

METALLOGRAPHY
PRINCIPLES AND PRACTICE

METALLOGRAPHY

Principles and Practice

George F. Vander Voort

*Supervisor
Applied Physics R & D
Carpenter Technology Corporation*

McGraw-Hill Book Company

New York St. Louis San Francisco Auckland Bogotá Hamburg
Johannesburg London Madrid Mexico Montreal New Delhi
Panama Paris São Paulo Singapore Sydney Tokyo Toronto

This book was set in Times Roman by Jay's Publishers Services, Inc.
The editors were Anne Murphy and Susan Hazlett;
the production supervisor was Leroy A. Young.
The drawings were done by Wellington Studios Ltd.
R. R. Donnelley & Sons Company was printer and binder.

METALLOGRAPHY
Principles and Practice

Copyright © 1984 by McGraw-Hill, Inc. All rights reserved. Printed in the United States of America. Except as permitted under the United States Copyright Act of 1976, no part of this publication may be reproduced or distributed in any form or by any means, or stored in a data base or retrieval system, without the prior written permission of the publisher.

1234567890 DOCDOC 8987654

ISBN 0-07-066970-8

Library of Congress Cataloging in Publication Data

Vander Voort, George F.

Metallography, principles and practice.

(McGraw-Hill series in materials science and engineering)

Includes bibliographical references and indexes.

1. Metallography. I. Title. II. Series.

TN690.V36 1984 669'.95 83-22272

ISBN 0-07-066970-8

McGraw-Hill Series in Materials Science and Engineering

Editorial Board

Michael B. Bever
Stephen M. Copley
M. E. Shank
Charles A. Wert
Garth L. Wilkes

Blatt: *Physics of Electronic Conduction in Solids*
Brick, Pense, and Gordon: *Structure and Properties of Engineering Materials*
Buerger: *Contemporary Crystallography*
Buerger: *Introduction to Crystal Geometry*
Darken and Gurry: *Physical Chemistry of Metals*
Dieter: *Engineering Design: A Materials and Processing Approach*
Dieter: *Mechanical Metallurgy*
Drauglis, Gretz, and Jaffee: *Molecular Processes on Solid Surfaces*
Elliott: *Constitution of Binary Alloys, First Supplement*
Flemings: *Solidification Processing*
Fontana and Greene: *Corrosion Engineering*
Gaskell: *Introduction to Metallurgical Thermodynamics*
Gordon: *Principles of Phase Diagrams in Materials Systems*
Guy: *Introduction to Materials Science*
Hansen: *Constitution of Binary Alloys*
Kanninen, Adler, Rosenfield, and Jaffee: *Inelastic Behavior of Solids*
Kehl: *The Principles of Metallographic Laboratory Practice*
Leslie: *The Physical Metallurgy of Steels*
Mills, Ascher, and Jaffee: *Critical Phenomena in Alloys, Magnets, and Superconductors*
Murr: *Electron Optical Applications in Materials Science*
Rhines: *Phase Diagrams in Metallurgy: Their Development and Application*
Rosenfield, Hahn, Bement, and Jaffee: *Dislocation Dynamics*
Rosenqvist: *Principles of Extractive Metallurgy*
Rozenfeld: *Corrosion Inhibitors*
Rudman, Stringer, and Jaffee: *Phase Stability in Metals and Alloys*
Shewmon: *Diffusion in Solids*
Shewmon: *Transformations in Metals*
Shunk: *Constitution of Binary Alloys, Second Supplement*
Smith: *Structure and Properties of Engineering Alloys*
Vander Voort: *Metallography: Principles and Practice*
Wert and Thomson: *Physics of Solids*

PREFACE

Metallography has proved to be an exceptionally useful metallurgical tool for both production and research work. Since the initial work of Sorby nearly 120 years ago, a multitude of techniques have been developed and applied to nearly every conceivable material. The vast scope of material available on this subject presents a formidable challenge to the student and to the practicing metallographer or metallurgist. This book brings together much of the existing knowledge pertaining to metallographic techniques and their application to the study of metals, ceramics, minerals, and polymers, although primary attention is given to metals.

