

X-RAY AND NEUTRON  
STRUCTURE ANALYSIS  
IN MATERIALS  
SCIENCE

# X-RAY AND NEUTRON STRUCTURE ANALYSIS IN MATERIALS SCIENCE

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## PREFACE

During the last few decades, crystallography has become a wide and economically important field of science with many interesting applications in materials research, in different branches of physics, chemistry, geology, pharmacology, biochemistry, electronics, in many technological processes, machinery, heavy industry, etc. Twenty Nobel prizes awarded for achievements belonging to this field only underline its distinction. Crystallography has become a commonly used term, but - like a whale - it is much easier to recognize than to describe because of an extreme diversity of subjects involved which range from highly sophisticated theories to the development of routine technological processes or testing of materials in production. It is apparent that only some aspects of selected topics could be included on a single occasion.

The conference "ADVANCED METHODS IN X-RAY AND NEUTRON STRUCTURE ANALYSIS OF MATERIALS" held in Karlovy Vary (Czechoslovakia) on October 5-9, 1987, was intended to cover the most important crystallographic aspects of materials science. The conference was attended by 250 people from 16 countries (Belgium, Bulgaria, China, Czechoslovakia, Finland, France, FRG, GDR, Hungary, Italy, The Netherlands, Poland, Sweden, USA, USSR and Yugoslavia). To advance the cooperation of people from different fields of structure analysis, no strict requirements were laid on the topics presented in poster communications, however the choice of invited lectures and oral contributions was aimed to promote the following topics:

- phase analysis, identification and structure analysis of powder materials
- structure analysis of amorphous materials and polymers
- real structure of crystalline materials
- thin layers and surface coatings (single and polycrystalline)
- difficult problems in crystal structure determination (modulated and polytype structures)
- data acquisition and processing.

This volume consists of 53 papers selected from 160 active contributions presented at the conference.

The principal subject of the first section is the identification of materials and quantitative phase analysis of powder materials. Four papers (by J. Fiala, B. L. Davis, A. Griger, S. Popovič and B. Gržeta) provide a sufficient theoretical background and a comprehensive review of the newest development in the methodology of quantitative analysis of a composition of mixtures. The following nine contributions add a number of significant applications to metals, their corrosion products, alloys, garnets, industrially important minerals and minerals deposited in the human body. The section is closed by papers dealing with the optical simulation of diffraction **patterns**.

The second section is devoted to the structure determination of amorphous materials and polymers. Highly interesting are the structure models of amorphous alloys given by L. Rang-Su. The neutron structure analysis of

amorphous  $\text{Fe}_{0.75}\text{B}_{0.25}$  and tellurite glasses are undoubtedly of great interest and practical impact. X-ray analyses of styrene-methacrylate copolymers and polypropylene fibres close this section.

An introduction to the investigation of the real structure of powder materials in the third section is given by P. Klimanek. Six papers in this section offer examples of studies of different structure defects, texture and interdiffusion in solids using a wide spectrum of systems (rolled FeSi, materials for microelectronics and minerals).

The next section gives a comprehensive view of polycrystalline thin films and surface coatings. The first paper by R. I. Barabash and M. A. Krivoglaz summarizes the theoretical aspects of scattering by thin surface layers influenced inevitably by different types of defects and stresses. An extensive methodological treatment of a number of specific systems is given in papers by H. Oettel, V. Valvoda, E. L. Haase, R. Delhez, J. Piela-szek and I. Tomov. From the material point of view, one can find interesting structure analyses of laser modified surface layers of alloys, analyses of ceramic coatings, nitrided steels, TiC,  $\text{SnO}_x$ , and others, with applications in microelectronics as resistors or potentiometers in hybrid circuits, or as diffusion barriers in integrated circuits. They can be used further as selectively transmitting coatings on architectural glass, in jewelry production, as thermal resistant and wear resistant coatings on cutting tools and as anticorrosive and antiabrasive coatings with interesting applications.

