

# **Metallographic and Materialographic Specimen Preparation, Light Microscopy, Image Analysis and Hardness Testing**

**Kay Geels**

**In collaboration with Daniel B. Fowler,  
Wolf-Ulrich Kopp, and Michael Rückert**



**INTERNATIONAL**

*Standards Worldwide*

ASTM International  
100 Barr Harbor Drive  
PO Box C700  
West Conshohocken, PA 19428-2959

Printed in U.S.A.  
ASTM Stock No. MNL46

**Library of Congress Cataloging-in-Publication Data**

Metallographic and materialographic specimen preparation, light microscopy, image analysis and hardness testing

Kay Geels; in collaboration with Daniel B. Fowler, Wolf-Ulrich Kopp, and Michael Rückert

p. cm.—(Manual; 46)

ASTM stock number: MNL 46.

Includes bibliographical references.

ISBN 978-0-8031-4265-7

E-book ISBN 978-0-8031-5691-3

1. Metallography. 2. Metallographic specimens. I. Title.

TN690.G3785 2006

669'.95028—dc22

2006103391

Copyright © 2007 ASTM International, West Conshohocken, PA. All rights reserved. This material may not be reproduced or copied, in whole or in part, in any printed, mechanical, electronic, film, or other distribution and storage media, without the written consent of the publisher.

**Photocopy Rights**

**Authorization to photocopy item for internal, personal, or educational classroom use, or the internal, personal, or educational classroom use of specific clients, is granted by ASTM International (ASTM) provided that the appropriate fee is paid to the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923; Tel: 978-750-8400; online: <http://www.copyright.com/>.**

The Society is not responsible, as a body, for the statements and opinions expressed in this publication.

ASTM International does not endorse any products represented in this publication.

Printed in City, State  
Month Year

## Preface

This book is written both for the experienced and unexperienced metallographer (materialographer) who wants specific advice and information. It is also for persons seeking a broader knowledge of metallographic/materialographic specimen preparation and the examination methods, light microscopy, image analysis, and hardness testing. Special emphasis has been made on relations between ASTM standards and metallography/materialography.

The book will be useful for students in courses devoted to practical metallography and materialography.

The scope of the book is to give relevant information, in an efficient and clear way, covering the daily work in a metallographic/materialographic laboratory.

### Metallographic/Materialographic Preparation

*Kay Geels and Michael Rückert (Sections 13.5/6)*

Part I is a description of sectioning, mounting, grinding, polishing, and etching of specimens for examination in reflected light, enabling the reader to understand the mechanisms of the entire preparation process. This is combined with practical advice on specimen preparation and an introduction to existing equipment and consumables.

Part II is a “Hands-on” Manual guiding the metallographer/materialographer to the correct preparation method, based on the material to be prepared and the purpose of examination. More than 150 methods are indicated covering practically all types of materials, describing the preparation process from sectioning to etching. This part also includes a section on Trouble Shooting, covering all stages in the preparation process and artifacts developed during the preparation.

### Light Microscopy

*Wolf-Ulrich Kopp*

Part III is a description of the optical reflected-light microscope with photomicroscopy giving the reader both an introduction to the subject and a manual for the daily work. Also, a short introduction to electron microscopy and scanning probe microscopy can be found in this part of the book.

### Quantitative Metallography/Materialography—Automatic Image Analysis

*Daniel B. Fowler*

Part IV gives an introduction to quantitative microstructural analysis and automatic image analysis, both theoretically and practically, with emphasis on the examinations based on ASTM standards and other types of commonly used analyses.

## Hardness Testing

*Wolf-Ulrich Kopp*

Part V gives a description of the hardness testing methods, Brinell, Vickers, Rockwell, microhardness and instrumented (nano) indentation testing based on ASTM standards, both theoretically and as a practical guide.

## The Metallographic/Materialographic Laboratory

*Kay Geels*

Part VI gives directions on how to establish and maintain a modern metallographic/materialographic laboratory. The important rules and regulations covering occupational safety are described and commented on.

The authors of this book, representing more than 100 years' experience with practical metallography and materialography, have tried to make this book a practical tool and helpful source of information to all who are involved in the noble art/science of metallography/materialography—Kay Geels.

