Characterization of Solid Materials and Heterogeneous Catalysts

The book *Characterization of Solid Materials and Heterogeneous Catalysts* consists of two volumes and has four themes. The first theme, "Molecular/Local Spectroscopies", is covered in Volume 1. The other three themes are covered in Volume 2; they are 1) "Macroscopic Techniques", 2) "Characterization of the Fluid Phase (Gas and/or Liquid)", and 3) "Advanced Characterization".

The book starts with a very useful "General Introduction", in which the editors classify the characterization techniques according to the nature of the incident and emitted beams (photons, electrons, neutrons) and the wavelength of the incident beam. They also provide tables that define SI units, interconvert energy and pressure units, and list values of fundamental physical constants. Their last table, on parameters relevant to selected physical techniques, is an easy guide for students and researchers in search of techniques to tackle a particular problem.

Each volume starts with two short general chapters written by two eminent scientists in the field: Nobel Prize winner G. Ertl discusses physical techniques for studying model solids, and J. M. Thomas gives an overview of physical techniques for studying porous solids.

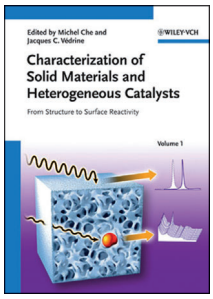
The first volume deals with an extremely wide range of spectroscopies. These include the most frequently used ones—such as infrared and Raman spectroscopies, UV/Vis/NIR, NMR, EPR, X-ray absorption, and X-ray and UV photoelectron and Auger electron spectroscopies—and the less well known ones such as Mössbauer, sum frequency generation, reflection–absorption spectroscopy, neutron scattering, SIMS, and the upcoming method of single-molecule spectroscopy. Each chapter follows a uniform pattern. It starts with a short historical overview. That is followed by the basic physical principles and technical/experimental aspects of good practice. Each chapter ends with a discussion of representative data that illustrate the possibilities offered by the technique. The procedures for using typical sample cells, the handling of samples before measurement, in-situ or operando spectroscopy, and the possibility of quantitative analysis are also treated. It is very difficult, if not impossible, to condense all these aspects of one technique into a chapter of 40–80 pages. Choices have to be made, and—for instance—the basic physical principles underlying each technique are kept to the very minimum. However, the reader obtains enough information to judge the pros and cons of each technique in relation to the subject being investigated. For a more in-depth knowledge the reader needs to study the specialized books.

Theme 2, "Macroscopic Techniques", covers methods that give long-range structural information, such as X-ray diffraction, TEM–SEM, and scanning probe microscopy, together with techniques that give macroscopic structural information such as surface areas and pore volumes. There is also a long chapter devoted to thermal methods. It includes calorimetry, thermogravimetry, temperature-programmed reduction and oxidation, and acidity/basicity probing of surfaces. Theme 3, "Characterization of the Fluid Phase", covers mass spectrometry, chromatography, and transient techniques. The last theme, "Advanced Characterization", has two chapters, one on combining two or more techniques for the study of a particular sample, and one on quantum chemistry and modeling.

In the last 10–15 years we have witnessed the growing importance of 1) in situ/operando measurements, 2) combinations of techniques, and 3) modeling and quantum chemistry. These three aspects are covered in the book, but they do not form the main body of the work. Combinations of techniques and quantum chemistry are covered in the last two chapters of Volume 2, but they are not well integrated with the other techniques that are described in the book. In situ/operando spectroscopy is included in each chapter where appropriate.

To convey an understanding of characterization techniques, it is necessary to explain the underlying fundamental physical principles, and to discuss how these physical principles are translated into chemistry/materials science. Students with a knowledge of chemistry need more in-depth knowledge of the basic physics underlying each technique. On the other hand, students with a background in physics require a knowledge of the chemistry for interpretation of data. The authors of the chapters have tried to achieve a good balance between physics and chemistry. In most cases, however, the physical background has been kept to the very minimum.