The fifth section dealing with single crystal thin layers is introduced by a comprehensive treatment of garnet films by S. Lagomarsino with applications in lasers, microwave devices, new types of magnetic memories and optoelectronics. A group of crystallographers from the Institute of Crystallography in Moscow describe new methods for the study of structure defects in epitaxial layers based on a standing wave technique (silicone films doped by B and Ge).

The sixth chapter is devoted to special problems in single crystal structure determination. The first paper formulates the conditions for optimal solution of the phase problem and reviews new methods of the crystal structure determination based on the principle of distribution fitting of seminvariants. These methods are suitable for well-ordered single crystals.

The paper written by P. M. de Wolff which offers the reader a new view of the use of superspace groups for incommensurate crystal structures is followed by an informative survey of modulated crystal structures determined at the State University at Buffalo (F. Coppens, V. Petříček), explaining a pressure dependent low-temperature organic superconductivity, transition to the ferroelectric phase and metal-insulator transitions of the organic materials under study.

The paper by Yu. M. Tairov and V. F. Tsvetkov introduces the part devoted to polytype structures. The growing interest in polytypism during the last two decades is mainly due to the fact that electrophysical properties of polytypic substances (e.g., ZnS, SiC, ferrites, chalcogenites) which depend on the actual stacking of layers can be "tuned" simply by their controlled growth. An introduction to the OD symmetry of these structures is given by K. Fichtner. A proper understanding of their symmetry plays a significant role in explaining the physico-chemical properties of polytypes and helps in interpreting the diffraction pattern. Methods of the structure solution of polytypes are discussed by B. B. Zvyagin and B. I. Nikolin. The section is closed by an analysis of statistical parameters describing the polytype stacking.

The last section is devoted to data acquisition and to special instrumental problems - an evaluation of different approximations of diffraction peak profiles, a geometry for multiple scattering, area detectors for powder diffractometers, and X-ray diffractometer for the lattice mismatch measurement in heterostructures.

It must be emphasized that the volume is not meant to provide a full coverage of all latest developments. However, it gives an informative survey of the contemporary state of the art and of new trends in research at universities, research and industrial laboratories. The final manuscripts were subjected to only a brief scrutiny for scientific and language correctness. Therefore, the views expressed in this volume do not necessarily correspond to those of the editors. The full responsibility for exactness, originality and language rests with the individual authors.

Finally, we express our gratitude to all contributors who willingly cooperated in the preparation of this volume, and to all who contributed significantly to the success of the conference in Karlovy Vary, namely, to all members of the organizing committee (J. Fiala, Z. Weiss, I. Vostřáková, A. Stopková, Z. Čížek, V. Petříček, V. Šubrtová, K. Malý and J. Pilná) for their efforts ensuring the smooth course of the conference. The organizers are also indebted to J. Tříška, Director of the Physical Institute of the Czechoslovak Academy of Sciences, Vl. Kubánek, Director of the Institute of Macromolecular Chemistry of the Czechoslovak Academy of Sciences, Z. Kletečka, Director of the Central Research Institute of ŠKODA works, and J. Garaj, Chairman of the Czechoslovak National Committee of IUCr for their great interest in and support of this conference. Lastly a special note of appreciation is due to Mrs E. Biskupová for the typing and artwork involved in producing this book.

Jindřich Hašek  
Jaroslav Fiala  
Václav Valvoda  
Václav Petříček  
Slavomil Ďurovič