## Acknowledgments

The authors wish to acknowledge the four reviewers, who brought forward valuable insight for improvement. Special thanks to R. C. Nester, for his advice and suggestions on extension and shortening of the chapters. Thanks to G. Petzow, F. Mücklich and L. E. Samuels for permission to use a number of illustrations, and to B. Ottesen and W. Taylor for reading the manuscript and giving good advice. A special acknowledgement goes to fellow-metallographers/materialographers for support and advice through the years and directly connected to the book. The list includes U. Täffner, S. Glancy, E. Weidmann, A. Z. Jensen and A. Guesnier. A special thanks to L. Bjerregaard for her very important advice on many of the preparation methods, and to H. Hellestad for her invaluable support in making the illustrations. Also, thanks go to W. Taylor and Struers GmbH for providing important micrographs. The authors acknowledge the following companies for supply of information and illustrations, Buehler Ltd., Carl Zeiss AG, DoAll Company, Emco-Test GmbH, Leica Microsystems AG, Olympus Optical Co. Inc., and Struers A/S. Particular thanks to G. E. Totten and K. Dernoga at ASTM International for establishing and maintaining the project of making this book. Last but not least, thanks to B. Freiberg and J. Hestehave for support and encouragement during the years of making the book.

## Abbreviations

AFM	Atomic Force Microscope
BF	Bright Field
CBN	Cubic Boron Nitride
DF	Dark Field
DIC	Differential Interference Contrast

EBSD	Electron Backscatter Diffraction
EDS	Energy Dispersive Spectroscopy
EPMA	Electron Probe Microanalyzer
FIB	Focused Ion Beam
MFM	Magnetic Force Microscope
PCB	Printed Circuit Board
POL	Polarized Light
SEM	Scanning Electron Microscope
SPM	Scanning Probe Microscope
STM	Scanning Tunnel Microscope
STEM	Scanning Transmission Electron Microscope
TEM	Transmission Electron Microscope

# Contents

## Part I: The Metallographic/Materialographic Preparation Process

<b>1</b>	<b>Introduction</b>	
1.1	Metallographic/Materialographic Preparation—The True Microstructure. . . . .	5
1.1.1	Henry Clifton Sorby (1826–1908). . . . .	5
1.2	The True Microstructure. . . . .	6
1.3	Selection of Preparation Method. . . . .	6
1.3.1	Artifacts. . . . .	7
1.3.2	Preparation Methods. . . . .	7
1.4	The Metallographic/Materialographic Specimen. . . . .	7
1.4.1	“Specimen” or “Sample”. . . . .	8
1.5	The Preparation Process. . . . .	9
1.5.1	Sectioning. . . . .	10
1.5.2	Mounting. . . . .	11
1.5.3	Preparation of the Surface. . . . .	11
1.5.4	Etching. . . . .	13
<b>2</b>	<b>Sectioning</b>	
2.1	Selection. . . . .	14
2.1.1	General Studies or Routine Work. . . . .	14
2.1.2	Study of Failures. . . . .	14
2.1.3	Research Studies. . . . .	14
2.1.4	Type of Section. . . . .	14
2.1.5	Reporting of Locations. . . . .	15
2.2	Sectioning. . . . .	15
2.3	Wet Abrasive Cutting. . . . .	15
2.3.1	The Cut-off Grinding Process. . . . .	15
2.3.2	The Cut-off Wheel—Abrasives and Bond Materials. . . . .	16
2.3.3	Grinding Mechanics. . . . .	21
2.3.4	Mechanical Damage. . . . .	22
2.3.5	Thermal Damage. . . . .	23
2.3.6	Cut-off Wheel Wear. . . . .	25
2.3.7	Cutting Fluids. . . . .	26
2.3.8	The Metallographic/Materialographic Cutting Operation. . . . .	29
2.4	Abrasive Cut-Off Wheels. . . . .	32
2.4.1	Consumable Wheels. . . . .	32
2.4.2	Slow Consumable Wheels. . . . .	34
2.5	Abrasive Cut-off Machines. . . . .	36
2.5.1	Design Principles of Wheel—Work Piece Contact. . . . .	36
2.5.2	Machine Designs. . . . .	39
2.6	Advice and Hints on Wet Abrasive Cutting. . . . .	43
2.6.1	Cut-off Wheel Selection. . . . .	44
2.7	Other Sectioning Methods. . . . .	45
2.7.1	Fracturing. . . . .	45
2.7.2	Sectioning by Melting. . . . .	46
2.7.3	Shearing. . . . .	46
2.7.4	Sawing—Table 2.1. . . . .	47
2.7.5	Wire Cutting. . . . .	52
<b>3</b>	<b>Mounting</b>	
3.1	Purpose and Criteria. . . . .	54
3.1.1	Purpose. . . . .	54
3.1.2	Criteria for a Good Mount. . . . .	54

