

MATERIALS CHARACTERIZATION[®] SERIES

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

材料表征原版系列丛书

硅加工中的表征

CHARACTERIZATION IN

Silicon Processing

Yale E. Strausser



哈尔滨工业大学出版社
HARBIN INSTITUTE OF TECHNOLOGY PRESS

MATERIALS CHARACTERIZATION

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

材料表征原版系列丛书

硅加工中的表征

CHARACTERIZATION IN **Silicon Processing**

Yale E. Strausser

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

黑版贸审字08-2013-079号

Yale E. Strausser

Characterization in Silicon Processing

9781606501092

Copyright © 2010 by Momentum Press, LLC

All rights reserved.

Originally published by Momentum Press, LLC

English reprint rights arranged with Momentum Press, LLC through McGraw-Hill Education (Asia)

This edition is authorized for sale in the People's Republic of China only, excluding Hong Kong, Macao SAR and Taiwan.

本书封面贴有McGraw-Hill Education公司防伪标签,无标签者不得销售。

版权所有,侵权必究。

图书在版编目(CIP)数据

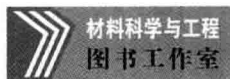
硅加工中的表征:英文/(美)布伦德尔(Brundle C. R.), (美)埃文斯(Evans C. A.), (美)斯特劳瑟(Strausser Y. E.)主编. —哈尔滨:哈尔滨工业大学出版社, 2014.1

(材料表征原版系列丛书)

ISBN 978-7-5603-4280-1

I. ① 硅… II. ① 布… ② 埃… ③ 斯… III. ① 半导体工艺—研究—英文 IV. ① TN305

中国版本图书馆CIP数据核字(2013)第273279号



责任编辑 杨 桦 许雅莹 张秀华

出版发行 哈尔滨工业大学出版社

社 址 哈尔滨市南岗区复华四道街10号 邮编 150006

传 真 0451-86414749

网 址 <http://hitpress.hit.edu.cn>

印 刷 哈尔滨市石桥印务有限公司

开 本 660mm × 980mm 1/16 印张 16.25

版 次 2014年1月第1版 2014年1月第1次印刷

书 号 ISBN 978-7-5603-4280-1

定 价 88.00元

(如因印刷质量问题影响阅读,我社负责调换)

CHARACTERIZATION IN SILICON PROCESSING

EDITOR

Yale E. Strausser

SERIES EDITORS

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



MOMENTUM PRESS

MOMENTUM PRESS, LLC, NEW YORK

MATERIALS CHARACTERIZATION SERIES

Surfaces, Interfaces, Thin Films

Series Editors: C. Richard Brundle and Charles A. Evans, Jr.

Series Titles

Encyclopedia of Materials Characterization, C. Richard Brundle, Charles A. Evans, Jr., and Shaun Wilson

Characterization of Metals and Alloys, Paul H. Holloway and P. N. Vaidyanathan

Characterization of Ceramics, Ronald E. Loehman

Characterization of Polymers, Ned J. Chou, Steven P. Kowalczyk, Ravi Saraf, and Ho-Ming Tong

Characterization in Silicon Processing, Yale Strausser

Characterization in Compound Semiconductor Processing, Yale Strausser

Characterization of Integrated Circuit Packaging Materials, Thomas M. Moore and Robert G. McKenna

Characterization of Catalytic Materials, Israel E. Wachs

Characterization of Composite Materials, Hatsuo Ishida

Characterization of Optical Materials, Gregory J. Exarhos

Characterization of Tribological Materials, William A. Glaeser

Characterization of Organic Thin Films, Abraham Ulman

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 in Silicon Processing*

When this volume was originally released in 1993 the chapters dealt with materials and manufacturing processes in the state of the art wafer fabrication facilities of the day. Mature fabs today still use many of those materials and processes, though state of the art fabs of today also include both different materials (e.g., Cu interconnects, organic dielectrics) and processing steps which did not exist then. After the reissue of this volume in a form close to the original, updates covering the changes that have occurred will be made and released as on-line downloads as they are completed.

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

Preface

This volume has been written to aid materials users working with silicon-based semiconductor systems. Materials problems arise in all stages of semiconductor device production: research and development of new processes, devices, or integrated circuit technologies; new process equipment definition and new process start-up; operation of state-of-the-art processes in wafer fabrication facilities; and throughout the life of each wafer fabrication process.

