Electron Crystallography

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Electron Crystallography

Novel Approaches for Structure Determination of Nanosized Materials

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Table of Contents

List of Contributors	xi
Preface	xvii
Lectures	
A. Introduction	
A.1 X.D. Zou – What is electron crystallography	3
A.2 C. Kisielowski – Exploiting sub-Ångstrom abilities <i>What advantages do different techniques offer?</i>	17
A.3 J.C.H. Spence – The way forward for electron crystallography	31
A.4 M. Hargittai, I. Hargittai – Symmetry and structure	43
B. Experimental techniques	
B.1 J.P. Morniroli – Introduction to electron diffraction	61
B.2 J.P. Morniroli – Symmetry determinations by electron diffraction	73
B.3 V. Klechkovskaya – Electron diffraction structure analysis <i>Specimens and their electron diffraction patterns</i>	85
B.4 A.S. Avilov – Quantitative electron diffraction structure analysis <i>Theory and practise of determining the electrostatic potential and chemical bonding in crystals</i>	97

B.5	P. Oleynikov, S. Hovmöller, X. D. Zou – Quantification of texture patterns	121
B.6	J.M. Zuo – Quantitative Convergent Beam electron diffraction <i>Measurements of lattice parameters and crystal charge density</i>	143
B.7	S. Nicolopoulos, A. Kuligin, K. Kuligin, B. Khalid, G. Lepeshov, J. L. DelPlancke, A. Avilov, M. Nickolsky, A. Ponce – New instrumentation for TEM electron diffraction structure analysis: Electron diffractometry combined with beam precession	169
B.8	J.L. Lábár – Role of electron powder diffraction in solving structures	185
B.9	I. Hargittai – Gas-phase electron diffraction for molecular structure determination	197
B.10	J.L. Lábár – Phase identification by combining local composition from EDX with information from diffraction database	207
B.11	J. Mayer – Analysis of local structure, chemistry and bonding by electron energy loss spectroscopy	219
C. Crystal structure determination from electron microscopy data		
C.1	T.E. Weirich – From Fourier series towards crystal structures <i>A survey of conventional methods for solving the phase problem</i>	235
C.2	H. Jiang, F.H. Li, E.I. Kauppinen – Crystal structure determination by image deconvolution and resolution enhancement	259

C.3	X.D. Zou, S. Hovmöller – Structure determination from HREM by crystallographic image processing	275
C.4	X.D. Zou, S. Hovmöller – 3D reconstruction of inorganic crystals <i>Theory and application</i>	301
C.5	C.J. Gilmore, A. Stewart – Solving and refining crystal structures from electron diffraction data	321
C.6	C.J. Gilmore – The maximum entropy method of solving crystal structures from electron diffraction data	337
C.7	J. Jansen – Structure refinement by taking dynamical diffraction into account <i>Multi-slice least-squares refinement</i>	355
C.8	C. Kübel, A. Thust – TrueImage: <i>A software package for focal-series reconstruction in HRTEM</i>	373

D. Applications

D.1	G. Cox, T. Breiner, H. Hibst, U. Müller – New insights into the nanoworld of functional materials	395
D.2	U. Kolb, T. Gorelik – Electron crystallography on polymorphs	409
D.3	D. Nihtianova, J. Li, U. Kolb – Electron crystallography in mineralogy and materials science	421
D.4	O. Terasaki – Structures of zeolites and mesoporous crystals determined by electron diffraction and high-resolution electron microscopy	435
D.5	A.M. Tonejc – HRTEM investigation of nanocrystalline materials	443

D.6	M. Tsuji – Electron crystallography on beam sensitive materials <i>Electron microscopy and electron diffraction of polymers</i>	455
D.7	D. Wang – Characterisation of catalysts by transmission electron microscopy	473
E. Extended abstracts		
E.1	L. Demchenko, V. Nadutov, D. Beke, L. Daroczi – Structural investigations of cold worked iron based alloys after nitriding	491
E.2	I. Djerdj, A.M. Tonejc, A. Tonejc – Structural refinement of nanocrystalline TiO ₂ samples	497
E.3	O.V. Hryhoryeva – Relation between magnetic properties and the structure of iron-based amorphous alloys determined by electron diffraction	503
E.4	D. M. Kepaptsoglou, M. Deanko, D. Janičkovič, E. Hristoforou, P. Švec – Selected issues in quantitative structure analysis of nanocrystalline alloys	507
E.5	E. Montanari, S. Fabbrici, G. Calestani, A. Migliori – Charge ordering and tilt modulation in multiferroic fluorides with TTB structure by electron diffraction and single crystal XRD	513
E.6	M.B. Shevchenko, O.V. Pobydaylo – Electron diffraction by distorted nanocrystals <i>Application of the eikonal representation</i>	517
E.7	T.G. Shumilova, J. Akai, Ye.A. Golubev – Electron microscopy investigations of glassy-like carbon	523