This book concentrates on techniques relevant to visual and light microscopy—techniques fundamental to the study of macrostructure and microstructure. A similar treatment of techniques relevant to electron metallography is beyond the scope of this book, although some of the information presented is directly applicable. The historical development of metallographic techniques and the underlying scientific principles are discussed. Emphasis, however, has been placed on the practical problems associated with the use of these methods in order to facilitate their implementation. Metallography is both an art and a science, and both of these areas have been covered in detail. A complete list of recipes for polishing and etching solutions has been included plus comments regarding their safe and successful application. There are also extensive reference lists of key work at the end of each chapter to permit the reader to obtain additional information when needed. An extensive collection of macrographs and micrographs has also been included to illustrate the various methods discussed and to provide examples of their application to various materials.

This book should be useful to both undergraduate and graduate students in courses devoted to microscopy and physical metallurgy but should also prove useful to those studying ceramics, minerals, polymers, and carbonaceous materials. Engineers and technicians should find the book to be a valuable source of reference for use on the job. Although metallography is a relatively mature field, there has been substantial progress made in recent years in automation of sample

preparation and in quantification of microstructural measurements, subjects that are thoroughly covered in this book.

The author wishes to acknowledge the contributions made by his colleagues during the preparation of this manuscript over the past 10 years. Specifically, he appreciates the advice and encouragement from the reviewers and the photographs of equipment supplied by their manufacturers. The advice and help provided by metallographers at Bethlehem Steel's Homer Research Laboratories—A. O. Benscoter, A. V. Brandemarte, J. W. Guidon, J. R. Gruver, L. L. Hahn, J. R. Kilpatrick, M. L. Longenbach, V. E. McGraw, E. C. Poetl, M. A. Rodriguez, and L. R. Salvage—and by his former coworkers—H. A. Abrams, R. L. Bodnar, B. L. Bramfitt, J. C. Chilton, R. J. Henry, R. W. Hinton, M. L. Lasonde, A. R. Marder, M. Schmidt, M. J. Roberts, J. P. Snyder, E. T. Stephenson, and L. R. Woodyatt—were invaluable. The author gratefully acknowledges the following people who offered advice or provided samples or photomicrographs: A. Boe (Struers, Inc.), G. W. Blann (Buehler Ltd.), R. D. Buchheit (Battelle-Columbus Labs), A. E. Calabra (Rockwell International), R. S. Crouse (Oak Ridge National Lab.), R. T. DeHoff (University of Florida), E. W. Filer (Cabot Corp.), N. J. Gendron (retired, General Electric Corp.), J. F. Golden (E. Leitz, Inc.), R. J. Gray (Oak Ridge National Lab.), N. D. Greene (University of Connecticut), J. A. Hendrickson (Wyman-Gordon Co.), J. N. Hoke (Pennsylvania State University), W. Hunn (E. Leitz, Inc.), H. M. James (Carpenter Technology Corp.), R. R. Jones (Lafayette College), G. Krauss (Colorado School of Mines), J. A. Nelson (Buehler Ltd.), E. C. Pearson (Aluminum Co. of Canada), A. W. Pense (Lehigh University), G. Petzow (Max-Planck Institute), T. Piotrowski (Engelhard Minerals & Chemicals), J. H. Richardson (The Aerospace Corp.), R. M. Slepian (retired, Westinghouse Electric Corp.), R. H. Stevens (Aluminum Co. of America), D. A. Thomas (Lehigh University), F. J. Warmuth (Special Metals Corp.), E. Weidmann (Struers, Inc.), W. E. White (Petro Canada Ltd.), D. B. Williams (Lehigh University), E. E. Underwood (Georgia Institute of Technology), and W. Yankauskas (retired, TRW).

