MATERIALS CHARACTERIZATION SERIES

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

材料表征原版系列丛书

陶瓷的表征

CHARACTERIZATION OF Ceramics

Ronald E. Loehman

McGraw Hill Education

哈爾濱工業大學出版社
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CHARACTERIZATION OF CERAMICS

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MATERIALS CHARACTERIZATION SERIES
Surfaces, Interfaces, Thin Films

Series Editors: 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 inl 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 case 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 Ceramics*

Fifteen specialists (ten from Sandia National Laboratories) combined their efforts to produce this comprehensive volume. Between them, they addressed the concerns and recommendations for the ceramics area presented in the National Academy of Sciences study, "Materials Science in the 1990's: Maintaining Competitiveness in the Age of Materials." The first five chapters deal with synthesis and processing, and the remainder cover structure, reaction, mechanical properties, composites and joining, and electronic and magnetic ceramics, all with an emphasis on characterization. Of course, there have been advances since the original publication, particularly with micro aspects being pushed down to the nano region, but all the principles involved in the characterization approaches discussed here remain valid and pertinent. Following the reissue of this volume, in a form close to the original, it is our intention to release updates and new material, as on-line downloads, as they become available.

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

Preface

Most ceramics are ionically bonded compounds found in complex crystal structures that are strong, stiff, lightweight, hard, and corrosion-resistant. Typically, they maintain their properties to high temperatures. In a broad sense, a ceramic is any manmade, inorganic, nonmetallic, solid material. Glass, usually considered a subset of ceramics, is any solid that lacks crystalline order. Traditionally, ceramics have been considered polycrystalline, although most ceramists today do not accept that restriction. Also traditional is the idea that high temperatures are required for the synthesis or processing of ceramics and glasses; but that limitation is no longer valid—new materials such as aerogels and tin fluorophosphate glasses are synthesized at room temperature or at a few hundred degrees above ambient.

Raw materials for ceramic and glass manufacture traditionally are earthy, oxide materials that are mined in high volume at low cost and are subjected to relatively little processing. The products made from them are commodity items such as brick, tile, bottles, and windows. Modern technical or engineering ceramics are higher-value materials that have superior properties as a result of more sophisticated processing and tighter control over raw materials. These advanced ceramics are much more varied in composition than simple oxides and include, for example, carbides, nitrides, and borides. The development of ceramic composites that are heterogeneous on the micrometer or nanometer scale is a rapidly expanding area of materials science and engineering. The need for better control of final properties requires increased use of modern characterization techniques at all stages of ceramic synthesis and processing. This volume describes characterization techniques and how they can be used to obtain that greater control.

This book is written in a time of changing priorities in materials science and engineering. Responding to a perception that research results in the United States were not being reliably translated into marketable products, the U.S. National Academy of Sciences conducted an influential study—the results of which were reported in a widely read book, *Materials Science in the 1990s: Maintaining Competitiveness in the Age of Materials*, National Academy Press, Washington, D.C., 1989—that recommended increased emphasis on materials synthesis and processing. These recommendations include

- interactive research on new materials synthesis that is linked with characterization and analysis of the product
- basic research on synthetic solid-state inorganic chemistry to produce new compounds

- synthesis of ultra pure materials, for example, fibers with low oxygen or carbon impurity levels
- research on techniques for synthesis to net-shape; that is, learning how to do synthesis, processing, and forming in a single step
- research on methods for processing ceramic materials far from equilibrium
- research on processing artificially structured or, as they are sometimes called, functionally gradient materials.

Characterization of Ceramics addresses these concerns and recommendations in two ways. First, the book stresses advanced synthesis and processing. Second, the central theme of the book, the application of characterization techniques, is a specific recommendation of the NAS study. The 13 chapters of this volume present a broad overview of ceramics and glasses. Each of the topics provides enough information for the reader to make intelligent choices among the myriad available characterization and analysis techniques. Many of the chapters are organized as case studies taken from the authors' own research, which help to illustrate how different methods can be integrated to give a more complete picture of a given process or phenomenon.