3.1.3	Surface Flatness—Edge Retention. . . . .	54
3.2	Mounting Methods. . . . .	57
3.2.1	Clamping. . . . .	57
3.2.2	Hot Compression Mounting. . . . .	58
3.2.3	Cold (Castable) Mounting. . . . .	58
3.3	Hot Compression Mounting. . . . .	58
3.3.1	Advantages of Hot Compression Mounting. . . . .	59
3.3.2	Disadvantages of Hot Compression Mounting. . . . .	59
3.3.3	MSDS (Material Safety Data Sheets). . . . .	59
3.4	Hot Mounting Resins. . . . .	60
3.4.1	Thermoplastic Resins. . . . .	60
3.4.2	Thermosetting Resins. . . . .	61
3.5	Mounting Presses. . . . .	62
3.5.1	The Heating/Cooling Unit. . . . .	62
3.5.2	The Hydraulic Press. . . . .	63
3.5.3	The Air-operated Press. . . . .	65
3.6	Advice and Hints on Hot Compression Mounting. . . . .	65
3.6.1	Selection of Resins for Hot Compression Mounting. . . . .	66
3.7	Cold (Castable) Mounting. . . . .	67
3.7.1	Advantages of Cold (Castable) Mounting. . . . .	68
3.7.2	Disadvantages of Cold (Castable) Mounting. . . . .	68
3.7.3	MSDS (Material Safety Data Sheets). . . . .	68
3.8	Cold Mounting Resins. . . . .	68
3.8.1	Acrylics. . . . .	68
3.8.2	Polyesters. . . . .	69
3.8.3	Epoxies. . . . .	69
3.9	Accessories for Cold (Castable) Mounting. . . . .	70
3.9.1	Mounting Molds. . . . .	70
3.9.2	Clips. . . . .	71
3.10	Vacuum Impregnation. . . . .	71
3.10.1	Dyes. . . . .	72
3.11	Special Mounting Techniques. . . . .	72
3.11.1	Taper Sectioning. . . . .	73
3.11.2	Edge Protection. . . . .	74
3.11.3	Mounting of Very Small Parts, Foils, and Wires. . . . .	75
3.11.4	Mounting of Powders. . . . .	76
3.11.5	Mounting of PCB Coupons. . . . .	76
3.11.6	Conductive Mounts. . . . .	77
3.12	Recovery of Mounted Specimen. . . . .	77
3.13	Advice and Hints on Cold Mounting. . . . .	78
3.13.1	Selection of Cold Mounting Materials. . . . .	79
<b>4</b>	<b>Marking—Storage—Preservation</b>	
4.1	Marking. . . . .	80
4.1.1	Marking with Waterproof Ink. . . . .	80
4.1.2	Identification Tag. . . . .	80
4.1.3	Engraving. . . . .	80
4.1.4	Stamping. . . . .	80
4.2	Storage. . . . .	81
4.3	Preservation. . . . .	81

<b>5</b>	<b>Cleaning and Cleanliness</b>	
5.1	Cleaning.....	82
5.1.1	Cleaning Before Start of Preparation.....	82
5.1.2	Cleaning During and After Preparation.....	82
5.2	Cleanliness.....	84
<b>6</b>	<b>Mechanical Surface Preparation—Grinding</b>	
6.1	Grinding—A Basic Process.....	85
6.1.1	Plane Grinding (PG).....	85
6.1.2	Fine Grinding.....	86
6.2	Material Removal.....	86
6.2.1	Rake Angle.....	87
6.2.2	Grain Shape—Contacting Points.....	88
6.2.3	Grain Penetration.....	89
6.2.4	Force on Specimens.....	89
6.2.5	Grinding/Polishing Fluids.....	89
6.3	Deformation.....	89
6.3.1	Metals.....	89
6.3.2	Brittle Materials—Ceramics.....	92
6.4	Grinding Abrasives.....	93
6.4.1	Aluminum Oxide.....	93
6.4.2	Silicon Carbide.....	93
6.4.3	Diamond—Diamond Products.....	94
6.4.4	Cubic Boron Nitride (CBN).....	97
6.4.5	Boron Carbide.....	97
6.4.6	Hardness of Abrasives and Materials—Table 6.1.....	97
6.5	Grinding/Polishing Fluids—Lubricants.....	97
6.5.1	Water-Based Lubricant.....	97
6.5.2	Alcohol-Based Lubricant.....	97
6.5.3	Water-oil Based Lubricant.....	98
6.5.4	Oil-Based Lubricant.....	98
6.6	Traditional Grinding.....	99
6.6.1	Grinding Stones/Disks.....	99
6.6.2	SiC Wet Grinding Paper—Table 6.2.....	100
6.6.3	Alumina—Zirconia Alumina Wet Grinding Paper.....	105
6.7	Contemporary Grinding.....	106
6.7.1	Magnetic Fixation.....	106
6.7.2	Resin-Bonded Diamond Grinding Disks.....	107
6.7.3	Resin-Bonded SiC Grinding Disks.....	108
6.7.4	Metal-Bonded Diamond-Coated Disks.....	109
6.7.5	Diamond Pads.....	109
6.7.6	Diamond/CBN/ Al <sub>2</sub> O <sub>3</sub> /SiC Film.....	109
6.7.7	Rigid Composite Disks.....	109
6.7.8	Fine Grinding Cloths.....	116
6.8	Grinding/Polishing Equipment.....	117
6.8.1	Plane Grinding.....	117
6.8.2	Fine Grinding.....	119
<b>7</b>	<b>Mechanical Surface Preparation—Polishing</b>	
7.1	Polishing: Producing the True Structure.....	120
7.1.1	Rough Polishing.....	120
7.1.2	Polishing.....	120



