NEUTRON SCATTERING – APPLICATIONS IN BIOLOGY, CHEMISTRY, AND MATERIALS SCIENCE

Edited by
FELIX FERNANDEZ-ALONSO
DAVID L. PRICE

VOLUME 49
EXPERIMENTAL METHODS IN THE PHYSICAL SCIENCES

ISBN 978-0-12-805324-9

ELSEVIER

ACADEMIC PRESS
An imprint of Elsevier
elsevier.com/books-and-journals

FERNANDEZ-ALONSO
PRICE

NEUTRON SCATTERING – APPLICATIONS IN BIOLOGY, CHEMISTRY, AND MATERIALS SCIENCE

49

EXPERIMENTAL
METHODS IN THE
PHYSICAL SCIENCES

ACADEMIC
PRESS

Experimental Methods in the Physical Sciences
Volume 49

Neutron Scattering - Applications in Biology, Chemistry, and Materials Science

Edited by

Felix Fernandez-Alonso
ISIS Facility
Rutherford Appleton Laboratory
Didcot, United Kingdom

and

Department of Physics & Astronomy
University College London
London, United Kingdom

David L. Price
Synchrotron Radiation and Neutron Research Group, CEMHTI
Centre National de la Recherche Scientifique
Orléans, France

ELSEVIER
ACADEMIC PRESS
An imprint of Elsevier

Academic Press is an imprint of Elsevier
50 Hampshire Street, 5th Floor, Cambridge, MA 02139, United States
525 B Street, Suite 1800, San Diego, CA 92101–4495, United States
The Boulevard, Langford Lane, Kidlington, Oxford OX5 1GB, United Kingdom
125 London Wall, London, EC2Y 5AS, United Kingdom

ISBN: 978-0-12-805324-9
ISSN: 1079-4042

For information on all Academic Press publications
visit our website at https://www.elsevier.com/books-and-journals

Publisher: John Fedor
Acquisition Editor: Anita A. Koch
Editorial Project Manager: Amy Clark
Production Project Manager: Maria Bernard
Cover Designer: Greg Harris

Typeset by SPi Global, India

Experimental Methods in the Physical Sciences
Volume 49

Neutron Scattering - Applications in Biology, Chemistry, and Materials Science

Experimental Methods in the Physical Sciences

Thomas Lucatorto, John T. Yates,
and Kenneth Baldwin
Editors in Chief

List of Contributors

Peter W. Albers, Evonik Technology & Infrastructure GmbH, Hanau (Wolfgang), Germany

Carla Andreani, Università degli Studi di Roma "Tor Vergata"; Museo Storico della Fisica e Centro Studi e Ricerche Enrico Fermi, Rome, Italy

C. Richard A. Catlow, UK Catalysis Hub, Rutherford Appleton Laboratory, Didcot; Cardiff Catalysis Institute, Department of Chemistry, Cardiff University, Cardiff; Department of Chemistry, University College London, London, United Kingdom

Felix Fernandez-Alonso, ISIS Facility, Rutherford Appleton Laboratory, Didcot; Department of Physics & Astronomy, University College London, London, United Kingdom

Zoe Fisher, European Spallation Source; Department of Biology, Lund University, Lund, Sweden

Malcolm Guthrie, European Spallation Source, Lund, Sweden

Louis Hennet, Conditions Extrêmes et Matériaux: Haute Température et Irradiation, Orléans, France

Dirk Holland Moritz, Institut für Materialphysik im Weltraum, Deutsches Zentrum für Luft- und Raumfahrt, Köln, Germany

Andrew Jackson, European Spallation Source; Department of Chemistry, Lund University, Lund, Sweden

Maths Karlsson, Department of Chemistry and Chemical Engineering, Chalmers University of Technology, Göteborg, Sweden

Andrey Kovalevsky, Biology and Soft Matter Division, Oak Ridge National Laboratory, Oak Ridge, TN, United States

Matthew Krzystyniak, ISIS Facility, Rutherford Appleton Laboratory, Didcot; Nottingham Trent University, Nottingham, United Kingdom

David Lennon, School of Chemistry, University of Glasgow, Glasgow, United Kingdom

Andreas Meyer, Institut für Materialphysik im Weltraum, Deutsches Zentrum für Luft- und Raumfahrt, Köln, Germany

