TECHNIQUES OF METALS RESEARCH
Volume I, Part 1

Techniques of Materials Preparation and Handling

Editor
E. F. BUNSHAH

TECHNIQUES OF METALS RESEARCH
Volume I, Part 1

Techniques of Materials Preparation and Handling

Editor
R. F. BUNSHAH

Authors of Part 1

A. L. AUSTIN
T. H. BATZER
R. F. BUNSHAH
P. DUVEZ
W. M. FASSELL
W. FULKERSON
C. D. HENNING
D. L. McELROY
R. PARKER
G. R. SPEICH
A. SZIRMAE

1968
INTERSCIENCE PUBLISHERS
a division of John Wiley & Sons, New York, London, Sydney

Library of Congress Catalog Number 67-20260
Printed in the United States of America

TECHNIQUES OF METALS RESEARCH

R. F. Bunshah, Editor

Volume I:	Techniques of Materials Preparation and Handling Parts 1 to 3
Volume II:	Techniques for the Direct Observation of Structure and Imperfections Parts 1 and 2
Volume IIA:	The Stereographic Projection and Its Applications
Volume IIB:	Transmission Diffraction Electron Microscopy
Volume IIC:	Handbook of Metallographic Techniques
Volume III:	Modern Analytical Techniques for Metals and Alloys
Volume IIIA:	Electrical Resistivity and Hall Effect Measurements
Volume IV:	Physicochemical Measurements in Metals Research (Editor: R. A. Rapp)
Volume V:	Measurement of Mechanical Properties
Volume VI:	Measurement of Physical Properties (Editor: E. Passaglia)
Volume VII:	Techniques Involving Extreme Environment, Nondestructive Techniques, Computer Methods in Metals Research, and Data Analysis

TECHNIQUES OF METALS RESEARCH

R. F. Bunshah, Editor

INTRODUCTION TO THE SERIES

Progress in metals research and all the related areas of materials research in the past two decades has been greatly affected by the introduction of new methods, techniques, and instruments, some of which are highly sophisticated. Much of the modern methodology of mathematics, physics, and chemistry has been called upon to refine the investigation of matter in the metallic state and in the solid state at large. Thus, a large number of many different types of techniques are currently used in metals research.

This has been the case not only for the fundamental and scientific exploration of the metallic state, but also for the techniques used to measure properties, and evaluate performance, and for applications in actual use.

In comparison to the immediate post World War II era (circa 1945), a much larger number of metals and their alloys are in use today. The much greater variety of their functions under normal and extreme conditions has led to the development of a vast body of knowledge and especially of highly specialized methods and techniques. As usual, information about techniques is more widely scattered than information on results.

The term "metals research" is used here in its broadest sense to include the "development" connotation, and it is not restricted to small-scale laboratory experiments. In fact, a successful pursuit of a research or development program nowadays often requires a thorough comprehensive consideration of all aspects of the problem, from preparation and handling of the primary purified metal through refining, consolidation, mechanical and heat treatment, sample machining, and preparation, to measurement of physical, chemical, and mechanical properties and chemical composition.

It seems therefore appropriate to put together a well-organized, integrated presentation of the total area of techniques of metals research comprising all of the aspects mentioned above.

One of the best ways to summarize a large body of information is a comprehensive, well-referenced review article. This series consists of review articles on the various techniques of metals research. The number of techniques is large and the rate of development of new techniques is rapid, which makes it possible that some newer techniques may have been

omitted in this edition, although a conscientious effort has been made to make the coverage as complete as possible. Some topics which are very marginal to the concept of this series, e.g., ore mining and beneficiation, have been intentionally omitted. Similarly, only the newer techniques of chemical analysis, particularly those dealing with trace analysis, and analysis of interstitials are covered.

Several books have been published at irregular intervals in the past 20 years dealing with fragments of the area we are trying to describe. Our primary aim is to bring together a comprehensive collection of the techniques of metals research.

This could, of course, be done only as a collaborative effort. The result will be a series of self-consistent volumes which will as a whole, we hope, form a reliable guide to the multifarious problems which arise in every laboratory in which metals, metallic substances, and other similar materials are investigated. Each volume will deal with a broad subject area.

While it is not the aim to tell the expert in a particular method or discipline more than he knows already, the articles in this series will enable the graduate student, the researcher, or the supervisor with an adequate background in the physical sciences, metallurgy, or engineering to gain access to a technique with which he is unfamiliar and to enable him to judge its applicability to a special problem and, even more important, its limitations. The contributions are written in such a style that the reader should be able to use them immediately without further reference. Ample literature quotations will guide him to more detailed information.

It is not intended that the articles in this series be written in cookbook style, so that it would enable somebody to go straight to the laboratory and use the techniques he has been reading about.

Each article in the series is an entity in itself and the reader will therefore occasionally find overlap of subject matter between articles. However, it is the editor's philosophy that it is far more important to preserve the integrity and continuity of statements in an article than prune out a few pages here and there. The general organization of each article is to give the theoretical background necessary to understand the various techniques, a description of the technique or techniques, a discussion of their advantages and limitations, and a bibliography for further reading.