7.2	Material Removal.....	120
7.2.1	Influence of Polishing Abrasive on Removal Rate.....	121
7.2.2	Force on Specimens.....	121
7.3	Deformation.....	122
7.3.1	The Beilby Layer.....	122
7.3.2	Influence of Polishing Abrasive, Cloth, and Fluid on Deformation... ..	123
7.4	Polishing Cloths.....	124
7.4.1	Edge Retention—Relief.....	126
7.4.2	Cloths for Fine Grinding and Rough Polishing.....	126
7.4.3	Cloths for Polishing.....	127
7.5	Polishing Abrasives.....	129
7.5.1	Diamond Suspensions.....	129
7.5.2	Diamond Spray.....	129
7.5.3	Diamond Paste.....	130
7.5.4	Alumina.....	130
7.5.5	Silica.....	131
7.5.6	Other Oxides.....	132
7.6	Polishing Lubricants.....	132
7.7	The Metallographic/Materialographic Preparation Methods— Method Parameters.....	132
7.7.1	RPM of Grinding/Polishing Disk.....	133
7.7.2	RPM of Specimen Holder.....	133
7.7.3	Direction of Specimen Holder.....	134
7.7.4	Force on Specimens.....	134
7.7.5	Process Time.....	134
7.7.6	Stock Removal.....	134
7.8	Grinding/Polishing Equipment—Manual Preparation.....	135
7.9	Grinding/Polishing Equipment—Automatic Preparation.....	135
7.9.1	Machine Design.....	135
7.9.2	Polishing Dynamics.....	139
7.9.3	Semiautomatic and Fully Automatic Systems.....	140
7.10	Special Preparation Techniques.....	143
7.10.1	PCB Coupons.....	143
7.10.2	Microelectronic Materials—Nonencapsulated Cross Sections.....	143
7.10.3	Microelectronic Packages—Table 7.2—Target Preparation.....	147
7.10.4	EBSD.....	149
7.11	Field Metallography/Materialography—Nondestructive Mechanical Preparation.....	150
7.11.1	Portable Grinder/Polishers.....	150
7.11.2	Replication.....	150
7.12	Chemical Mechanical Polishing.....	151
7.12.1	Protection—Corrosion at CMP.....	152
7.13	Thin Sections.....	152
7.13.1	Thin Sections of Petrographic/Ceramic Materials.....	152
7.13.2	Thin Sections of Plastics/Polymers.....	153
7.14	Microtomy—Ultramilling.....	155
<b>8</b>	<b>Electrolytic Polishing/Etching</b>	
8.1	The Electrolytic Polishing/Etching Process.....	156
8.1.1	The Polishing Cell.....	157
8.1.2	Smoothing and Brightening.....	157