Masato Ohnuma, Hokkaido University, Sapporo, Japan

Esko Oksanen, European Spallation Source; Department of Biochemistry and Structural Biology, Lund University, Lund, Sweden

Alexander J. O'Malley, UK Catalysis Hub, Rutherford Appleton Laboratory, Didcot; Cardiff Catalysis Institute, Department of Chemistry, Cardiff University, Cardiff, United Kingdom

Stewart F. Parker, ISIS Facility, Rutherford Appleton Laboratory, Didcot, United Kingdom

Adrien Perrichon, Department of Chemistry and Chemical Engineering, Chalmers University of Technology, Göteborg, Sweden

Giovanni Romanelli, ISIS Facility, Rutherford Appleton Laboratory, Didcot, United Kingdom

Olga Russina, Department of Chemistry, University of Rome "Sapienza", Rome, Italy

Roberto Senesi, Università degli Studi di Roma "Tor Vergata"; Museo Storico della Fisica e Centro Studi e Ricerche Enrico Fermi, Rome; Consiglio Nazionale delle Ricerche, CNR-IPCF, Messina, Italy

Tilo Seydel, Institut Max von Laue – Paul Langevin, Grenoble, France

Mitsuhiro Shibayama, The University of Tokyo, The Institute for Solid State Physics, Kashiwa, Chiba, Japan

Alan K. Soper, Rutherford Appleton Laboratory, Chilton, United Kingdom

Alessandro Triolo, Istituto Struttura della Materia, Consiglio Nazionale delle Ricerche, Rome, Italy

Hanna Wacklin, European Spallation Source; Department of Chemistry, Lund University, Lund, Sweden

Xun-Li Wang, City University of Hong Kong, Kowloon, Hong Kong

Richard Weber, Materials Development, Inc., Arlington Heights, IL, United States

Wanchuck Woo, Korea Atomic Energy Research Institute, Daejeon, South Korea

Volumes in Series

Experimental Methods in the Physical Sciences (Formerly Methods of Experimental Physics)

Volume 1. Classical Methods
Edited by Immanuel Estermann

Volume 2. Electronic Methods, Second Edition (in two parts)
Edited by E. Bleuler and R. O. Haxby

Volume 3. Molecular Physics, Second Edition (in two parts)
Edited by Dudley Williams

Volume 4. Atomic and Electron Physics - Part A: Atomic Sources and Detectors; Part B: Free Atoms
Edited by Vernon W. Hughes and Howard L. Schultz

Volume 5. Nuclear Physics (in two parts)
Edited by Luke C. L. Yuan and Chien-Shiung Wu

Volume 6. Solid State Physics - Part A: Preparation, Structure, Mechanical and Thermal Properties; Part B: Electrical, Magnetic and Optical Properties
Edited by K. Lark-Horovitz and Vivian A. Johnson

Volume 7. Atomic and Electron Physics - Atomic Interactions (in two parts)
Edited by Benjamin Bederson and Wade L. Fite

Volume 8. Problems and Solutions for Students
Edited by L. Marton and W. F. Hornyak

Volume 9. Plasma Physics (in two parts)
Edited by Hans R. Griem and Ralph H. Lovberg

Volume 10. Physical Principles of Far-Infrared Radiation
Edited by L. C. Robinson

Volume 11. Solid State Physics
Edited by R. V. Coleman

Volume 12. Astrophysics - Part A: Optical and Infrared Astronomy
Edited by N. Carleton
Part B: Radio Telescopes; Part C: Radio Observations
Edited by M. L. Meeks

Volume 13. Spectroscopy (in two parts)
Edited by Dudley Williams

Volume 14. Vacuum Physics and Technology
Edited by G. L. Weissler and R. W. Carlson

Volume 15. Quantum Electronics (in two parts)
Edited by C. L. Tang

Volume 16. Polymers - Part A: Molecular Structure and Dynamics; Part B: Crystal Structure and Morphology; Part C: Physical Properties
Edited by R. A. Fava

