Progress in

# Transmission Electron Microscopy

II. Applications in Materials Science

Editors: Xiao-Feng Zhang Ze Zhang







# Progress in Transmission Electron Microscopy

II . Applications in MaterialsScience

透射电子显微学新进展

Ⅱ卷. 在材料科学中的应用

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# 内容简介

本书是海内外电子显微学工作者国际合作的成果。全书分为1. II两卷。I卷阐述电子显微学理论及方法,这里涉及到的都是近年来的最新进展,同时介绍了这些理论和方法在材料科学中的重要作用。II卷主要介绍电子显微学在材料科学等方面的应用。涉及到三类材料。一类是具有巨大工业应用前景的材料,如半导体,超导体;第二类包括用其他手段难以进行研究的严重畸变材料,低维纳米材料;第三类是具有非常复杂结构的高分子材料。

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# Frontiers of Science and Technology for the 21st Century

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# 《21世纪科技前沿》 丛 书 序 言

由清华大学出版社出版的这套丛书是基础科学和应用科学领域内的专门著作。除了可作为研究生教材外,也可作为科研和工程技术人员的参考书。在丛书的题材选择中,着重考虑目前比较活跃而且具有发展前景的新兴学科。因此,这套丛书大都涉及交叉和新兴学科的内容。编写的方式大多由主编策划并组织本学科有影响的专家共同执笔完成,从而使每一本书的系统性和各章节内容的连贯性得到了充分的兼顾。丛书涵盖学科的最新学术进展,兼顾到基本理论和新技术、新方法的介绍,并引入必要的导论和充分的参考文献以适应具有不同学术背景的读者。编撰一套容纳多学科的科技丛书是一项浩繁的工作,我们希望通过主编和作者的集体努力和精诚协作,使整套丛书的学术水准能够保持在较高的水平上。

编辑《21 世纪科技前沿》丛书是由"旅美中国科学家工程师协会"发起的一项国际科技界的合作。传递信息,加强交流,促进新世纪的科技繁荣是编著者们参与此项工作的共同信念。此外,这套丛书还具有特别的纪念意义。20 年前,历史的进程使成千上万的中国学生、学者有机会走出国门,到世界各地学习和从事科学研究。今天,活跃在世界科技前沿领域的中华学子们没有忘记振兴祖国科技教育事业的责任和推动国际学术交流与合作的义务。正是基于这一共同的心愿,大家积极参与这套系列丛书的撰写、组稿和编辑工作。为此,我们愿以这套丛书来纪念中国改革开放 20 周年。

编委会 1999.6

# (Frontiers of Science and Technology for the 21st Century) FOREWORD

Over the next several years, Tsinghua University Press will publish a series of books addressing progress in basic sciences and innovations in technology. We have made no attempt to pursue a comprehensive coverage of all disciplines of science and technology. Rather, topics for this series were selected with an emphasis on the currently active forefront of science and technology that will be contemporary in the next century. Most books in this series will deal with subjects of cross disciplines and newly emerging fields. Each book will be completed by individual authors or in a collaborative effort managed by an editor(s), and will be self-consistent, with contents systematically focused on review of the most recent advances and description of current progresses in the field. Sufficient introduction and references will be provided for readers with varying backgrounds. We have realize clearly the challenge of encompassing the diverse subjects of science and technology in one series. However, we hope that, through intensive collaboration between the authors and editors, high standards in editorial quality and scientific merit will be maintained for the entire series.

The international collaboration on this series has been coordinated by the Association of Chinese Scientists and Engineers-USA(ACSE). In the science community, authors voluntarily publish their results and discoveries in the full conviction that science should serve human society. The editors and authors of this series share this academic tradition, and many of them are fulfilling a spiritual commitment as well. For our editors and authors who were graduated

from universities in China and further educated abroad in science and engineering, this is an opportunity to dedicate their work to the international education community and to commemorate the historical open-door movement that began in China two decades ago. When the human society enters the information age, there is no geographic boundary for science. The Editorial committee hopes that this series will promote further international collaboration in scientific research and education at the dawn of the new century.

The Editorial Committee 1999.6

# (Progress in Transmission Electron Microscopy) FOREWORD

In the field of electron microscopy, China is a late comer owing to historical reasons. With the development of sophisticated electron microscopes equipped with double condenser and intermediate lenses and the advent of theory of electron diffraction and imaging in the fifties, the application of transmission electron microscopy (TEM) in materials science experienced a rapid and fruitful expansion in the sixties and seventies. During this period, however, China was trapped in political chaos. When the nightmare was over, the scientists in China were confronted with the high-resolution electron microscopy (HREM) at the atomic level and the analytical electron microscopy (AEM) at the nanometer scale. Amazed at these great achievements, electron microscopists in China took the challenge heroically and worked feverishly to bridge the gap between China and the well developed countries in TEM and its application in materials science.