8.1.3	Electrolytic Etching. . . . .	159
8.1.4	Advantages and Disadvantages. . . . .	160
8.2	Electrolytes. . . . .	163
8.3	Electropolishing in Practice. . . . .	164
8.3.1	Factors Influencing Electrolytic Polishing. . . . .	164
8.3.2	Example of Electrolytic Polishing/Etching. . . . .	165
8.4	Electrolytic Polishing Equipment. . . . .	165
8.4.1	Electropolishers for Laboratory Use. . . . .	165
8.5	Field Metallography—Nondestructive Electropolishing. . . . .	166
8.6	Electrolytic Thinning for TEM. . . . .	167
8.7	Chemical Polishing. . . . .	168
<b>9</b>	<b>Etching</b>	
9.1	Microetching—Contrast. . . . .	169
9.2	Contrast Without Surface Modifications—Microscope Techniques. . . . .	169
9.2.1	Dark-Field Illumination (DF). . . . .	169
9.2.2	Differential Interference Contrast (DIC). . . . .	169
9.2.3	Polarized Light (POL). . . . .	169
9.2.4	Fluorescence. . . . .	170
9.3	Contrast with Surface Modification—Etching. . . . .	170
9.3.1	Grain Contrast Etching. . . . .	170
9.3.2	Grain Boundary Etching. . . . .	171
9.3.3	Reproducibility. . . . .	171
9.3.4	Safety Precautions. . . . .	172
9.4	Classical Etching. . . . .	172
9.4.1	Chemical Etching. . . . .	172
9.4.2	Precipitation (Color) Etching. . . . .	172
9.4.3	Heat Tinting. . . . .	172
9.5	Electrolytic Etching. . . . .	172
9.5.1	Anodic Etching. . . . .	172
9.5.2	Anodizing. . . . .	173
9.5.3	Potentiostatic Etching. . . . .	173
9.6	Physical Etching. . . . .	173
9.6.1	Relief Polishing. . . . .	173
9.6.2	Ion Etching. . . . .	173
9.6.3	Thermal Etching. . . . .	174
9.6.4	Vapor Deposition. . . . .	174
9.6.5	Sputtering. . . . .	174
9.7	Macroetching. . . . .	174
<b>Part II: Metallographic/Materialographic Specimen Preparation—A Hands-On Manual</b>		
<b>10</b>	<b>Introduction</b>	
10.1	Specimen Material. . . . .	179
10.2	Purpose of Examination. . . . .	179
10.3	Specimen Preparation. . . . .	179
<b>11</b>	<b>Specimen Material—Table 11.1</b>	
11.1	Classification of Materials. . . . .	181
11.2	How to Use Table 11.1. . . . .	181
11.3	Table 11.1—Materials/Methods. . . . .	182

**xii      Metallographic and Materialographic Specimen Preparation**

<b>12</b>	<b>Purpose of Examination</b>	
12.1	Purpose in General.....	188
12.2	Purpose: ASTM Standards.....	188
12.3	Table 12.1: Purpose/ASTM Standards.....	188
12.4	ASTM Standards—Metallography.....	188
12.4.1	Introduction.....	188
12.4.2	ASTM Standards in this Book.....	190
12.4.3	ASTM Standards—Document Summaries.....	193
12.5	Chemical Microetching—Table 12.2—Table 12.3.....	194
12.5.1	Etching Practice.....	194
12.5.2	Table 12.2—Numerical List of Etchants.....	195
12.5.3	Table 12.3—Etchant Names.....	217
<b>13</b>	<b>Specimen Preparation</b>	
13.1	Introduction.....	218
13.2	Mechanical Preparation—The “Traditional” and “Contemporary” Methods.....	218
13.2.1	Material/Preparation Tables.....	218
13.2.2	Method Tables—Generic Methods—Parameters/Consumables— Table 13.1.....	219
13.2.3	Material/Preparation Tables—Methods C-01/T-01 to C-68/T-68.....	222
13.2.4	Manual Preparation.....	450
13.3	Electrolytic Polishing and Etching.....	453
13.3.1	Electropolishers.....	454
13.3.2	Electrolytes—Methods for Electropolishing—Table 13.2.....	454
13.3.3	Table 13.2—Electrolytes for Electropolishing/Etching.....	454
13.3.4	Mechanical Preparation for Electropolishing.....	456
13.3.5	Electropolishing—Method Tables.....	456
13.3.6	Electropolishing—Methods EI-01 To EI-25.....	456
13.4	Field Metallography/Materialography—Nondestructive Preparation.....	475
13.4.1	Mechanical Preparation.....	475
13.4.2	Electrolytic Polishing.....	475
13.4.3	Replication.....	475
13.5	Trouble Shooting—How to Improve Preparation Results.....	476
13.5.1	Sectioning.....	477
13.5.2	Mounting.....	479
13.5.3	Mechanical Preparation.....	482
13.5.4	Electrolytic Polishing.....	483
13.5.5	General Rules—“The Metallographer’s Rule of Thumb”.....	483
13.6	Trouble Shooting—How to Overcome Preparation Artifacts.....	484
13.6.1	Preparation Artifacts—Flow Charts.....	484
13.6.2	Sectioning—General Problems—Flow Charts.....	485
13.6.3	Mounting—General Problems—Artifacts.....	495
13.6.4	Grinding and Mechanical Polishing—Flow Charts.....	498
13.6.5	Electropolishing—General Problems—Artifacts.....	521
<b>Part III: Light Microscopy</b>		
<b>14</b>	<b>Introduction</b>	
14.1	Visible Light—Table 14.1—Table 14.2.....	525
14.2	The Human Eye.....	526