Within this general scope, the author has been given the freedom to develop his topic as he sees fit. Therefore, the relative weight given to each of these categories in a given article will depend on the subject matter being treated. Some articles dealing with new techniques are almost entirely descriptive, whereas others dealing with old, established techniques, like x-ray diffraction, are essentially organized, well-referenced summaries of groups of techniques which are dealt with in detail elsewhere.

It is rather difficult to organize and arrange the rather heterogeneous topics in a completely rational and systematic fashion. For example, the same topic could equally well be placed in more than one volume. I have chosen the following overall breakdown:

Volume I:	Techniques of Materials Preparation and Handling
Volume II:	Techniques for the Direct Observation of Structure and Imperfections
Volume IIA:	The Stereographic Projection and Its Applications
Volume IIB:	Transmission Diffraction Electron Microscopy
Volume IIC:	Handbook of Metallographic Techniques
Volume III:	Modern Analytical Techniques for Metals and Alloys
Volume IIIA:	Electrical Resistivity and Hall Effect Measurements
Volume IV:	Physicochemical Measurements in Metals Research
Volume V:	Measurement of Mechanical Properties
Volume VI:	Measurement of Physical Properties
Volume VII:	Techniques Involving Extreme Environment, Nondestructive Techniques, Computer Methods in Metals Research, and Data Analysis

When a particular article becomes sufficiently large or a particular topic is unusually broad, it will be published as a separate part under its own cover. It is hoped that the comprehensive Index which will be published at the end of the series will help in locating any and all of the topics discussed.

In planning and preparing the work, the editor has enjoyed the assistance and counsel of the Editorial Advisory Board as well as many other colleagues. Their help is gratefully acknowledged herewith.

He is also very grateful to the Editors for Volumes IV and VI, Dr. Robert A. Rapp and Dr. E. Passaglia, respectively.

My sincere thanks are also due to the authors of the various articles in the series and to the Editorial and Production Departments of the publisher.

This is a first attempt to perform a very large task. The editor, therefore, invites the comments, suggestions, and criticism of the readers to improve these volumes in future editions.

R. F. Bunshah

Livermore, California

PREFACE TO VOLUME I

As the title indicates, this volume describes the various methods used in materials preparation and handling. This is done because it is felt that the investigator who wishes to carry out studies on a particular material cannot divorce himself from a thorough knowledge of the steps used in the preparation of the material. As materials requirements and specifications become more stringent, all the steps which are included in the preparation of a test specimen are as important, if not more important, than the property measurement itself.

The scope of this volume, which is divided into three parts, can be seen from the titles of the various sections, which are as follows:

I. Environment Control
II. Temperature Measurement and Control
III. Heat and Quenching Techniques and the Heat Treatment of Metals
IV. Preparation of Primary Purified Metals, Refining of Metals, and Separation of Isotopes
V. Techniques to Consolidate and Refine Primary Purified Metals
VI. Casting Techniques
VII. Techniques of Crystal Growth and Controlled Microstructures
VIII. Deposition Processes for Various Applications
IX. Fabrication Processes
X. Joining Methods
XI. Special Machining Methods
XII. Powder Metallurgy Techniques

The size of the product considered in the various technologies described ranges from tiny whiskers (fractions of a millimeter) to large billets, fabricated products, etc.

No excuse is made for the fact that several of the techniques are of "industrial scale." The nature of the beast demands a large laboratory, if you will. There should be no misconceptions that this series is concerned

only with laboratory scale experiments. The words "metals research" are used in their broadest connotation.

As necessary, I have added editorial comments at the start of a few major sections.

R. F. BUNSHAH

Livermore, California
January, 1968

Techniques of Materials Preparation and Handling

Volume 1

CONTENTS

PART 1

PART 2

PART 3

Séction I
ENVIRONMENT CONTROL

Chapter 1
VACUUM TECHNIQUES

R. F. BUNSHAH and T. H. BATZER, Lawrence Radiation Laboratory, University of California, Livermore, California

I. INTRODUCTION

A large number of operations in metallurgical research necessitate the use of vacuum techniques. The requirements for the various applications are very diverse. In general, they are based on three parameters:

1. The operational pressure P_w at which the process should be carried out.

2. The quantity of "process gas" (i.e., the gas evolved or produced during processing) to be pumped while maintaining the operational pressure, i.e. the pumping speed of the system at the operational pressure must be sufficiently high to handle the maximum process gas load.

3. The permissible pressure of background gas, P_b, which determines the amount of contamination during processing.

Thus, a wide variety of different types of vacuum equipment is utilized. Table 1 lists the various pressure regimes in vacuum technology and their associated properties.

The object of this article is to review the salient features of the following topics—system design considerations, the various types of vacuum hardware (pumps, gauges, valves, seals, materials of construction, etc.), leak-hunting, and the precautions for the proper operation of vacuum systems. For further details, the reader is referred to several books and review articles on vacuum techniques and their applications to metallurgy (see list of general references).