To promote TEM development in China, TEM laboratories equipped with up-to-date electron microscopes were established in the eighties and a large number of graduates, mostly young physicists, were attracted to learn the fundamentals of TEM in these laboratories. Talent young scientists also had got opportunities to work at the frontier of TEM and its application in materials science in internationally well known TEM laboratories, such as those located in Oxford, Cambridge, Tempe, Antwerp, Osaka, Sydney etc. After ten years of hard working, the young generation of electron microscopists from China started to receive recognition in the international community. One example is that five young

Chinese scientists were elected the IFSEM (International Federation of Societies for Electron Microscopy) Presidential Scholars, and seven the EMSA (Electron Microscopy Society of America, later MSA) Presidential Scholars at the 12th International Congress for Electron Microscopy in 1990. Among the chapter authors of this book, Dr. Zhonglin Wang won recently the 1999 Burton Award of MSA granted to a selected scientist who has made important contributions to electron microscopy in the preceding five years, and Dr. Zhenghong Zhou was named a 1999 Pew Scholar for his outstanding promise in Biomedical Sciences.

This book reflects, to a lesser extent, the manifold results of the above-mentioned international cooperation. In addition to contributions from eminent senior scientists in the world, many chapter authors are young Chinese electron microscopists. I happen to know most of them personally, including the editors. With such a multitude of talented, enthusiastic, and devoted electron microscopists, I am highly confident of more extensive and significant contributions that China will make to TEM and its application in materials science in the coming years.

Finally, I would like to congratulate the editors for a fine work well done.

K. H. Kuo
Past President, Chinese Electron Microscopy So

Past President, Chinese Electron Microscopy Society (1982 – 1996) Beijing, China

Past President Committee of Asia-Pacific Societies of Electron Microscopy (1992-1996)

# 《透射电子显微学的新进展》

本书是"旅美中国科学家工程师协会"编著的《21 世纪科技前沿》丛书的一部分。鉴于电子显微学在凝聚态物理、材料科学和生命科学应用中的重要作用,丛书编委会认为,编著此类专业书籍将对促进材料科学等学科的发展具有积极意义。本书分为 I、II 两卷,I 卷包括电子显微学理论及方法,II 卷主要介绍电子显微学在材料科学等方面的应用。所涉及到的理论和方法大都是近年来的最新进展。

自 1931 年以来,电子显微学一直在材料科学发展中起着重要的推动作用,目前人们对电子显微学的需求已发展到高分辨(0.1 纳米)、多功能和信息数字化阶段。为满足这些要求,不仅需要最先进的电子显微镜,最佳样品制备方法等基本要素,也需要采用相应高水平的实验技术以获取实验数据,而且更需要完善的电子显微学理论用以解释这些结构信息。

# I 卷各章内容的题目为:

- 1. 现代电子显微镜
- 2. 超高分辨的追求
- 3. 扫描透射电子显微学的原子序数衬度成像
- 4. 电子非弹性散射效应,损失谱和成像
- 5. 定量高分辨电子显微学
- 6. 电子晶体学:用高分辨、电子衍射和图像处理确定晶体结构
- 7. 非晶态电子显微研究
- 8. 弱束电子显微学
- 9. 用会聚束电子衍射方法确定晶体的点群和空间群
- 10. 透射电镜样品制备技术

需要指出,本书的第 1 卷并非一本电子显微学教科书,因此不难发现,

其中缺少许多电子显微学的基本内容。然而,本书涉及到许多电子显微学理论和方法的最新进展,且反映了这些方法在材料科学研究中的重要作用,因而对电子显微学乃至材料科学工作者来说,不失为一本有意义的参考书。

本书的 II 卷介绍了以电子显微学为主要研究手段的应用进展。从材料科学及工程技术的发展来看,半导体、超导体器件的空间尺寸正在趋向10 纳米数量级;纳米材料呈现出令人惊讶的物理和力学性能并显示出广阔的应用前景;对这些新材料新器件的深入研究都将限制在纳米甚至原子层次,电子显微学都因其独特优越性而起着极其重要的作用。II 卷中涉及到三类材料:一类是具有巨大工业应用前景的材料,如半导体,超导体;第二类包括用其它手段难以进行研究的严重畸变结构材料,低维纳米材料;第三类是具有非常复杂结构的高分子材料等。本卷涉及的内容为:

# 纳米和低维结构

- 高分辨电子显微学在发展中孔分子筛中的指导作用
- 电弧放电制备的纳米碳管的高分辨电子显微学研究
- 利用电子衍射确定纳米碳管的螺旋度
- 低维材料及其结构的高分辨电子显微研究

# 界面结构

- 高温超导约瑟夫森结的显微结构
- 缺陷和位错
- YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-8</sub>高温超导快速重离子辐照损伤
- 半导体异质结构中点阵失配位错的透射电子显微学研究
- 位错衬度分析: 位错上的异质成核研究