Volume 17. Accelerators in Atomic Physics
Edited by P. Richard

Volume 18. Fluid Dynamics (in two parts)
Edited by R. J. Emrich

Volume 19. Ultrasonics
Edited by Peter D. Edmonds

Volume 20. Biophysics
Edited by Gerald Ehrenstein and Harold Lecar

Volume 21. Solid State Physics: Nuclear Methods
Edited by J. N. Mundy, S. J. Rothman, M. J. Fluss, and L. C. Smedskjaer

Volume 22. Solid State Physics: Surfaces
Edited by Robert L. Park and Max G. Lagally

Volume 23. Neutron Scattering (in three parts)
Edited by K. Skold and D. L. Price

Volume 24. Geophysics - Part A: Laboratory Measurements; Part B: Field Measurements
Edited by C. G. Sammis and T. L. Henyey

Volume 25. Geometrical and Instrumental Optics
Edited by Daniel Malacara

Volume 26. Physical Optics and Light Measurements
Edited by Daniel Malacara

Volume 27. Scanning Tunneling Microscopy
Edited by Joseph Stroscio and William Kaiser

Volume 28. Statistical Methods for Physical Science
Edited by John L. Stanford and Stephen B. Vardaman

Volume 29. Atomic, Molecular, and Optical Physics - Part A: Charged Particles; Part B: Atoms and Molecules; Part C: Electromagnetic Radiation
Edited by F. B. Dunning and Randall G. Hulet

Volume 30. Laser Ablation and Desorption
Edited by John C. Miller and Richard F. Haglund, Jr.

Volume 31. Vacuum Ultraviolet Spectroscopy I
Edited by J. A. R. Samson and D. L. Ederer

Volume 32. Vacuum Ultraviolet Spectroscopy II
Edited by J. A. R. Samson and D. L. Ederer

Volume 33. Cumulative Author Index and Tables of Contents, Volumes 1-32

Volume 34. Cumulative Subject Index

Volume 35. Methods in the Physics of Porous Media
Edited by Po-zen Wong

Volume 36. Magnetic Imaging and its Applications to Materials
Edited by Marc De Graef and Yimei Zhu

Volume 37. Characterization of Amorphous and Crystalline Rough Surface: Principles and Applications
Edited by Yi Ping Zhao, Gwo-Ching Wang, and Toh-Ming Lu

Volume 38. Advances in Surface Science
Edited by Hari Singh Nalwa

Volume 39. Modern Acoustical Techniques for the Measurement of Mechanical Properties
Edited by Moises Levy, Henry E. Bass, and Richard Stern

Volume 40. Cavity-Enhanced Spectroscopies
Edited by Roger D. van Zee and J. Patrick Looney

Volume 41. Optical Radiometry
Edited by A. C. Parr, R. U. Datla, and J. L. Gardner

Volume 42. Radiometric Temperature Measurements. I. Fundamentals
Edited by Z. M. Zhang, B. K. Tsai, and G. Machin

Volume 43. Radiometric Temperature Measurements. II. Applications
Edited by Z. M. Zhang, B. K. Tsai, and G. Machin

Volume 44. Neutron Scattering – Fundamentals
Edited by Felix Fernandez-Alonso and David L. Price

Volume 45. Single-Photon Generation and Detection
Edited by Alan Migdall, Sergey Polyakov, Jingyun Fan, and Joshua Bienfang

Volume 46. Spectrophotometry: Accurate Measurement of Optical Properties of Materials
Edited by Thomas A. Germer, Joanne C. Zwinkels, and Benjamin K. Tsai

Volume 47. Optical Radiometry for Ocean Climate Measurements
Edited by Giuseppe Zibordi, Craig J. Donlon, and Albert C. Parr

Volume 48. Neutron Scattering - Magnetic and Quantum Phenomena
Edited by Felix Fernandez-Alonso and David L. Price

Volume 49. Neutron Scattering - Applications in Biology, Chemistry, and Materials Science
Edited by Felix Fernandez-Alonso and David L. Price

Preface

Just over 80 years ago, a brief letter from James Chadwick to *Nature* [1,2] presented conclusive experimental evidence unveiling the existence of a neutral particle (nearly) isobaric with the proton. The discovery of the henceforth-to-be-known-as "neutron" had profound consequences for both scientific research and the destiny of humankind, as it led to the unleashing of the might of nuclear power in less than a decade [3].