## CONTENTS

### POWDER DIFFRACTION ANALYSIS

Identification of X-ray Diffraction Patterns of Multicomponent Mixtures . . . . .	3
J. Fiala	
Quantitative Reference Intensity Analysis: Methodology and Means for Verification of Results . . . . .	15
B. L. Davis	
Correction of Compositional Variability in the X-ray Diffraction Phase Analysis . . . . .	23
A. Griger	
Dilution and Addition Methods in Quantitative X-ray Diffraction Phase Analysis . . . . .	29
S. Popovič and B. Gržeta	
Shape Memory Effect Analysis by X-ray Diffraction . . . . .	43
Z. Bojarski and H. Morawiec	
Formation of a Satellite Phase in the Neutron Irradiated Reactor Steel . . . . .	49
F. Haramia and F. Hanic	
X-ray Phase Analysis of Cu and Pb Anodic Oxidation Products . . . . .	59
E. Źagiewka and A. Budniok	
Phase Analysis of Rusts by X-ray Diffraction, IR-Spectroscopy and Thermal Analysis . . . . .	67
J. Had, J. Balcar, and H. Prachařová	
Dynamical X-ray Diffraction Study on the Phase Transformation in Rapidly Quenched Au <sub>71</sub> Sn <sub>29</sub> Alloy . . . . .	73
N. Mattern	
Process Kinetics Studied by X-ray Diffraction . . . . .	77
T. Havlík and M. Škrobán	
X-ray Diffraction Analysis of 12 CaO · 7 Al <sub>2</sub> O <sub>3</sub> Polymorphs . . . . .	81
A. Derdacka-Grzymek, Z. Konik, J. Iwančiw, M. Pyzalski, and A. Stok	
X-ray Analysis of Mineral Substances of Blood Vessels . . . . .	85
A. Szytuła and E. Rokita	

Optical Diffraction of Computer Simulated Patterns of Transitions in Two Dimensional Regular Lattice . . .	89
J. Sołtys and W. Kołomyjaniak	

DIFFRACTION ANALYSIS OF AMORPHOUS MATERIALS, GLASSES AND POLYMERS

Investigation of the Microscopic Mechanism of the High Strength of Amorphous Alloys . . . . .	95
L. Rang-Su	
Partial Structure Factors for Amorphous $Fe_{75}B_{25}$ Determined by Diffraction of Polarized Neutrons . . . . .	99
W. Matz, J. Kulda, and P. Mikula	
Neutron Diffraction Investigation of the Short-Range Atomic Order in Tellurite Glasses . . . . .	105
S. Neov, I. Gerasimova, and P. Mikula	
Supermolecular Structure of the "Ladder-Type" Styrene-Multimethacrylate Copolymers . . . . .	109
S. Rabiej and A. Włochowicz	
The Structure Evaluation of Polypropylene Fibres by Computational Resolution of WAXS . . . . .	117
V. Marcian and J. Karniš	

REAL STRUCTURE OF CRYSTALLINE MATERIALS

Problems in Diffraction Analysis of Real Polycrystals . . . . .	125
P. Klimanek	
Texture Investigation of Natural Rock-Salt by Neutron Diffraction . . . . .	139
A. Ertel, M. Betzl, and H. Kaempf	
Texture and Structure of Anisotropic FeSi Materials after Hot Rolling . . . . .	143
J. Švantner, V. Frič, D. Matisová, and A. Sólyom	
Real Structure of $Gd_3Co$ Single Crystals A. Winiarski, E. Talik, A. Winiarska, J. Heimann, and J. Szade . . . . .	147
High Temperature Elastic Diffuse Neutron Scattering Study of the Defect Structure in $TiN_{0.82}$ . . . . .	149
T. Priem, B. Beuneu, and Ch. de Novion	
X-ray Diffraction Studies of Interdiffusion in Solid Solutions of Semiconductors $A^{III}B^V$ . . . . .	153
U. Volland, P. Deus, R. Černý, and V. Valvoda	

POLYCRYSTAL THIN LAYERS

Kinematic Theory of X-ray and Neutron Scattering by Defects in Thin Films and Surface Layers . . . . .	161
R. I. Barabash and M. A. Krivoglaz	



