



Book Review: In-Situ Spectroscopy in Heterogeneous Catalysis

Edited by James Haw

F. C. Jentoft

Fritz-Haber-Institut of the Max Planck Society Faradayweg 4-6, 14195 Berlin

Because of the necessity to study heterogeneous catalysts "at work", in situ methods have developed considerably in the past decades, and the field appears ready for a comprehensive overview. The book "In-Situ Spectroscopy in Heterogeneous Catalysis", edited by James Haw, is a collection of individual articles on selected methods (not all of them spectroscopies), written by specialists. The intent is to attract "young scientists with training in diverse areas" to heterogeneous catalysis research.

In his introduction the editor briefly addresses pressure gap, materials gap, spectator species, and the impact of theory, and he uses these issues to refer to each of the chapters. Haw also attempts to define "in situ". This section is called "compromises" and accordingly, the author allows not only the "purest definition" but also a pragmatic approach in which he reduces "in situ" to any study that "teaches us about the chemistry [...] under reaction conditions".

The structure of the articles is similar, after an introduction into the technique a number of examples are provided. One issue is certainly, how far such a book should address the principles of the techniques themselves. This is resolved quite well in that only the more exotic techniques that are not part of textbooks are introduced at length. A plus is the even level of difficulty of the articles. The experimentalist who would wish to profit from developments in techniques other than his or her own must resort to the secondary literature.

Graphs with schemes of setups or experimental data are abundant and constitute part of the quality of the book; mostly they are equally well reproduced and labeled. The extensive use of subheadings in all chapters is very useful, and all of these subheadings are reproduced in the table of contents, which thus reflects the structure of the articles well. The table of contents is a better means to find something in the book than is the index, which is characterized by mistakes and repetitions of terms in slight modifications.

The book starts with a chapter on surface science whose first part is dedicated to the SFG method and nicely demonstrates the power of a true in situ experiment, showing plots in which catalytic and spectroscopic data - from CO oxidation on Pt - are really correlated. The second chapter, on STM, deals with adsorbed species rather than actual catalysis but brings up an, in principle, important subject, i.e. processes induced by the experiment ("tip-induced catalysis").

The chapter written by the editor presents an enlightening overview of the ways to employ NMR for in situ studies in catalysis. The most suitable nucleus that can be probed and thus the focus is ^{13}C , delivering information on surface species and products, not on changes of the catalyst itself. The examples show the tedious and often indirect approach of this method. The author is very honest about limitations, admitting what applies to most of the techniques, "[NMR] cannot work alone"; and follows this up with an example where an additional technique provides complementary information.

The chapter on theory gives an overview of the principles of different methods, leading to a tour de force through the acronyms of theoretical chemistry. A definite strength is the vast list of references. The examples are all from the field of acid-base catalysis, are all based on cluster models, and compare the calculated energies (activation, adsorption) or NMR isotropic shifts to experimental results.

The Raman spectroscopy chapter is focussed on the author's experimental setup (UV-Raman) and work. While the challenges of an in situ Raman application are laid out clearly, the examples - one of them not from the field of catalysis - fail to demonstrate the benefits of an in situ experiment; the nature of coke on a zeolite and the decomposition of lubricants upon grinding with different tools are reported.

The XAS chapter emphasizes technique and thereof selected aspects. Some recent cell developments as well as

the important quick and dispersive XAS techniques are not mentioned. The complications of the data analysis remain sketchy and the examples are extremely condensed. A useful hint is made towards the application of in situ techniques to monitor catalyst preparation.

A detailed introduction is given into the applications of positron emission for in situ research, followed by the study of axial concentration profiles in flow reactors. This longest chapter of the book includes extensive descriptions of the mathematical modeling of the data, i.e. of concentration profiles in a powder bed.

The IR and the TAP (temporal analysis of products) chapters are informative and fun to read. A variety of IR techniques, among them again SFG, is presented including descriptions of cell constructions. The examples are diverse, and show the strengths and limitations of the IR method very well. The TAP chapter captivates through its

structure and clarity; it covers all aspects from types of experiments to analyzing the data.

Unclear remains why certain spectroscopies such as e.g. UV-vis or EPR, and surface science methods that are inching into interesting pressure ranges such as XPS, and environmental microscopy have been entirely omitted and are not even addressed in the introductory overview. A chapter compiling information on all techniques, such as attainable temperature and pressure ranges, sensitivities, limitations, etc. in a few tables to allow a critical comparison would have set the book off as an entity in itself as compared to review articles on single methods. Nevertheless, the book offers interesting material for any researcher with a background in catalysis and also for the specialist who wants to extend his knowledge.