MANUAL ON ELECTRON METALLOGRAPHY TECHNIQUES

Sponsored by
Subcommittee E04.11 on
Electron Microscopy and Diffraction of
Committee E-4 on Metallography
AMERICAN SOCIETY FOR TESTING AND MATERIALS

ASTM SPECIAL TECHNICAL PUBLICATION 547
G. N. Maniar and Albert Szirmae, coordinators

List price $5.25
04-547000-28

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race Street, Philadelphia, Pa. 19103
NOTE

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

Related
ASTM Publications

Applications of Modern Metallographic Techniques,
STP 480 (1970), $17.00 (04-480000-28)

Application of Electron Microfractography to Materials
Research, STP 493 (1971), $8.25 (04-493000-30)

Stereology and Quantitative Metallography, STP 504
(1972), $9.75 (04-504000-28)
Contents

Introduction

Chapter 1—Procedures for Standard Replication Techniques for Electron Microscopy

1.1 Introduction
1.2 Specimen Preparation for Replication
   1.2.1 Mounting
   1.2.2 Polishing
   1.2.3 Etching
1.3 Replication
   1.3.1 Direct Methods
   1.3.2 Indirect Methods
   1.3.3 Fracture Replication
1.4 Summary
References

Chapter 2—Extraction Replica Techniques

2.1 Introduction
2.2 General Methods
   2.2.1 Direct Stripped Plastic Extraction Replicas
   2.2.2 Indirect Stripped Plastic Extraction Replicas
   2.2.3 Positive Carbon Extraction Replica
   2.2.4 Direct Carbon Extraction Replica
   2.2.5 Extraction Replicas Removed by Two Stage Etching
   2.2.6 Replication of Thin Surface Films
   2.2.7 Aluminum Oxide Extraction Replica
2.3 Tables of Extraction Replica Techniques
2.4 Bibliography
References

Chapter 3—Thin Foil Preparation for Transmission Electron Microscopy

3.1 Introduction
3.2 Bulk Thinning to 500 μm (0.5 mm)
   3.2.1 Cutoff Wheel
   3.2.2 Spark Machining
   3.2.3 Electrolytic Acid Saw and Acid Planing Wheel
3.3 Prethinning to 50 μm (0.05 mm)
   3.3.1 Surface Grinding and Hand Grinding
   3.3.2 Cold Rolling
   3.3.3 Chemical Prethinning
   3.3.4 Electrolytic and Jet Prethinning

References
3.4 Final Thinning to Less Than 0.5 μm
   3.4.1 Bollmann Method
   3.4.2 Window Method
   3.4.3 Disa Electropol Polishing
   3.4.4 Chemical Polishing
   3.4.5 Electrolytic and Automatic Jet Polishing
3.5 Unique Thinning Techniques
   3.5.1 Small Diameter Wires
   3.5.2 Microtomy
   3.5.3 Ion Micro Milling
3.6 General Precautions
   3.6.1 Mixing Electrolytes
   3.6.2 Polishing Film and Staining
   3.6.3 Electrolyte and Specimen Temperature
   3.6.4 Cutting and Mounting
3.7 Summary
3.8 Acknowledgments
Appendix 3.1
Appendix 3.2
Appendix 3.3
References

Chapter 4—Selected Area Electron Diffraction Analysis of Extraction Replica and Thin Foil Specimens in the Transmission Electron Microscope

4.1 Introduction
Part I—Particle or Second Phase Identification Using Extraction Replica and Selected Area Electron Diffraction
4.2 Introduction
4.3 Technique for Preparing Extraction Replicas
4.4 Indexing Selected Area Electron Diffraction Patterns
   4.4.1 Calibration of the Microscope Constant
   4.4.2 Identification of Unknown Diffraction Patterns
   4.4.3 Indexing Simple Single Particle Spot Patterns
4.5 Summary
Part II—Analysis of Crystallographic Features and Defects in Thin Foil Specimens
4.6 Introduction
4.7 Steps in the Solution of a Selected Area Spot Electron Diffraction Pattern of a Thin Foil Specimen
Appendix 4.1
Appendix 4.2
Appendix 4.3
Appendix 4.4
References