

MATERIALS CHARACTERIZATION SERIES

SERIES EDITORS: **C. Richard Brundle** and **Charles A. Evans, Jr.**

材料表征原版系列丛书

催化材料的表征

CHARACTERIZATION OF

Catalytic Materials

Israel E. Wachs

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哈爾濱工業大學出版社
HARBIN INSTITUTE OF TECHNOLOGY PRESS

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Characterization of Catalytic Materials

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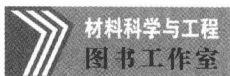
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CHARACTERIZATION OF CATALYTIC MATERIALS

EDITOR

Israel E. Wachs

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C. Richard Brundle and Charles A. Evans, Jr.



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Preface to the Reissue of the Materials Characterization Series

The 11 volumes in the Materials Characterization Series were originally published between 1993 and 1996. They were intended to be complemented by the *Encyclopedia of Materials Characterization*, which provided a description of the analytical techniques most widely referred to in the individual volumes of the series. The individual materials characterization volumes are no longer in print, so we are reissuing them under this new imprint.

The idea of approaching materials characterization from the material user's perspective rather than the analytical expert's perspective still has great value, and though there have been advances in the materials discussed in each volume, the basic issues involved in their characterization have remained largely the same. The intent with this reissue is, first, to make the original information available once more, and then to gradually update each volume, releasing the changes as they occur by on-line subscription.

C. R. Brundle and C. A. Evans, October 2009

Preface to Series

This Materials Characterization Series attempts to address the needs of the practical materials user, with an emphasis on the newer areas of surface, interface, and thin film microcharacterization. The Series is composed of the leading volume, *Encyclopedia of Materials Characterization*, and a set of about 10 subsequent volumes concentrating on characterization of individual materials classes.

In the *Encyclopedia*, 50 brief articles (each 10 to 18 pages in length) are presented in a standard format designed for ease of reader access, with straightforward technique descriptions and examples of their practical use. In addition to the articles, there are one-page summaries for every technique, introductory summaries to groupings of related techniques, a complete glossary of acronyms, and a tabular comparison of the major features of all 50 techniques.

The 10 volumes in the Series on characterization of particular materials classes include volumes on silicon processing, metals and alloys, catalytic materials, integrated circuit packaging, etc. Characterization is approached from the materials user's point of view. Thus, in general, the format is based on properties, processing steps, materials classification, etc., rather than on a technique. The emphasis of all volumes is on surfaces, interfaces, and thin films, but the emphasis varies depending on the relative importance of these areas for the materials class concerned. Appendixes in each volume reproduce the relevant one-page summaries from the *Encyclopedia* and provide longer summaries for any techniques referred to that are not covered in the *Encyclopedia*.

The concept for the Series came from discussion with Marjan Bace of Manning Publications Company. A gap exists between the way materials characterization is often presented and the needs of a large segment of the audience—the materials user, process engineer, manager, or student. In our experience, when, at the end of talks or courses on analytical techniques, a question is asked on how a particular material (or processing) characterization problem can be addressed the answer often is that the speaker is “an expert on the technique, not the materials aspects, and does not have experience with that particular situation.” This Series is an attempt to bridge this gap by approaching characterization problems from the side of the materials user rather than from that of the analytical techniques expert.

We would like to thank Marjan Bace for putting forward the original concept, Shaun Wilson of Charles Evans and Associates and Yale Strausser of Surface Science Laboratories for help in further defining the Series, and the Editors of all the individual volumes for their efforts to produce practical, materials user based volumes.

C. R. Brundle C. A. Evans, Jr.

Preface to the Reissue of *Characterization of Catalytic Material*

This comprehensive volume on catalytic materials, catalytic properties, and the techniques needed to characterize both materials and properties over the wide range involved, was put together by 11 individual experts, split between academia, oil company research and engineering laboratories, and a national laboratory. Though, of course, there have been technological advances in the areas covered since the original publication, the methodology discussed for characterization and evaluation of catalysts remains as valid today as it was then. After reissuing the volume in close to its original form, it is our intent to release updates to individual chapters, plus new material, as on-line downloads, as they become available.

C. R. Brundle and C. A. Evans, Jr., January 2010

Preface

Heterogeneous catalysis has undergone a revolutionary change in the past two decades due to the development of sophisticated characterization methods that provide fundamental information about the catalyst bulk structures, surfaces, and their properties. For the first time, these characterization methods have allowed researchers to “see” the surfaces of catalytic materials, their bulk structures (crystalline as well as amorphous phases), the influence of the process conditions on the catalytic material, as well as the effect of different synthesis methods. This new information has tremendously advanced our understanding of catalytic materials and their properties. These characterization methods have become our “eyes” and are indispensable in the development of new catalytic materials. It is hard to conceive of a modern heterogeneous catalysis activity, be it research or manufacturing, without the aid of these new characterization techniques.