The first part of the book deals with the techniques of ceramic synthesis. Increasingly, advanced ceramics are being produced from highly processed powders made by methods collectively known as chemical preparation. Some of the more promising routes to the production of advanced ceramic powders are sol–gel processing, precipitation from solution, gas-phase synthesis, and powder-surface modification. J. A. Voigt discusses recent trends in the use of near–room temperature solution techniques to make ceramic precursors. An example of this is the sol–gel method, in which organometallic reagents in solution are hydrolyzed and condensed to form an inorganic polymeric gel that, when dried and fired, gives the desired ceramic composition. These chemical methods can generate controlled-size distributions, extremely reactive precursors, unusually shaped particles, and gels. Solution methods permit the intimate mixing of components, easy dispersion of second phases, and surface modification of precursor particles. Liquid precursor solutions also can be used to make thin films by dipping or spinning; because of the high reactivity of the precursor particles, film consolidation occurs at moderate temperatures. The chapter by R. W. Schwartz on electronic ceramics shows how analytical methods such as NMR are used to guide the solution synthesis of electronic ceramic films such as PZT (lead zirconate-lead titanate). Voig's chapter illustrates the importance of thorough characterization in the development of better synthesis methods.

Ceramic powders and films made by gas-phase techniques and their characterization are discussed by C. L. J. Adkins and D. E. Peebles. Ultrafine ceramic particles with enhanced surface reactivity, such as SiO_2, can be synthesized through nucleation or condensation reactions in gas-phase aerosols. Ceramic films and coatings, such as diamond and diamond-like materials, are synthesized by a variety of vapor deposition techniques.

Ceramic processing methods are extremely diverse, with new ones being constantly developed. The technique with widest application is sintering bulk ceramics, in which a powder preform is typically converted to a dense, consolidated object through solid or liquid-phase diffusion. The driving force for diffusion is the lowering of the Gibb's energy by minimizing surface area and, possibly, by reaction to more stable products. K. G. Ewsuk discusses the essential features of bulk sintering and the analytical methods used to characterize the process. By contrast, T. J. Garino's chapter is concerned with the densification of ceramic thick films and the phenomena distinctive to them. For example, ceramic films usually are deposited on substrates, and differential shrinkage in drying or firing leads to stresses and possibly warping. Garino's discussion emphasizes characterization methods applicable to those ceramic films.

Much current ceramic processing research for both bulk materials and films is directed toward eliminating flaws, thereby increasing strength and fracture toughness. L. Neergaard's chapter on nondestructive evaluation shows how to detect flaws that are frequently generated in a ceramic despite the best of processing efforts.

Other types of ceramic processing discussed in this volume are inorganic glasses and glass-ceramics by R. K. Brow, ceramic composites by S. J. Glass, and ceramic joining by A. P. Tomsia. This selection of processing methods is not exhaustive, but is broad enough for most of the applicable characterization techniques to be presented. These three chapters share a common concern with interfaces and how to characterize their reactivities, compositions, and microstructures.

Because ceramics are brittle, they are susceptible to catastrophic failure under mechanical load. The useful strength of a ceramic is determined by the flaw population: stresses are concentrated at flaws, which cause cracks to propagate to failure. The critical property for ceramics in load-bearing uses is not the strength, but the fracture toughness—the resistance of the ceramic to crack propagation. The fracture surface of a ceramic bears the evidence of its failure. One must read the features in a fracture surface to understand the origin and path of the fracture. The case study by E. K. Beauchamp shows how much practical information can be obtained from ceramic fracture analysis.

The other two chapters are basic to much of ceramics. In ceramics, microstructure determines properties; the study of that relationship has been a main theme for decades. A. H. Carim's chapter illustrates the range of microscopic and microanalytic techniques used to determine the structures and composition of ceramic microstructures. Another foundation of ceramics is reactivity and phase behavior. Knowledge of these topics is basic to understanding all forms of thermal processing of ceramics. P. K. Gallagher's chapter on reactivity and thermal analysis is an authoritative account by one of the experts of the field.