14.3	Magnifying Lens and Microscope.....	527
14.4	Magnification.....	527
<b>15</b>	<b>The Optical Reflected Light Microscope</b>	
15.1	The Path of Light Rays.....	528
15.2	The Objective.....	528
15.2.1	Numerical Aperture—Resolution-Magnification—Table 15.1—Table 15.2.....	528
15.2.2	Aberrations in Image-Formation.....	532
15.2.3	Available Objectives.....	533
15.3	Eyepieces.....	535
15.4	Illumination.....	536
15.4.1	Koehler's Illumination System.....	536
15.5	Microscope Options.....	537
15.6	The Reflected-Light Microscope.....	538
15.6.1	Upright Type of Reflected-Light Microscope.....	538
15.6.2	Inverted Type of Reflected-Light Microscope.....	538
15.7	Optical Examination Methods.....	540
15.7.1	Bright-Field (BF) Illumination.....	541
15.7.2	Dark-Field (DF) Illumination.....	541
15.7.3	Polarization Contrasting (POL).....	542
15.7.4	Differential Interference Contrasting (DIC).....	544
15.7.5	Fluorescence in Reflected Light.....	545
15.8	Practical Use of the Microscope.....	546
15.8.1	Setting up the Microscope.....	546
15.8.2	Working with the Microscope.....	547
15.8.3	Correct Adjustment of the Microscope.....	548
15.8.4	Focusing and Practical Use.....	548
15.8.5	Measurements of Length.....	549
15.8.6	Measurements of Height Differences.....	550
15.8.7	Maintenance of the Microscope.....	550
15.9	Documentation.....	550
15.10	The Confocal Laser Scan Microscope.....	552
15.10.1	Function of Confocal Laser Scan Microscope.....	552
15.10.2	Applications of Confocal Laser Scan Microscope.....	554
15.11	Stereo Microscopy.....	555
<b>16</b>	<b>Electron Microscopy—Scanning Probe Microscopy</b>	
16.1	The Transmission Electron Microscope (TEM).....	558
16.1.1	The Scanning Transmission Electron Microscope (STEM).....	558
16.2	The Scanning Electron Microscope (SEM).....	558
16.2.1	Energy Dispersive Spectroscopy (EDS).....	559
16.2.2	Electron Backscatter Diffraction (EBSD).....	559
16.2.3	The Electron Probe Microanalyzer (EPMA).....	560
16.3	Focused Ion Beam (FIB).....	560
16.4	Scanning Probe Microscopes (SPM).....	560
<b>Part IV: Quantitative Metallography/Materialography— Automatic Image Analysis</b>		
<b>17</b>	<b>Quantitative Metallography/Materialography—An Introduction</b>	
17.1	Quantitative Metallography/Materialography.....	565
17.1.1	Stereology—Table 17.1.....	565