These materials problems are sometimes investigated using only electrical tests, but they can often be more clearly identified by using an appropriate selection of materials characterization techniques. However, the research and development scientists and engineers who work with new technologies and define or implement new processes are typically not experts in these techniques. This volume, and indeed the Materials Characterization Series, is intended to help the nonspecialist determine the best selection of techniques for a surface- or thin film materials-based problem.

This volume should be used in conjunction with the lead volume of the series, *Encyclopedia of Materials Characterization*, which defines boundary conditions for fifty widely used surface and thin-film materials characterization techniques. Each technique description discusses

- the type of information to be obtained about a sample
- appropriate samples and required sample preparation
- limitations and hardware requirements with regard to spatial resolution, compositional resolution, and sensitivity
- time required for an analysis
- destructiveness to the sample
- other important characteristics of the technique.

Each technique description also lists authoritative references for further research. The descriptions are succinct and do not discuss operation of the instruments or lengthy derivations of basic principles. They are jargon-free guidelines to aid the nonspecialist in understanding the type of information a technique provides and in selecting the appropriate technique to solve a problem.

This volume approaches materials characterization from the materials properties, processing, and problems point of view. It discusses typical materials and processes used in the manufacture of today's silicon-based semiconductor devices and

provides examples of typical problems encountered in the real silicon-processing world and their identification and characterization using techniques described in the *Encyclopedia*.

The organization of the chapters in this volume is similar to the process flow of a wafer. Each material commonly used in silicon integrated circuit manufacture is the topic of a chapter, including epitaxial silicon (including silicon-germanium alloys), polycrystalline silicon, metal silicides, aluminum and copper conductors, tungsten conductors, and barrier films. Dielectric films are not covered. Each chapter discusses a typical process history of the material—deposition, thermal treatment, lithography, etc.—and the desired properties of the material, with examples of common problems seen in producing materials having the desired properties. These examples illustrate the application of appropriate characterization techniques to solve the problems.

The fifty techniques discussed in the *Encyclopedia* are the most widely used for a broad range of materials problems. Some of these techniques are seldom used in characterizing silicon-based semiconductor materials, and some techniques specific to semiconductor characterization are not included in the *Encyclopedia*. For these reasons, an appendix is provided in this volume that contains pertinent summary pages taken from the *Encyclopedia* plus lengthier descriptions of the important semiconductor-specific methods not covered in the *Encyclopedia*.

This volume is not sufficient to make one an expert in any of the materials characterization techniques (“a little knowledge is a dangerous thing”). Its purpose is to guide one in determining which techniques to be aware of and approach first in problem-solving. Further information to help solve a materials-based problem may be obtained from the references at the close of each chapter and from experts who use characterization techniques to solve problems. (Experts are employed in the materials characterization organizations of large companies and in independent analytical service laboratories.)

I would like to acknowledge the contributions of a number of people in the preparation of this volume. Dick Brundle, the Series editor has helped beyond the call of duty in many ways. He has been patient and persistent and he has assisted in much of the editing. Gary McGuire pitched in at a time when I was unavailable and proofread all the chapters in draft form, making suggestions for improvements. Penny Strausser, my wife, was helpful in every way possible—discussing ideas, proofreading, typing—and was forgiving of my time. Finally, I thank the authors of the individual chapters for being patient and for seeing this through.

Yale Strausser

Contributors

Roc Blumenthal Motorola, Inc. Austin, TX	Tungsten-Based Conductors
Roger Brennan Solecon Laboratories Sunnyvale, CA	Spreading Resistance Analysis (SRA)
M. Lawrence A. Dass Intel Corporation Santa Clara, CA	Barrier Films
David Dickey Solecon Laboratories Sunnyvale, CA	Spreading Resistance Analysis (SRA)
C. I. Drowley Motorola, Inc. Mesa, AZ	Application of Materials Characterization Techniques to Silicon Epitaxial Growth
David Fanger Intel Corporation Rio Rancho, NM	Aluminum- and Copper-Based Conductors
N. M. Johnson Xerox Research Center Palo Alto, CA	Deep Level Transient Spectroscopy (DLTS)
Walter Johnson Prometrics Corporation Santa Clara, CA	Sheet Resistance and the Four Point Probe
David C. Joy The University of Tennessee-Knoxville Knoxville, TN	Electron Beam Induced Current (EBIC) Microscopy
George N. Maracas Arizona State University Tempe, AZ	Capacitance–Voltage (C–V) Measurements; Hall Effect Resistivity Measurements
S. P. Murarka Rensselaer Polytechnic Institute Troy, NY	Silicides
Philipp Niedermann University of Geneva Geneva	Ballistic Electron Emission Microscopy (BEEM)
Jon Orloff University of Maryland Washington, DC	Focused Ion Beams (FIBs)