Ronald E. Loehman

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Contents

Preface to the Reissue of the Materials Characterization Series xi

Preface to Series xii

Preface to the Reissue of *Characterization of Ceramics* xiii

Preface xiv

Contributors xvii

POWDER AND PRECURSOR PREPARATION BY SOLUTION TECHNIQUES

1.1 Introduction 1
Mixed Oxide Processing 2, Chemical Synthesis of Powders 2

1.2 Powder Characterization 3
Physical Characteristics 3, Chemical Properties 4

1.3 Precursor Powder Synthesis 8
Speciation and Supersaturation 8, Growth 10, Nucleation 19, Agglomeration 22

1.4 Summary 23

POWDER PREPARATION BY GAS-PHASE TECHNIQUES

2.1 Introduction 29

2.2 Powder Production by Thermal Decomposition Techniques 30
Aerosol Precursor Processes 30, Vapor Precursor Processes 33

2.3 Powder Production by Plasma Techniques 35

2.4 Powder Production by Supercritical Fluid Techniques 37

2.5 Powder Characterization 39

2.6 Summary 40

FORMATION OF CERAMIC FILMS AND COATINGS

3.1 Introduction 43

3.2 Film Deposition and Coating Processes 44
Physical Vapor Deposition 44, Chemical Vapor Deposition 45, Solution and Sol–Gel Techniques 45, Thermal Spray Processing 46, Hard Carbon Coatings 46

3.3 Physical Characterization 47
Density, Porosity and Voids 47, Morphology 48, Thickness 48, Surface Finish 49

3.4 Chemical Characterization 50
Elemental Analysis 50, Chemical State Analysis 53, Microstructure 56

3.5 Mechanical Characterization 57
Adhesion 57, Hardness 59, Internal Stress 60

3.6 Summary 60

CONSOLIDATION OF CERAMIC THICK FILMS

4.1 Introduction 63

4.2 Thick Film Processing 64

4.3 Characterization of Ceramic Thick Film Consolidation 65
Characterization of Films Before Thermal Processing 65, Characterization of Thick Films During Thermal Processing 68, Characterization of Sintered Thick Films 70

4.4 Summary 75

CONSOLIDATION OF BULK CERAMICS

5.1 Introduction 77

5.2 Ceramic Consolidation 78
Green Body Fabrication 78, Pre-Sinter Thermal Processing 79, Sintering/Thermal Consolidation 80

5.3 Characterization of Ceramics 82
Characteristics and Characterization of Green Ceramic Compacts 83, Characterization of Pre-Sinter Thermal Processes 90, Characteristics and Characterization of Sintered Ceramics 90

5.4 Summary 96

INORGANIC GLASSES AND GLASS-CERAMICS

6.1 Introduction 103

6.2 Possible Surface Analytical Artifacts 104

6.3 XPS Studies of Bonding in Glass 108

6.4 Corrosion in Water 110
Water Vapor 111, Aqueous Solutions 112

6.5 Glass Crystallization 114

CERAMIC MICROSTRUCTURES

7.1 Introduction 119

7.2 Bulk Microstructural Features 120
Grain Size, Shape, and Growth 120, Connectivity 122, Boundary Layers and Inclusions 123, Porosity and Density 123

7.3 Interfaces and Planar Defects 124
Grain Boundaries and Domain Boundaries 124, Heterogeneous Interfaces 125, Stacking Faults and Twins 126

7.4 Dislocations 127

7.5 Methods of Phase Identification 129
Phase Distribution 130, Crystal Structure of Phases 131, Chemical Composition of Phases 132

7.6 Stereology for Phase Quantification 133
Grain Size and Mean Lineal Intercept 134, Volume Fraction of Phases 135

7.7 Summary 135

CERAMIC REACTIONS AND PHASE BEHAVIOR

8.1 Introduction 137

8.2 Starting Materials 140

8.3 Phase Equilibria 140
General Aspects 140, Determining the Chemical and Structural Aspects 141, Determining the Physical Variables 154

8.4 Rates and Mechanisms of Reaction 156
General Considerations 156, Decomposition of Precursors 158, Solid-Solid Reactions 161, Solid–Liquid Reactions 164, Solid–Gas Reactions 165

8.5 Summary 166