**xiv      Metallographic and Materialographic Specimen Preparation**

17.1.2	Specimen Preparation.....	567
17.1.3	Calibration.....	568
17.1.4	Field Selection—Bias.....	568
17.2	Volume Fraction—Point Count.....	569
17.2.1	ASTM Test Method for Determining Volume Fraction by Systematic Manual Point Count (E 562).....	569
17.3	Inclusion Rating.....	570
17.3.1	ASTM Standard Test Method For Determining the Inclusion Content of Steel (E 45).....	570
17.3.2	ASTM Practice for Obtaining JK Inclusion Ratings Using Automatic Image Analysis (E 1122) (withdrawn 2006, replaced by E 45).....	570
17.3.3	ASTM Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis (E 1245).....	570
17.4	Grain Size.....	571
17.4.1	ASTM Test Methods for Determining Average Grain Size (E 112)...	571
17.4.2	ASTM Test Methods for Estimating the Largest Grain Observed in a Metallographic Section (ALA Grain Size) (E 930).....	573
17.4.3	ASTM Test Methods for Characterizing Duplex Grain Sizes (E 1181).....	573
17.4.4	ASTM Test Methods for Determining Average Grain Size Using Semiautomatic and Automatic Image Analysis (E 1382).....	573
17.5	Banding.....	574
17.5.1	ASTM Practice for Assessing the Degree of Banding or Orientation of Microstructures (E 1268).....	574
17.6	Porosity in Thermal Spray Coatings.....	574
17.6.1	ASTM Test Methods for Determining Area Percentage Porosity in Thermal Sprayed Coatings (E 2109).....	574
17.7	Decarburization—Case Depth—Coatings.....	575
17.7.1	Specimen Preparation.....	575
17.7.2	ASTM Test Methods for Estimating the Depth of Decarburization of Steel Specimens (E 1077).....	575
17.7.3	Case Depth.....	575
17.7.4	ASTM Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section (B 487)..	576
17.7.5	ASTM Test Methods for Thickness of Diffusion Coating (C 664).....	576
17.8	Other ASTM Standards for Quantitative Materialography.....	576
<b>18</b>	<b>Automatic Image Analysis</b>	
18.1	Introduction.....	577
18.2	Qualitative and Quantitative Metallography/Materialography.....	577
18.2.1	The Transition to Quantitative Standards.....	577
18.2.2	Structure, Stereology, and Statistics.....	578
18.3	Principles of Digital Imaging.....	579
18.3.1	What is Digital Image Analysis?.....	579
18.3.2	Image Acquisition.....	579
18.3.3	Image Digitization—Gray Scale.....	580
18.3.4	The Histogram.....	581
18.3.5	The Effects of Brightness and Contrast on Illumination Distribution.....	581
18.3.6	Image Processing and True Microstructure.....	586
18.3.7	Image Calibration.....	595

18.4	Image Measurement. . . . .	598
18.4.1	Manual Measurements (Operator Defines Points, Lines, or Areas)...	599
18.4.2	Automatic Measurements (Objects Defined by Image Segmentation). . . . .	600
18.5	Digital Imaging Applied to Quantitative Materialography. . . . .	602
18.5.1	Percent Area (Volume Fraction). . . . .	602
18.5.2	Inclusion Rating. . . . .	603
18.5.3	Grain Size. . . . .	606
18.5.4	Degree of Banding. . . . .	608
18.5.5	Depth or Thickness Measurements. . . . .	608
18.5.6	Graphite in Iron Castings. . . . .	610
18.6	Digital Imaging Technology. . . . .	613
18.6.1	Hardware. . . . .	613
18.6.2	Software. . . . .	616
18.7	Digital Imaging System Implementation. . . . .	617
<b>19</b>	<b>Digital Image Management (Archiving)</b>	
<b>Part V: Hardness Testing</b>		
<b>20</b>	<b>Introduction</b>	
20.1	Indentation Hardness. . . . .	623
20.2	ASTM Standards. . . . .	625
<b>21</b>	<b>Static Hardness Testing Procedures</b>	
21.1	Brinell Hardness Testing. . . . .	626
21.1.1	Calculations and Procedures. . . . .	626
21.1.2	Brinell Hardness Testers. . . . .	628
21.2	Vickers Hardness Testers. . . . .	628
21.2.1	Calculations and Procedures. . . . .	628
21.2.2	Vickers Hardness Tester. . . . .	632
21.3	Knoop Hardness Testing. . . . .	633
21.3.1	Calculations and Procedures. . . . .	633
21.4	Rockwell Hardness Testing. . . . .	634
21.4.1	Calculations and Procedures. . . . .	634
21.4.2	Rockwell Hardness Testers. . . . .	636
21.5	Microindentation Hardness. . . . .	636
21.5.1	Methods. . . . .	636
21.5.2	Specimen Preparation. . . . .	637
21.5.3	Taking the Measurements. . . . .	638
21.5.4	Microindentation Hardness Testers. . . . .	639
21.5.5	Examples of Indentations. . . . .	639
21.6	Universal Hardness—Martens Hardness—Instrumented Indentation Testing—Nano Indentation. . . . .	639
21.6.1	Instrumented Indentation Testing—Nano Indentation. . . . .	641
21.7	Precision of Hardness Values. . . . .	642
21.8	Conversion of Hardness Values. . . . .	642
<b>22</b>	<b>Dynamic Hardness Testing Procedures</b>	
<b>23</b>	<b>Special Methods for Hardness Testing</b>	

