM	Scanning Electron Microscope
TEM	Transmission Electron Microscope
TGG	Templating Grain Growth
TSMTG	Top-Seeded Melt Texture Growth
XRR	X-Ray specular Reflectivity
Y123	$YBa_2Cu_3O_{7-\delta}$
Y211	Y_2BaCuO_5

Constants

We used in this book the following values of the constants of physics:

Avogadro number	N =	$6.022\ 10^{23}$	
Electron charge	e =	$-1{,}6021892.10^{-19}$	C
Boltzmann constant	k_B =	$1{,}380662.10^{-23}$	$J.K^{-1}$
Planck constant	h =	$6{,}626176.10^{-34}$	J.s
Rydberg constant	R_H =	$1.1\ 10^{7}$	m^{-1}
Electron mass	m_e =	$9{,}109534.10^{-31}$	kg
Free-space permittivity	ε_0 =	$8{,}8541878.10^{-12}$	$A\ s\ A^{-1}\ m^{-1}$
Free-space permeability	μ_0 =	$4\pi\ 10^{-7}$	$V\ s\ A^{-1}\ m^{-1}$
Free-space light speed	c =	299792458	$m.s^{-1}$

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Warnings and comments

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Figures caption

Tables caption