George F. Vander Voort

CONTENTS

Preface	xiii
Chapter 1 Macrostructure	1
1-1 Introduction	1
1-2 Visualization and Evaluation of Macrostructure by Etching	2
1-2.1 Macroetching with Acid Solutions	3
1-2.2 Copper-Containing Macroetchants for Primary Structure	5
1-2.3 Macroetchants for Revealing Strain Patterns	7
1-2.4 Macroetch Specifications	9
1-2.5 Classification of Macroetch Features	11
1-3 Applications of Macroetching	13
1-3.1 Solidification Structures	13
1-3.2 Billet and Bloom Macrostructures	15
1-3.3 Continuously Cast Steel Macrostructures	19
1-3.4 Consumable Electrode Remelted Steel Macrostructures	20
1-3.5 Dendrite Arm Spacing	21
1-3.6 Forging Flow Lines	27
1-3.7 Grain or Cell Size	30
1-3.8 Alloy Segregation	32
1-3.9 Carbide Segregation	33
1-3.10 Weldments	33
1-3.11 Strain Patterns	36
1-3.12 Failure Analysis	36
1-3.13 Response to Heat Treatment	39
1-3.14 Flame Cutting	39
1-4 Macrostructure Revealed by Machining	41
1-5 The Fracture Test	41
1-5.1 Composition	42
1-5.2 Inclusion Stringers	42

1-5.3	Degree of Graphitization	43
1-5.4	Grain Size	44
1-5.5	Depth of Hardening	44
1-5.6	Detection of Overheating	45
1-5.7	Evaluation of Quality	45
1-6	Special Print Methods	47
1-6.1	Contact Printing	47
1-6.2	Sulfur Printing	47
1-6.3	Oxide Printing	52
1-6.4	Phosphorus Printing	52
1-6.5	Lead Printing and Exudation Test	53
1-6.6	Miscellaneous Print Methods	56
1-7	Summary	57
Chapter 2	Specimen Preparation for Light Microscopy	60
2-1	Introduction	60
2-2	Sample Selection	60
2-3	Sectioning	62
2-3.1	Fracturing	62
2-3.2	Shearing	62
2-3.3	Sawing	63
2-3.4	Abrasive Cutting	63
2-3.5	Microtomy	69
2-3.6	Wire Saws	69
2-3.7	Electric Discharge Machining	70
2-3.8	Micromilling	70
2-3.9	Summary	70
2-4	Mounting	71
2-4.1	Cleaning	72
2-4.2	Adhesive Mounting	73
2-4.3	Clamps	73
2-4.4	Plastic Mounting Materials	75
	<i>Compression Mounting / Castable Mounts</i>	
2-4.5	Vacuum Impregnation	85
2-4.6	Taper Mounting	86
2-4.7	Edge Preservation	86
2-4.8	Conductive Mounts	91
2-4.9	Special Mounting Techniques	92
2-4.10	Mount Marking and Storage	92
2-4.11	Summary	93
2-5	Grinding	93
2-5.1	Grinding Media	95
2-5.2	Equipment	98
2-5.3	Lapping	100
2-6	Polishing	101
2-6.1	Equipment	103
2-6.2	Polishing Cloths	104
2-6.3	Polishing Abrasives	105
2-7	Grinding and Polishing Theory	112