This book offers a general introduction to the wide range of techniques that one can use to characterize solids, in particular heterogeneous catalysts. The book is well written by experts in the field. I have not found errors. In the chapter on electron spin resonance, the authors did not use the dimensionless electron spin angular momentum. As a consequence, they had to include $h/2\pi$ in the denominator of the expressions for the spin magnetic moment and the energy. This is unusual. In almost all textbooks on electron spin resonance, the authors use the dimensionless spin angular momentum, and that division by $h/2\pi$ does not occur in the



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expressions for the magnetic moment and the energy. The title mentions both materials and heterogeneous catalysts, but the emphasis is clearly on the latter. The book is useful as a general introduction and as a source of literature references. I see the following uses of the book: 1) as a general reference book that must be available in a research laboratory for a quick introduction to a technique and for screening of techniques; 2) the beginning PhD student can consult the book when searching for techniques to apply; 3) it is a quick guide for selection of a new technique to be introduced into the laboratory.

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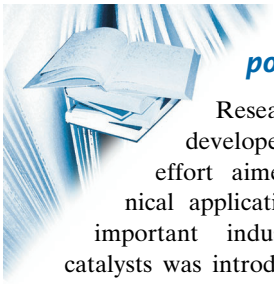


Diffusion in Nanoporous Materials

Research on zeolites has developed into an intensive effort aimed towards their technical applications, since their use as important industrial adsorbents and catalysts was introduced by Union Carbide in the 1950s. New technological developments in oxidation catalysis (e.g., in the production of propylene oxide by BASF) and in the treatment of exhaust gases are two current examples of applications that continue to drive the growth in the importance of zeolites in heterogeneous catalysis. Also, the vital role of zeolite catalysts in the production of liquid fuels (methanol-to-olefins and methanol-to-gasoline processes) is now experiencing a new upturn after some years of lower activity. The diffusion of molecules in zeolite pores is fundamental for their wide range of applications. Developments in mesoporous materials with ordered pore structures and in porous coordination polymers (metal–organic frameworks, MOFs) are two new areas for theoretical and experimental studies on diffusion applications.

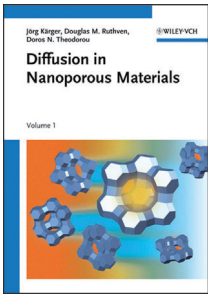
Following on from the 1992 book *Diffusion in Zeolites and Other Microporous Solids* by J. Kärger and D. M. Ruthven, which has become a classic, the title of their new work *Diffusion in Nanoporous Materials* reflects the fact that it is directed to a much broader subject area, which also includes new developments such as mesoporous materials and MOFs. In order to ensure that the rapidly growing importance of theoretical methods and molecular modeling applied to diffusion processes is expertly covered, D. N. Theodorou, one of the leading experts in this field, was persuaded to become a co-author of this work. The authors have taken the opportunity to include modern developments in techniques for measuring diffusion, such as quasi-elastic neutron scattering, NMR and micro-imaging techniques, and single molecule detection, to name just a few.

The two volumes are quite distinct in the nature of their contents. While the first volume gives a comprehensive overview of the fundamentals of the physics of diffusion and of experimental measurement techniques, the second volume is primarily devoted to the application of these principles to different materials and the importance of diffusion in separation processes and heterogeneous catalysis.

In the first volume, starting from the basic principles and the description of motion using the random-walk model, the authors focus on the peculiarities of the theory of diffusion in a porous environment. This volume ends with several long chapters on the principles of simulation methods and the measurement of diffusion processes. The reader is given an easily understandable explanation of the importance of differences in length scales and in diffusion properties (transport diffusion, self-diffusion), as well as in sample properties (surface resistance, barriers to internal transport) when different experimental methods are employed.

The second volume deals with diffusion in some specific systems. The subject matter is arranged according to the classification of the materials (e.g., on the basis of pore diameters) and their technological purpose (e.g., separation, catalysis), and through this arrangement one also obtains an excellent literature survey, including important applications in various technological processes.

More recently, new developments in the area of porous materials have been focused on the preparation of hierarchical materials with micro- and mesopores, on the development of zeolites for use in membranes, and on efforts to synthesize nanocrystals. Although some of these subdisciplines are relatively new and there is still a comparative lack of relevant experimental diffusion data, the two volumes by Kärger et al. offer a comprehensive description of the underlying fundamental principles that apply, for example, to the different diffusion mechanisms in pores of different size, or to the influence of particle size on the results obtained by different experimental methods. The authors explain very clearly the structural and dynamic factors that lead to the discrepancies often found between the results obtained with different experimental techniques for measuring diffusion, such as PFG–NMR (pulsed field gradient NMR



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