E.8	C.Y. Tang, F.H. Li – Image deconvolution on crystals with interfaces	527
E.9	Y.M. Wang, H.B. Wang, F.H. Li – Crystal structure determination by maximum entropy image deconvolution in combination with image simulation	533

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Preface

The discovery of electron diffraction in the laboratories of C.J. Davisson and G.P. Thomson in 1927 certainly ranks as one of the monumental discoveries in the history of science. Soon after this invention, it was realised that electrons - like X-rays - are diffracted by the atoms in matter and thus can be used for structure determination. Because the attainable scattered intensities for electrons are about 10^6 times larger than for X-rays, electron diffraction allows us to study the interior structure of matter down to a few cubic-nanometers in size. This is far beyond the capabilities of X-ray crystallography or even a modern synchrotron. However, with increasing understanding of the electron scattering process, in particular by development of *n*-beam scattering theory during the 1950s of the past century, it became general knowledge that it could normally *not* be assumed that the kinematical or single-scattering approximation holds even for very thin crystals. For this reason nearly all activities in this direction declined within a few years from that time, even though the groups of Z.G. Pinsker, B.K. Vainshtein and B.B. Zvyagin in Russia had proved the feasibility of this approach since about 1940. While X-ray diffraction turned over the following years into a powerful quasi-automatic method for structure determination, structure analysis by electron diffraction was not seriously developed further for a period of more than twenty years. It took until 1976 to perform the first structure determination by direct phasing methods from electron diffraction data of an organic compound by D.L. Dorset & H. Hauptman, and another eight years for the first *ab-initio* structure determination of a heavy-metal oxide from high-resolution electron microscopy images by S. Hovmöller and co-workers. Despite these achievements, the earlier skepticism against structural results from electrons continued, and remained a common mindset until a few years ago.

Nevertheless, during the last decade we have witnessed several exciting achievements in this field. This includes structural and charge density studies on organic molecules and protein structures, complicated inorganic and metallic materials in the amorphous, nano-, meso- and quasi-crystalline state and also development of new software, tailor-made for the special needs of electron crystallography. Moreover, these developments have been accompanied by a now available new generation of computer controlled electron microscopes equipped with high-coherent field-emission sources, cryo-specimen holders, ultra-fast CCD cameras, imaging plates, energy filters and even correctors for electron optical distortions. Thus, a fast and semi-automatic data acquisition from small sample areas, similar to what we

today know from imaging plates diffraction systems in X-ray crystallography, can be envisioned for the very near future. This progress clearly shows that the contribution of electron crystallography is quite unique, as it enables us to reveal the intimate structure of samples with high accuracy, but on much smaller samples than have ever been investigated by X-ray diffraction. As a tribute to these tremendous recent achievements, this NATO Advanced Study Institute was devoted to the novel approaches of electron crystallography for structure determination of nano-sized materials. The course took place 9 – 20 June 2004 at the renowned Ettore Majorana Centre for Scientific Culture in Erice, Sicily and brought together 87 participants from 32 different countries.

The present volume contains the lectures given during this ASI and covers almost all theoretical and practical aspects of advanced transmission electron microscopy techniques and crystallographic methods that are relevant for determining structures of organic and inorganic materials. Moreover a number of extended abstracts on the presented posters during this ASI have been added to this volume.

Supplementary material (videos, handouts) of the course is available at <http://erice2004.docking.org/>

We are grateful for a number of sponsors of this ASI. First and foremost is the support from NATO (ASI 979903) acknowledged. The International Association for the Promotion of Co-operation with Scientists from the New Independent States (NIS) of the Former Soviet Union (INTAS) gave generous financial support as did the International Union of Crystallography, FEI Company (Kassel, Germany), Carl Zeiss NTS (Oberkochen, Germany), Ditabis (Pforzheim, Germany) and Gatan (Munich, Germany). We are also extremely grateful to Prof. Paola Spadon for her constant aid toward making this ASI possible. Special thanks are also given to Prof. Lodovico Riva di Sanseverino for his organisational skills, constant attention to all details, his valuable advice based on more than 30 years experience with these courses, and for competent managing of the budget of this ASI. Last but not least, we thank John Irwin for his rapid troubleshooting of all IT problems, and for setting up the web site for the supplementary material.

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