The first use of these "neutral protons" to probe the microscopic underpinnings of the materials world around us also dates back to those early years, with pioneering neutron-diffraction experiments at Oak Ridge National Laboratory (USA) in the mid 1940s, and the subsequent development of neutron spectroscopy at Chalk River (Canada) in the 1950s. Since then, neutron-scattering techniques have matured into a robust and increasingly versatile toolkit for physicists, chemists, biologists, materials scientists, engineers, and technologists. At the turn of the last century, the 1994 Nobel Prize in Physics awarded to C.G. Shull and B.N. Brockhouse recognized their ground-breaking efforts towards the development and consolidation of neutron science as a discipline in its own right [4]. This milestone also served to define neutron scattering as the technique *par excellence* to investigate *where atoms are* (structure) and *what atoms do* (dynamics), a popular motto across generations of neutron-scattering practitioners.

Sustained and continued developments in experimental methods over the past few decades have greatly increased the sensitivity and range of applications of neutron scattering. While early measurements probed distances on the order of interatomic spacings (fractions of a nm) and characteristic times associated with lattice vibrations (picoseconds), contemporary neutron-scattering experiments can cover length scales from less than 0.01 to 1000 s of nanometers, and time scales from the attosecond to the microsecond. These advances have been made possible via a significant expansion of the range of neutron energies available to the experimenter, from micro-electron-volts (particularly at cold sources in research reactors) to hundreds of electron-volts (at pulsed spallation sources), as well as by unabated progress in the implementation of a variety of novel and ingenious ideas such as position- and polarization-sensitive detection or back-scattering and spin-labeling methods. As a result, neutron science has grown beyond traditional research areas, from the conventional determination of crystal structures and lattice dynamics of half-a-century ago (not to forget their magnetic analogs), to high-resolution structural studies of disordered thin films, liquid interfaces, biological structures, macromolecular and supramolecular architectures and devices, or the unraveling of the dynamics and energy-level structure of complex molecular solids, nanostructured materials and surfaces, or magnetic clusters and novel superconductors. Along with these scientific and technical developments, the community of neutron scientists has also expanded and diversified beyond recognition. Whereas the early stages of neutron scattering had its roots in condensed-matter physics and crystallography, present-day users of central neutron-scattering facilities include chemists, biologists, ceramicists, and metallurgists, to name a few, as well as physicists with an increasingly diverse range of transdisciplinary interests, from the foundations of quantum mechanics to soft matter, food science, biology, geology, or archeometry.

This book series seeks to cover in some detail the production and use of neutrons across the aforementioned disciplines, with a particular emphasis on technical and scientific developments over the past two decades. As such, it necessarily builds upon an earlier and very successful three-volume set edited by K. Sköld and D.L. Price, published in the 1980s by *Academic Press* as part of *Methods of Experimental Physics* (currently *Experimental Methods in the Physical Sciences*). Furthermore, new third-generation spallation sources have either been constructed in the United States and Japan, or are in the advanced construction stage in China. Likewise, the development of long-pulse, ultra-intense spallation neutron sources is well underway in Europe, including the construction of the European Spallation Source. These global efforts have been accompanied by an increasing interest in time-of-flight and broadband neutron-scattering techniques. Correspondingly, the improved performance of cold moderators at both reactors and spallation sources has extended long-wavelength capabilities to such an extent that a sharp distinction between fission- and accelerator-driven neutron sources may no longer be of relevance to the future of the discipline.

On a more practical front, the chapters that follow are meant to enable you to identify aspects of your work in which neutron-scattering techniques might contribute, conceive the important experiments to be done, assess what is required to carry them out, write a successful proposal to a user facility, and perform these experiments under the guidance and support of the appropriate facility-based scientist. The presentation is aimed at professionals at all levels, from early-career researchers to mature scientists who may be insufficiently aware or up-to-date with the breadth of opportunities provided by neutron techniques in their area of specialty. In this spirit, it does not aim to present a systematic and detailed development of the underlying theory, which may be found in superbly written texts such as those of Lovesey [5] or Squires [6]. Likewise, it is not a detailed hands-on manual of experimental methods, which in our opinion is best obtained directly from experienced practitioners or, alternatively, by attending practical training courses at the neutron facilities. As an intermediate (and highly advisable) step, we also note the existence of neutron-focused thematic schools, particularly those at Grenoble [7] and Oxford [8], both of which have been running on a regular basis since the 1990s. With these primary objectives in mind, each chapter focuses on well-defined areas of neutron science and has been written by a leading practitioner or practitioners of the application of neutron methods in that particular field.