**Part VI: The Metallographic/Materialographic Laboratory**

<b>24</b>	<b>Introduction</b>	
24.1	Establishing a Metallographic/Materialographic Laboratory. . . . .	649
24.2	Running a Metallographic/Materialographic Laboratory. . . . .	649
24.3	Occupational Safety and Health. . . . .	649
<b>25</b>	<b>How to Build a Metallographic/Materialographic Laboratory</b>	
25.1	Purpose. . . . .	650
25.1.1	Quality Control (QC). . . . .	650
25.1.2	Research and Education. . . . .	651
25.1.3	Testing and Inspection Laboratories—Failure Analysis. . . . .	651
25.2	Rationalization and Automation. . . . .	651
25.2.1	Reproducibility—Standards—Occupational Safety. . . . .	652
25.2.2	Productivity—Cost Per Specimen. . . . .	653
25.3	Planning the Metallographic/Materialographic Laboratory. . . . .	654
25.3.1	Basic Planning. . . . .	654
25.3.2	Detailed Planning. . . . .	655
25.4	Equipment and Laboratory Layout. . . . .	656
25.4.1	Equipment—Table 25.1. . . . .	656
25.4.2	Layout—Furniture—Installations. . . . .	660
25.5	Maintenance. . . . .	662
25.5.1	Organizing. . . . .	662
25.5.2	Cleaning. . . . .	662
25.5.3	Servicing. . . . .	663
<b>26</b>	<b>Occupational Safety and Health in the Metallographic/ Materialographic Laboratory</b>	
26.1	Dangers in the Metallographic/Materialographic Laboratory. . . . .	664
26.1.1	Sectioning. . . . .	664
26.1.2	Mounting. . . . .	664
26.1.3	Mechanical Preparation. . . . .	665
26.1.4	Electrolytic Polishing/Etching. . . . .	665
26.1.5	Etching—Etchants—Electrolytes. . . . .	665
26.1.6	Dust. . . . .	667
26.1.7	Cold (Castable) Mounting Resins. . . . .	667
26.1.8	Standard Guide on Metallographic Laboratory Safety (E 2014). . . . .	668
26.2	Safety Information. . . . .	668
26.2.1	Identification. . . . .	668
26.2.2	Material Safety Data Sheet (MSDS). . . . .	670
26.2.3	Standard Operating Procedure (SOP). . . . .	672
26.2.4	Job Safety Analysis (JSA). . . . .	672
26.3	Disposal of Chemicals. . . . .	672
26.4	Occupational Safety in General. . . . .	673
26.4.1	Standards. . . . .	673
26.4.2	Training. . . . .	673
26.4.3	Maintenance and Service. . . . .	673
26.5	Standards and Regulations—Organizations. . . . .	673
26.5.1	Designations and Abbreviations Used to Describe a Chemical Substance. . . . .	673
26.5.2	ASTM Standard. . . . .	674
26.5.3	OSHA—OSHA Standards. . . . .	674

26.5.4	National Institute for Occupational Safety and Health (NIOSH) . . . .	681
26.5.5	International Chemical Safety Cards (ICSCS) . . . . .	682
26.5.6	Environmental Protection Agency (EPA) . . . . .	683
26.5.7	National Technical Information Service (NTIS) . . . . .	683
26.5.8	American Conference of Government Industrial Hygienists (ACGIH) . . . . .	683
26.5.9	National Toxicology Program (NTP) . . . . .	683
26.5.10	Agency for Toxic Substance and Disease Registry (ATSDR) . . . . .	683
26.5.11	National Fire Protection Association (NFPA) . . . . .	684
26.5.12	National Paint and Coatings Association (NPCA)—HMIS . . . . .	684
26.5.13	BSI—ISO . . . . .	684
26.5.14	EU . . . . .	684
26.6	Literature on Laboratory Safety . . . . .	684
<b>27</b>	<b>Literature</b>	
27.1	Books . . . . .	685
27.2	Periodicals . . . . .	686
<b>Appendixes</b>		
<b>Appendix I: Other Standards on Metallography/Materialography . . . . .</b>		<b>686</b>
<b>Appendix II: Other Standards on Hardness Testing . . . . .</b>		<b>691</b>
<b>Appendix III: Hardness Conversion Tables for Metals (E140) . . . . .</b>		<b>694</b>
<b>Appendix IV: SI Quick Reference Guide: International System of Units (SI) . . . .</b>		<b>694</b>
<b>Glossary . . . . .</b>		<b>695</b>
<b>Index . . . . .</b>		<b>727</b>