2-8	Electromechanical Polishing	115
2-9	Attack Polishing	116
2-10	Chemical Polishing	117
2-11	Electropolishing	119
2-11.1	Advantages	120
2-11.2	Disadvantages	120
2-11.3	Equipment	121
2-11.4	Theory	121
2-11.5	Factors Influencing Electropolishing	124
2-11.6	Comparison of Mechanically and Electrolytically Polished Surfaces	124
2-12	Specific Polishing Recommendations	127
2-12.1	Universal Methods	127
2-12.2	Common Problems	127
	<i>Coatings / Graphite and Inclusion Retention</i>	
2-12.3	Metals	132
	<i>Aluminum / Antimony and Bismuth / Beryllium / Cadmium, Lead, Tin, and Zinc / Chromium, Molybdenum, and Tungsten / Cobalt, Manganese, Nickel, and Iron / Copper / Germanium and Silicon / Indium and Thallium / Magnesium / Niobium, Tantalum, and Vanadium / Precious Metals / Radioactive Metals / Rare Earth Metals / Selenium and Tellurium / Sodium / Titanium / Zirconium and Hafnium</i>	
2-12.4	Borides and Carbides	141
2-12.5	Carbonaceous Materials	142
2-12.6	Ceramics	143
2-12.7	Composites	144
2-12.8	Minerals	144
2-12.9	Polymers	146
2-13	Safety	148
2-13.1	Solvents	151
2-13.2	Acids	153
2-13.3	Other Chemicals	157
2-13.4	Summary	158
2-14	Summary	159
Chapter 3	Microstructure	165
3-1	Introduction	165
3-2	Etching	166
3-2.1	Etching Theory	166
3-2.2	Etching Technique	171
3-2.3	Etching Problems	172
3-2.4	Tint Etching	174
3-2.5	Electrolytic Etching	177
3-2.6	Anodizing	178
3-2.7	Potentiostatic Etching	178
3-2.8	Polarized-Light Etchants	180
3-3	Heat Tinting	182

3-4	Thermal Etching	185
3-5	Gas Contrasting	186
3-6	Vapor-Deposited Interference Films	187
3-7	Magnetic "Etching"	190
3-8	Ion-Bombardment Etching	191
3-9	Dislocation Etch Pitting	192
3-10	Corrosion Tests	194
3-11	Specific Etching Recommendations	195
	3-11.1 Metals	195
	<i>Aluminum and Alloys / Antimony and Bismuth / Beryllium / Cadmium, Lead, Tin, and Zinc / Chromium, Molybdenum, and Tungsten / Cobalt and Manganese / Copper and Alloys / Germanium and Silicon / Indium and Thallium / Iron and Steels / Magnesium and Alloys / Nickel and Alloys / Niobium, Tantalum, and Vanadium / Precious Metals / Radioactive Metals / Rare Earth Metals / Selenium and Tellurium / Titanium and Alloys / Zirconium and Hafnium</i>	
	3-11.2 Borides, Carbides, Nitrides, and Oxides	254
	3-11.3 Polymers	257
	3-11.4 Minerals	257
3-12	Summary	258
Chapter 4	Light Microscopy	267
4-1	Introduction	267
4-2	Basic Concepts in Light Optical Theory	268
4-3	The Light Microscope	270
	4-3.1 Illumination	271
	<i>Low-Voltage Tungsten Filament Lamp / Carbon Arc / Xenon Arc / Quartz-Iodine Lamp / Zirconium Arc Lamp / Mercury Vapor Lamp</i>	
	4-3.2 Condenser System	274
	4-3.3 Light Filters	274
	4-3.4 Objective Lens	275
	4-3.5 Eyepieces	278
	4-3.6 Stage	280
	4-3.7 Control of Microscope Variables	281
	4-3.8 Lens Defects	282
	4-3.9 Resolution and Depth of Field	282
4-4	Examination Modes in Light Microscopy	292
	4-4.1 Methods of Examination	292
	<i>Bright-Field illumination / Oblique Illumination / Dark-field Illumination / Polarized Light / Phase-Contrast Illumination / Interference Techniques / Ultraviolet Microscopy / Light-Section Microscopy / Fluorescence Microscopy / Infrared Microscopy</i>	
4-5	Light Phenomena	309
4-6	Photomicrography	312
	4-6.1 Obtaining Good Photomicrographs	313

4-6.2	Black-and-White Photography	314
4-6.3	Color Photography	316
4-6.4	Film Handling	317
4-7	Photomacrography	318
4-8	Auxiliary Techniques	322
4-8.1	Microhardness	322
4-8.2	Hot-Stage Microscopy	322
4-8.3	Special States for <i>In Situ</i> Experiments	323
4-8.4	Hot-Cell Microscopy	326
4-8.5	Field Microscopy	327
4-8.6	Comparison Microscopes	328
4-8.7	Television Monitors	328
4-8.8	Clean-Room Microscopy	329
4-9	Summary	329