Catalytic materials exist in various forms (bulk metals and alloys, supported metals, bulk metal oxides, supported metal oxides, bulk metal sulfides, supported metal sulfides, zeolites, molecular sieves, and pillared clays) which, consequently, require somewhat different characterization approaches. Thus, catalytic scientists and engineers specializing in one area of heterogeneous catalysis may not be intimately familiar with other areas of heterogeneous catalysis since vastly different characterization methods may be required. For example, catalytic scientists and engineers who typically were involved with supported metal catalysts find that they have to familiarize themselves with rather different characterization methods when they wish to study metal oxide catalytic materials (bulk metal oxides, supported metal oxides, zeolites, molecular sieves, and pillared clays). Similarly, scientists and engineers new to the area of heterogeneous catalysis need some guidance as to the applicable characterization methods. To satisfy these requirements this volume is organized by type of heterogeneous catalytic material, and emphasizes the different properties that can be determined by various characterization methods for each class of material. The Series, of which this volume is part, has the title “Materials Characterization: Surfaces, Interfaces, Thin Films” and the general concept, or intent is to provide a practical guide to people working in various materials classes within the framework of this title. In the case of *Characterization of Catalytic Materials*, the emphasis is not always on surfaces, thin films, or interfaces. For those catalytic materials that possess the active component at the surface, the surface characterization methods are critical, even though we currently have still some way to go before we can say that all the important information can be readily accessed. For those materials that possess the active catalytic component in the bulk, the bulk characterization methods are critical. An attempt has been made to focus many of the chapters on the catalytic properties to be measured rather than on the details of the characterization techniques. Thus, the information required in order to understand the structure and performance

of a given catalytic material is emphasized. For ease of reference, short summaries of the techniques discussed in the book are presented in a collection of appendixes. This book is a practical guide for the characterization of catalytic materials as it is done today.

I want to thank the authors who made this book possible and were responsible for the individual chapters on different catalytic materials. It was a pleasure, as well as an education, to work with individuals who are experts in their respective fields. The high quality of the chapters greatly simplified my task as Editor. I also wish to acknowledge our Managing Editor, Lee Fitzpatrick, who orchestrated the publication in an efficient and professional manner.

Israel E. Wachs

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Bulk Metal Oxides

Alumina Pillared Clays:
Methods of Preparation and Characterization

Bulk Metal Sulfides

Zeolites and Molecular Sieves

Zeolites and Molecular Sieves

Supported Metals

Alumina Pillared Clays:
Methods of Preparation and Characterization

Supported Metal Sulfides

Bulk Metals and Alloys

Supported Metal Oxides

Supported Metal Oxides

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Bulk Metals and Alloys

JOHANNES SCHWANK

Contents

- 1.1 Introduction
- 1.2 Preparation of Bulk Alloy or Bimetallic Catalysts
- 1.3 Bulk Metal Characterization Methods
- 1.4 Surface Composition–Structure and Catalysis Relationship

1.1 Introduction

The Role of Metals and Alloys in Catalysis

Metals and alloys play a key role in catalytic technology, especially in reactions involving hydrogen transfer and in hydrocarbon conversion reactions. This chapter discusses the characterization of catalytic metals and alloys in their bulk form. Many catalytic applications require catalysts with high surface area to provide adequate contact between the reactants and the catalyst. Therefore, a large number of catalytic processes rely on supported-metal catalysts, in which the metal particles are well dispersed on a high surface area support material. However, unsupported bulk metals or alloys are sometimes employed in the form of films, foils, wires, or powders. Because of their high catalytic activity, platinum group metals find the widest application. The reactions of hydrocarbons on Pt surfaces include hydrogenation and dehydrogenation; hydrogenolysis of C–C, C–S, and C–N bonds; isomerization; and cyclization reactions.

On an industrial scale, ammonia is oxidized to nitric acid in the presence of a Pt–Rh wire gauze or Pd–Au alloy wires. Palladium–gold alloys are very effective catalysts for the selective hydrogenation of unsaturated hydrocarbons, such as the hydrogenation of acetylene to ethylene. Catalytic hydrogenation can also be accomplished over Raney nickel or cobalt catalysts. Silver gauze or granular silver screened