# 大分子复合物

• 冷冻电子显微学和大分子复合物的三维重构

本书是国际电子显微学工作者合作的成果。它充分反映了中国电子显微学工作者在几乎所有材料科学领域中的积极探索。随着新世纪的到来,国内外电子显微学工作者期待着更多的机会来加强相互之间的合作与交流。编辑这本书的目的之一就是为了促进这样的学术交流。这在本书II卷中得到了充分的反映,因为其中各章的作者都是海内外青年电子显微学工作者。从这种意义上讲,可以认为本书是对中国科学家在国际科技领域作出新的和有价值贡献的一种激励。

美国劳伦斯-伯克利国家实验室材料科学部 章效锋

中国科学院北京电子显微镜实验室 张 泽

# (Progress in Transmission Electron Microscopy) PREFACE

This book is a part of the series 《Frontiers of Science and Technology for the 21st Century》, organized by the Association of Chinese Scientists and Engineers-USA (ACSE). Based on a rapidly increasing recognition of the crucial role the transmission electron microscopy (TEM) is playing in materials science, the ACSE editorial committee strongly believes that TEM warrants a significant topic in the series. This truly admirable vision led to the accomplishment of this book which contains two volumes: Volume I, Concepts and Techniques; and Volume II, Applications in Materials Science. The book covers a great deal of aspects of modern electron microscopy, from architecture of new-concept electron microscopes, advanced theories and techniques in TEM and specimen preparation, to a variety of hands-on examples for TEM applications.

Today, the demands for the TEM lean toward ultra-high resolution (0.1nm), broad operational functions, and quantitative structural and chemical analyses. Such high-level demands require an integration of state-of-the-art electron microscopes, theories, sample preparations, TEM techniques, and data analyses. Volume I actually follows this sequence giving readers a comprehensive introduction for what has been done, what is ongoing, and what can be expected in future. Concepts and techniques of quantification are emphasized. However, volume I is not intended to serve as a TEM text book, therefore readers may find the omission of many traditional TEM lessons. The value of volume I can be best appreciated for its review of the remarkable advances in newly developed concepts and techniques which are making

TEM among the most powerful and indispensable techniques in materials research. The chapters in volume I include:

- The modern microscope today
- The quest for ultra-high resolution
- Z-contrast imaging in the scanning transmission electron microscope
- Inelastic scattering in electron microscopy-Effects, spectrometry and imaging
- · Quantitative analysis of high-resolution atomic images
- Electron crystallography-structure determination by combining HREM, crystallographic image processing and electron diffraction
- · Electron amorphography
- · Weak-beam electron microscopy
- Point group and space group identification by convergent-beam electron diffraction
- Advanced techniques in TEM specimen preparation

While volume I focuses mainly on advances in the modern electron microscopy, the guidance role of electron microscopy in developing and characterizing advanced materials is demonstrated in volume II. The contribution of electron microscopy is prominent and highly appreciable in researches dealing with objects with submicron-size or complicated structures. The objects studied in volume II include:

### Nano-scale and low-dimensional structures:

- The guidance role of HREM in developing mesoporous molecular sieves
- HREM study of carbon nanoclusters grown from carbon arc-discharge
- Determining the helicity of carbon nanotubes by electron diffraction
- Low dimensional materials and their microstructures studied by high-resolution electron microscopy

### Interfacial structures:

Microstructures of high-T<sub>c</sub> superconducting Josephson junctions

### Defects and dislocations:

- Swift heavy ion irradiation damage in  $YBa_2Cu_3O_{7-\delta}$  superconductors
- Transmission electron microscopy investigations of misfit dislocations in latticemismatched semiconductor heterostructures
- Dislocation contrast analysis: a study of  $\,\delta\,'$  helerogeneous nucleation on dislocations Macromolecular Complexes:
- · Transmission electron cryomicroscopy and three-dimensional reconstruction of

### macromolecular complexes

Contributions to this book were from distinguished scholars and engineers in the world. The authorship of this book represents an international collaboration in the field of electron microscopy. In fact, one of our purposes for editing this book is to promote further academic exchange between international and Chinese TEM communities. This intention is especially reflected in volume II, of which most contributions were from young electron microscopists originated from China.

In addition to the new developments, comprehensive and updated introductions for background are also given in chapters. Readers may benefit from the historical and up-to-date information and gain instant knowledge and thorough understanding of the subjects under discussion. We hope that the extensive coverage and outstanding academic level of this book can make it a very valuable resource for graduate students, researchers, engineers, and others who are studying or working in the field of electron microscopy.

Xiao-feng Zhang Materials Sciences Division Lawrence Berkeley National Laboratory

Ze Zhang Beijing Laboratory of Electron Microscopy

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