Chapter 5	Hardness	334
5-1	Introduction	334
5-2	Indentation Hardness	335
5-2.1	Relationship to Stress-Strain Curve	335
5-2.2	Effects of Time, Velocity, and Size	337
5-2.3	Effects of Lubrication and Adhesion	338
5-2.4	Indentation Size and Shape Changes	338
5-2.5	Surface Roughness	339
5-3	Static Hardness Tests	339
5-3.1	Brinell Hardness	339
5-3.2	Meyer Hardness	346
5-3.3	Vickers Hardness	350
5-3.4	Rockwell Hardness	355
5-3.5	Other Static Hardness Tests	366
	<i>Ludwik Conical Indentation Test / Mutual Indentation Tests / Mallock Cone Test / Scratch Hardness / Hardness Tests for Nonmetallic Materials</i>	
5-4	Dynamic Hardness Tests	369
5-4.1	Shore Scleroscope	370
5-4.2	Pendulum Hardness	371
5-4.3	Cloudburst Test	371
5-4.4	Equotip Hardness	371
5-5	Nondestructive Hardness Tests	372
5-6	Microindentation Hardness	373
5-7	Hardness Conversions	382
5-8	Applications	383
5-8.1	Anisotropy	383
5-8.2	Indentation Fracture	385
5-8.3	Machinability	389
5-8.4	Phase Identification	391
5-8.5	Prediction of Other Properties	391
5-8.6	Quality Control	393
5-8.7	Residual Stress	396
5-8.8	Temperature Effects	396

5-8.9	Wear	398
5-8.10	Miscellaneous Applications	403
5-9	Summary	404
Chapter 6	Quantitative Microscopy	410
6-1	Introduction	410
6-2	Basic Measurement Variables	412
6-2.1	Sampling	412
6-2.2	Sample Preparation	412
6-2.3	Field Selection	413
6-3	Standard Chart Methods	414
6-4	Measurement of Structural Gradients	414
6-4.1	Decarburization	415
6-4.2	Case Depth	417
6-4.3	Coating Thickness	422
6-5	Stereology Terminology	423
6-6	Volume Fraction	425
6-6.1	Areal Analysis	426
6-6.2	Lineal Analysis	426
6-6.3	Point Counting	426
6-6.4	Statistical Analysis	428
6-6.5	Comparison of Methods	432
6-6.6	Summary	435
6-7	Grain Size	435
6-7.1	Grain Shape	435
6-7.2	Grain Size Measurement	436
	<i>Delineation of Grain Boundaries / Standard Chart Methods / Jeffries Planimetric Method / Triple-Point Count Method / Heyn Intercept Method / Nonequiaxed Grains / Duplex Grain Structures / Two-Phase Structures / Snyder-Graff Intercept Method / Fracture Grain Size / Accuracy of Grain Size Estimates / Relationship of \bar{L}_3 to Other Grain Parameters</i>	
6-7.3	Grain Size Distributions	465
6-7.4	Summary	471
6-8	Inclusion Rating Methods	472
6-8.1	Chart Comparison Methods	472
6-8.2	Nonchart Rating Methods	474
6-8.3	Inclusion Deformability	477
6-8.4	Summary	479
6-9	Line Length	480
6-10	Spacings	480
6-10.1	Mean Free Path and Mean Spacing	480
6-10.2	Interlamellar Spacing	481
6-11	Contiguity	485
6-12	Anisotropy	486
6-13	Shape	486
6-14	Particle Size	488

6-15	Electron Microscopy Techniques	493
6-16	Quantitative Fractography	494
6-17	Image Analysis	499
6-18	Applications	502
6-19	Summary	502

Appendixes 509

A	Etchants for Revealing Macrostructure	509
B	Macroetchants Based on Copper-Containing Compounds—For Etching of Iron and Steel	533
C	Macroetchants for Revealing Strain Patterns in Nonferrous Metals	536
D	Electroless and Electrolytic Plating Procedures	538
E	Electromechanical Polishing Procedures	541
F	Attack Polishing Procedures	543
G	Chemical Polishing Solutions	552
H	Electrolytic Polishing Solutions	562
I	Etchants for Revealing Microstructure	610
J	Dislocation Etching Techniques	712