Gregory C. Smith
Texas Instruments
Dallas, TX

Yale Strausser^{*}
Digital Instruments
Santa Barbara, CA

Roger Tonneman
Intel Corporation
Rio Rancho, NM

Chuck Yarling
Prometrics Corporation
Santa Clara, CA

Tungsten-Based Conductors

Polysilicon Conductors

Aluminum- and Copper-Based Conductors

Sheet Resistance and the Four Point Probe

Contents

Preface to the Reissue of the Materials Characterization Series ix

Preface to Series x

Preface to the Reissue of *Characterization in Silicon Processing* xi

Preface xii

Contributors xiv

APPLICATION OF MATERIALS CHARACTERIZATION TECHNIQUES TO SILICON EPITAXIAL GROWTH

- 1.1 Introduction 1
- 1.2 Silicon Epitaxial Growth 2
 - Basic Chemical Reactions 2, Precleaning Considerations 3, Reactor Types 3
- 1.3 Film and Process Characterization 4
 - Crystal Quality 4, Preclean Quality 6, Thickness 9, Dopant Concentration and Dopant Profiling 12
- 1.4 Selective Growth 14
 - Basic Process Considerations 14, Defect Density and Growth Morphology 15, Preclean Quality 18, Thickness 18
- 1.5 $\text{Si}_{1-x}\text{Ge}_x$ Epitaxial Growth 18
 - Material Considerations 18, Reactor Types 19
- 1.6 $\text{Si}_{1-x}\text{Ge}_x$ Material Characterization 20
 - Composition and Thickness 20, Growth Morphology 22, Lattice Strain and Critical Thickness 23, Relaxation Kinetics 24, Bandgap Measurements 24, Interfacial Abruptness and Outdiffusion 25, Impurity Profiles 25
- 1.7 Summary 26

POLYSILICON CONDUCTORS

- 2.1 Introduction** 32
- 2.2 Deposition** 33
Surface Preparation 34, Nucleation and Growth 35, Postgrowth Analysis 38, High-Quality Polysilicon 42, Integrated Circuit Fabrication Issues 43
- 2.3 Doping** 45
Dopant Distribution 45, Deglaze 46, Ion Implantation Doping 46
- 2.4 Patterning** 47
Lithography 47, Etching 47
- 2.5 Subsequent Processing** 48
Polycides 48, Dielectric Encapsulation 49

SILICIDES

- 3.1 Introduction** 53
- 3.2 Formation of Silicides** 57
Sheet Resistance Measurements 57, Rutherford Backscattering Measurements 60, X-Ray Diffraction Measurements 72, Ellipsometric Measurements 74
- 3.3 The Silicide–Silicon Interface** 76
- 3.4 Oxidation of Silicides** 82
- 3.5 Dopant Redistribution During Silicide Formation** 84
- 3.6 Stress in Silicides** 87
- 3.7 Stability of Silicides** 90
- 3.8 Summary** 92

ALUMINUM- AND COPPER-BASED CONDUCTORS

- 4.1 Introduction** 96
History 96
- 4.2 Film Deposition** 98
Techniques 98, Problems with Deposition 101
- 4.3 Film Growth** 104
Substrate Surface Properties 104, Surface Preparation 107, Film Formation 108, Microstructure 110, Patterning and Etching 110
- 4.4 Encapsulation** 113
- 4.5 Reliability Concerns** 114

TUNGSTEN-BASED CONDUCTORS

- 5.1 Applications for ULSI Processing** 121
- 5.2 Deposition Principles** 122
- 5.3 Blanket Tungsten Deposition** 123
 - Film Thickness 123, Film Conformality 124, Film Resistivity 124,
 - Film Stress 125, Surface Roughness 126, Film Microstructure 127
- 5.4 Selective Tungsten Deposition** 127
 - Selectivity Breakdown 129, Substrate Interaction 131