Methodical Aspects of X-ray Diffraction Analysis of Surface Treated Materials . . . . .	171
H. Oettel	
Structure Investigation of Hard Coatings by Total Pattern Analysis . . . . .	181
V. Valvoda	
The Determination of Lattice Parameters and Strains in Stressed Thin Films Using X-ray Diffraction: Extensions . . . . .	191
E. L. Haase	
Use and Perspective of X-ray Diffraction in Science and Technology of Ceramic Coatings . . . . .	199
R. Delhez, Th. H. de Keijser, and E. J. Mittemeijer	
Application of X-ray Diffraction Techniques to Study Highly Dispersed Supported Metals . . . . .	209
J. Pielaszek	
Effective Depth of X-ray Penetration and its Orientation Dependence . . . . .	215
I. Tomov	
Structure of Laser Modified Surface Layers of AlZn Alloys . . . . .	223
V. Syneček, J. Lašek, P. Baturška, and M. Simerská	
Structure Characterization of Laser Treated WC-Co by Means of Position Sensitive Detectors . . . . .	237
M. Ermrich	
Diffraction Studies of SnO <sub>x</sub> Thin Films Prepared by Vacuum Methods . . . . .	241
G. Beensh-Marchwicka and L. Król-Stepniewska	
X-ray Diffractational Investigation of the Copper-Aluminium Interface Reaction . . . . .	249
E. Zschech and A. Kaiser	
X-ray Examination of (PbTe) <sub>1-x</sub> (GeTe) <sub>x</sub> Crystals . . . . .	253
M. Leszczynski	

#### SINGLE CRYSTAL THIN LAYERS

Structural Studies of Garnet Films . . . . .	261
S. Lagomarsino	
Location of Impurity Atoms in the Volume and Surface Layers of Silicon Crystals by X-ray Standing Wave in the Laue Geometry . . . . .	273
A. Yu. Kazimirov, M. V. Kovalchuk, and I. V. Kurchatov	
X-ray Standing Waves in the Study of Crystals and Surface Layers . . . . .	277
M. V. Kovalchuk and V. G. Kohn	
Development of Theoretical and Experimental Investigations of Thin Surface Structures by X-ray Methods . . . . .	283
I. A. Vartanyantz, A. Yu. Kazimirov, M. V. Kovalchuk, and V. G. Kohn	

CRYSTAL STRUCTURE DETERMINATION

New Trends in Determination of Crystal Structure . . . . .	293
J. Hašek	
Easy and Uneasy Superspace Groups for Incommensurate Crystals . . . . .	307
P. M. de Wolff	
Structure Analysis of Modulated Molecular Crystals IV: Survey of our Recent Studies . . . . .	317
F. Coppens and V. Petříček	
Controlled Growth of Polytypes and their Importance for Science and Technology . . . . .	331
Yu. M. Tairov and V. F. Tsvetkov	
Symmetry and Diffraction Patterns of Polytypes . . . . .	341
K. Fichtner	
Structural Characteristics of Polytypes Delivered by Texture Diffraction Patterns . . . . .	353
B. B. Zvyagin	
The X-ray Method for Determining Crystalline Multilayer Polytype Structures in Metal Alloys . . . . .	361
B. I. Nikolin	
The Analysis of Disorder Polytype Structures by Statistical Parameters . . . . .	363
M. J. Kozielski and A. Tomaszewicz	

DATA ACQUISITION

Bragg Reflection Analysis for Powdered Samples . . . . .	371
G. Stanisz, J. Holender, and J. Sołtys	
Rietveld Refinement of $Y_2O_3$ - Comparison of Two Profile Functions . . . . .	375
L. Smrček and P. Duriš	
Three-Dimensional Multiple X-ray Diffraction . . . . .	379
D. Korytár	
Reduction of the Electronics of a ID Position Sensitive Detector . . . . .	383
J. H. Duijn, C. W. E. van Eijk, R. W. Hollander, and G. W. Sloof	
A High Resolution Monochromator Module with X-ray Optics for Multipurpose Measurement of $A^3B^5$ Heterostructures . . . . .	389
P. Kacerovský	
LIST OF AUTHORS . . . . .	393
INDEX . . . . .	395
ABBREVIATIONS . . . . .	405