The first volume of this series *Neutron Scattering – Fundamentals* [9] gave a self-contained survey of the theoretical concepts and formalism of the technique and established the notation used throughout the series. Subsequent chapters in this first volume reviewed neutron production and instrumentation, respectively, areas that have profited enormously from recent developments in accelerator physics, materials research and engineering, or computing, to name a few. The remaining chapters treated several basic applications of neutron scattering including the structure of complex materials, large-scale structures, and dynamics of atoms and molecules. The appendix went back to some requisite fundamentals linked to neutron-matter interactions, along with a detailed compilation of neutron scattering lengths and cross sections across the periodic table.

The second volume *Neutron Scattering – Magnetic and Quantum Phenomena* [10] placed the focus on traditional (and still highly popular) areas of neutron scattering. These included a detailed presentation of neutron optics and spin-labeling methods along with a broad introduction to the interaction of neutrons with electronic spins in condensed matter, a subject also of direct relevance to the further development of novel instrumentation at present and in the foreseeable future. The following chapters discussed recent developments in the use of neutron scattering to investigate quantum phase transitions, high-temperature superconductors, magnetic structures, multiferroics, nanomagnetism, and nuclear magnetism.

The present and final volume of the series is dedicated to the application of neutron scattering techniques across contemporary biology, chemistry, and materials science. As such, our presentation has been necessarily restricted to a handful of topics, in order to highlight and bring to the fore specific areas of activity that continue to gain importance and relevance. We start with the structure and dynamics of biological systems, followed by what one may consider as "more chemical" applications including water and aqueous systems, ionic liquids, catalysis on surfaces and nanostructured media, ionic and proton conductors, and soft matter. In some cases, we have chosen to place the emphasis on a detailed description of specific techniques and their recent use across disciplines. These include high-temperature and high-pressure techniques, or the (quite unique) use of epithermal neutrons to interrogate the quantum behavior of atoms in materials, and how it relates to function and performance. Our presentation is wrapped-up by an up-to-date overview of applications in engineering, an area which certainly links the technique to an everyday context quite close to our lives. Relative to the previous two volumes, we have found it much more difficult to keep the overall size of this third and final volume within reasonable bounds. In our view, this challenge is a testament to the growth and success witnessed by the field over the past two decades. The future of neutron scattering as a discipline, therefore, looks very bright!

In closing this preface, we wish to thank all authors for taking time out of their busy schedules to be part of this venture, Drs. T. Lucatorto, J.T. Yates, and K. Baldwin for inviting us to undertake this work, and the staff of *Academic Press* for their encouragement, diligence, and forbearance along the way.

Felix Fernandez-Alonso
David L. Price

REFERENCES

[1] J. Chadwick, Nature 129 (1932) 312.
[2] www.nobelprize.org/nobel_prizes/physics/laureates/1935/ (last accessed on 12 March 2017).
[3] A. MacKay, The Making of the Atomic Age, Oxford University Press, Oxford, 1984.
[4] www.nobelprize.org/nobel_prizes/physics/laureates/1994/ (last accessed on 12 March 2017).
[5] S.W. Lovesey, Theory of Neutron Scattering from Condensed Matter, vols. I and II, Oxford University Press, Oxford, 1986.
[6] G.L. Squires, Introduction to the Theory of Thermal Neutron Scattering, third ed., Cambridge University Press, Cambridge, 2012.
[7] www.hercules-school.eu/ (last accessed on 12 March 2017).
[8] www.oxfordneutronschool.org/ (last accessed on 12 March 2017).
[9] F. Fernandez-Alonso, D.L. Price (Eds.), Neutron Scattering – Fundamentals, Experimental Methods in the Physical Sciences, vol. 44, Academic Press, Amsterdam, 2013.
[10] F. Fernandez-Alonso, D.L. Price (Eds.), Neutron Scattering – Magnetic and Quantum Phenomena, Experimental Methods in the Physical Sciences, vol. 48, Academic Press, Amsterdam, 2015.