BARRIER FILMS

- 6.1 Introduction** 138
- 6.2 Characteristics of Barrier Films** 139
- 6.3 Types of Barrier Films** 140
- 6.4 Processing Barrier Films** 140
 - Inert Sputtering 141, Reactive Sputtering 141, Chemical Vapor
 - Deposition 142, Nitridation and Rapid Thermal Annealing 143
- 6.5 Examples of Barrier Films** 143
 - Titanium Thin Films 144, Tungsten–Titanium Thin Films 149
 - Titanium Nitride 151
- 6.6 Summary** 163

APPENDIX: TECHNIQUE SUMMARIES

- 1 Auger Electron Spectroscopy (AES)** 169
- 2 Ballistic Electron Emission Microscopy (BEEM)** 170
- 3 Capacitance–Voltage (C–V) Measurements** 177
- 4 Deep Level Transient Spectroscopy (DLTS)** 179
- 5 Dynamic Secondary Ion Mass Spectrometry (Dynamic SIMS)** 181
- 6 Electron Beam Induced Current (EBIC) Microscopy** 182
- 7 Energy-Dispersive X-Ray Spectroscopy (EDS)** 188
- 8 Focused Ion Beams (FIBs)** 189
- 9 Fourier Transform Infrared Spectroscopy (FTIR)** 193
- 10 Hall Effect Resistivity Measurements** 194
- 11 Inductively Coupled Plasma Mass Spectrometry (ICPMS)** 196
- 12 Light Microscopy** 197

13	Low-Energy Electron Diffraction (LEED)	198
14	Neutron Activation Analysis (NAA)	199
15	Optical Scatterometry	200
16	Photoluminescence (PL)	201
17	Raman Spectroscopy	202
18	Reflection High-Energy Electron Diffraction (RHEED)	203
19	Rutherford Backscattering Spectrometry (RBS)	204
20	Scanning Electron Microscopy (SEM)	205
21	Scanning Transmission Electron Microscopy (STEM)	206
22	Scanning Tunneling Microscopy and Scanning Force Microscopy (STM and SFM)	207
23	Sheet Resistance and the Four Point Probe	208
24	Spreading Resistance Analysis (SRA)	217
25	Static Secondary Ion Mass Spectrometry (Static SIMS)	225
26	Surface Roughness: Measurement, Formation by Sputtering, Impact on Depth Profiling	226
27	Total Reflection X-Ray Fluorescence Analysis (TXRF)	227
28	Transmission Electron Microscopy (TEM)	228
29	Variable-Angle Spectroscopic Ellipsometry (VASE)	229
30	X-Ray Diffraction (XRD)	230
31	X-Ray Fluorescence (XRF)	231
32	X-Ray Photoelectron Spectroscopy (XPS)	232
	Index	233

Application of Materials Characterization Techniques to Silicon Epitaxial Growth

C. I. DROWLEY

Contents

- 1.1 Introduction
- 1.2 Silicon Epitaxial Growth
- 1.3 Film and Process Characterization
- 1.4 Selective Growth
- 1.5 $\text{Si}_{1-x}\text{Ge}_x$ Epitaxial Growth
- 1.6 $\text{Si}_{1-x}\text{Ge}_x$ Material Characterization
- 1.7 Summary

1.1 Introduction

Silicon epitaxial growth has emerged as a major process technology for VLSI circuit production during the last decade. Prior to that time, silicon epitaxial growth technology had been used primarily for bipolar IC, discrete device, and power device applications. The ability to reduce latchup in CMOS circuitry by growing a lightly doped epitaxial layer (for the active device region) over a heavily doped substrate (which provides a low-resistance shunt path for substrate currents, and thus suppresses turn-on of parasitic devices) has led to the adoption of epitaxy for high-volume CMOS processes.¹ Silicon epitaxial growth is also a critical process for the production of high-performance circuits incorporating both bipolar and CMOS devices (i.e., BiCMOS technology).²

Epitaxial growth applications have expanded to include the selective epitaxial growth of silicon on patterned substrates. Selective epitaxy has been demonstrated on a number of VLSI structures. Some examples of such selective growth applications are the creation of low-encroachment isolation,^{3, 4} elevated MOS source/drain formation,⁵ and DRAM cells.^{6, 7} Three-dimensional structures such as folded CMOS inverters have also been fabricated.⁸