Indexes 733

Author Index
Subject Index

MACROSTRUCTURE

1-1 INTRODUCTION

Macroscopic examination techniques are frequently employed in routine quality control, in failure analysis, and in research studies. These techniques are generally a prelude to microscopic examination; however, in quality control, they are often used alone as a criterion for acceptance or rejection. A great variety of destructive and nondestructive procedures are available. The most basic procedure involves simple visual examination for surface features such as seams, laps, or scale.

This chapter describes only destructive test procedures; nondestructive methods are not covered. These destructive methods include the following procedures:

- Macroetching
- Contact printing
- Fracturing
- Lead exudation

Proper implementation of these methods is fundamental to the manufacture of materials. In quality control, the manufacturing routine is usually established according to set practices, and the macroscopic methods are used to detect deviations from the norm. In failure studies, one often does not know specific details of the manufacturing process and practices, and the engineer uses these tests to judge quality, to locate problem areas for further study, and, in some cases, to determine how the component was produced. In research studies, the processing steps are often varied, and the macroexamination is designed to show differences due to changes in manufacturing practices. Thus for each type of study, the specific details of the macroscopic examination will vary somewhat, and

the practitioner must have a thorough understanding of the test method, its application, and the interpretation of test data.

Interpretation of the data from these tests requires an understanding of the manufacturing process, since the macrostructure is dependent on the solidification and hot- or cold-working procedures used. There can be pronounced differences in macrostructure because factors such as casting method, ingot size and shape, and chemical properties will significantly alter the solidification pattern. In addition, the use of manufacturing techniques other than traditional ingot casting, such as continuous casting, centrifugal casting, electroslag remelting, or hot-isostatic pressing, produce noticeably different as-cast patterns. Also, there is a wide variety of metalworking processes that can be applied to material made by any of the above processes, and each exerts a different effect upon the material. All these factors influence the interpretation of the test results.

No material can be said to be entirely homogeneous either macroscopically or microscopically. The degree of heterogeneity can vary widely depending on the nature of the material, the method of manufacture, and the cost required to produce the material. Fortunately, the usual degree of heterogeneity is not a serious problem in the use of commercial materials as long as these variances are held within certain prescribed limits. Certain problems, such as pipe and hydrogen flakes, are in general, quite harmful. The effect of other features, such as porosity, segregation, and inclusions, can be quite difficult to evaluate, and one must consider the extent of these features, the amount of subsequent metalworking, and the nature of the application of the material.

Of the metallographic procedures listed, the macroetch test is probably the most informative, and it is widely used for quality control, failure analysis, and research studies. Classification of the features observed with the macroetch test is often confusing because of the use of "jargon" created since the introduction of this test procedure. The macroetch test is covered in considerable detail in this chapter, and numerous examples of its application to a variety of materials are presented.

1-2 VISUALIZATION AND EVALUATION OF MACROSTRUCTURE BY ETCHING

All quality evaluations should begin on the macroscale using tests designed to survey the overall field in a simple and reliable manner. After the macrostructure of a material has been evaluated, specific features can then be examined microscopically. Abnormalities observed on the etch disc can be studied by fracturing the disc or by preparing metallographic polished samples. Macroetching of transverse or longitudinally oriented samples, i.e., oriented with respect to the hot-working axis, enables the mill metallurgist to evaluate the quality of a relatively large area quickly and efficiently. Thus, macroetching is an extremely powerful tool and is a cornerstone of the overall quality program.

The earliest macroetchants were rather weak solutions used at room temperature. Reaumur (1683–1757) used macroetchants to distinguish between different types of steel and sketched the appearance of macroetched pieces of steel in his work. Rinmann promoted this technique in his book *On the Etching of Iron and Steel*, written in the late 1700s. Sorby, in his classic work published in 1887 “On the Microscopical Structure of Iron and Steel,” showed “nature prints,” which were inked contact prints of steel etched in moderately strong aqueous nitric acid solutions [1]. The early etching solutions have been reviewed in the classic text by Berglund [2].

1-2.1 Macroetching with Acid Solutions

The first “deep”-etching procedure for steel was developed by Waring and Hofamman using nine parts hydrochloric acid, three parts sulfuric acid, and one part water. Considerable adverse comment about the use of strong acids to evaluate highly stressed components was generated by this paper. Overall, the initial response to deep-acid etching was negative; however, numerous subsequent studies revealed the great value of such etchants.

After the initial work by Waring and Hofamman, considerable attention was devoted to the study of strong acids for deep etching steels. The most widely used deep etch consists of a 1:1 solution of reagent-grade† hydrochloric acid and water heated to 160 to 180°F for 15 to 45 min. Etching can be conducted on a saw-cut face, but better resolution is obtained with ground faces. Gill and Johnstin found that this etch was more selective in its attack than similar solutions involving nitric acid and water or sulfuric acid and water [3]. An important feature of this etchant is that evaporation does not significantly vary its composition during use.

The following items should be considered in the development of a macroetchant:

- The etchant should produce good all-around results, should be applicable to the majority of materials, and should reveal a great variety of structural characteristics and irregularities.
- The etchant should be simple in composition, inexpensive, and easy to prepare.
- The etchant should be stable during use or storage.
- The etchant must be safe to use and should not produce noxious odors.

The widespread popularity of the 1:1 hydrochloric acid and water etch is due to the fact that it satisfies these requirements better than other etchants. Appendix A lists macroetchants for iron and steel as well as for other metals.

The 1:1 hydrochloric acid and water etch attacks manganese sulfides readily but does not attack aluminum oxides. Steels high in aluminum content, such as the nitriding alloys, are etched best with an aqueous solution containing 10% hydro-

†The reagent grade contains 36.5 to 38% HCl, whereas the technical grade contains 28% HCl.

chloric acid and 2% nitric acid, developed by V. T. Malcolm. Etching is conducted at 180°F for 15 to 60 min.

As the alloy content increases, so does the degree of segregation and its associated problems. Etching is pronounced at the segregate-matrix interface, and segregate or matrix areas may etch out, leaving pits. Sulfides or carbides may also etch out, leaving pits. Before the investigator can distinguish between pits due to nonmetallic inclusions or segregates and carbides, the disc must be hardened and reetched. If the pits were due to nonmetallics, they will be present to the same degree in both the annealed and the hardened discs.

Watertown Arsenal [4] developed a variant of the standard etch that consists of 38 parts of hydrochloric acid, 12 parts sulfuric acid, and 50 parts water.† This reagent often produces a sharper definition of features than the standard etch, and like the standard etch, its acid concentration does not change markedly during use.

Macroetching provides an overall view of the degree of uniformity of metals and alloys by revealing:

- Structural detail resulting from solidification or working
- Chemical uniformity in qualitative terms
- Physical discontinuities due to solidification, working, etc.
- Weldment structure or heat-affected zones from burning operations
- Hardness patterns in non-through-hardened steels or patterns due to quenching irregularities
- Grinding damage
- Thermal effects due to service abuse

The first three features are best revealed by hot-acid etching, and the remaining four are best revealed by room temperature etchants. Macroetching is usually performed on ground surfaces, although in some cases, especially with cold etchants, better results are obtained when the surface is polished. Chemical segregation can be shown by certain cold etchants. The information obtained can be recorded by photographing the samples or, where possible, by contact printing.

In order to observe these features, one must sample the material properly and use the macroetch test procedure correctly. Fortunately, these test procedures are straightforward and simple to use as long as a few precautions are followed. In practice, one must consider the following test variables:

- Selection of representation samples
- Choice of surface orientation
- Proper preparation of sample surface
- Selection of the best etch composition
- Control of etchant temperature and etch time
- Documentation of test results

†Add the sulfuric acid *slowly* to the water and allow it to cool; then add the hydrochloric acid.