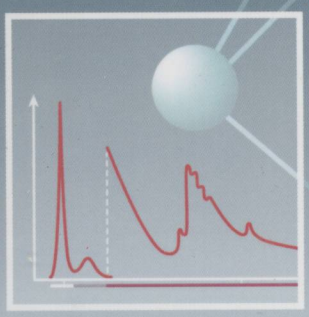
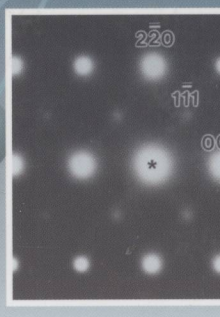
David Brandon and Wayne D. Kaplan

Microstructural Characterization of Materials





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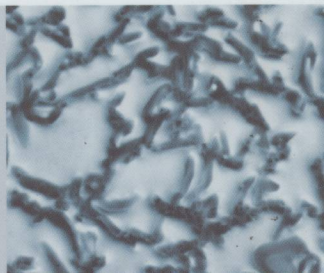

Microstructural Characterization of Materials

2nd Edition

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Preface to the Second Edition

The last decade has seen several major changes in the armoury of tools that are routinely available to the materials scientist and engineer for microstructural characterization. Some of these changes reflect continuous technological improvements in the collection, processing and recording of image data. Several other innovations have been both dramatic and unexpected, not least in the rapid acceptance these tools have gained in both the research and industrial development communities.

The present text follows the guidelines laid down for the first edition, exploring the methodology of materials characterization under the three headings of *crystal structure, microstructural morphology* and *microanalysis*. One additional chapter has been added, on *Scanning Probe Microscopy*, a topic that, at the time that the first edition was written, was very much a subject for active research, but a long way from being commonly accessible in university and industrial laboratories. Today, *atomic force* and *scanning tunnelling microscopy* have found applications in fields as diverse as optronics and catalysis, friction and cosmetics.

It has proved necessary to split the chapter on *Electron Microscopy* into two chapters, one on *Transmission* techniques, and the other on *Scanning* methods. These two expanded chapters reflect the dramatic improvements in the resolution available for *lattice imaging* in transmission, and the revolution in sampling and micro-machining associated with the introduction of the *focused ion beam* in scanning technology.

The final chapter, on *Quantitative Analysis*, has also been expanded, to accommodate the rapid advances in three-dimensional reconstruction that now enable massive data sets to be assembled which include both chemical and crystallographic data embedded in a frame of reference given by microstructural morphology. Not least among the new innovations are *orientation imaging microscopy*, which allows the relative crystallographic orientations of the grains of a polycrystalline sample to be individually mapped, and *atom probe tomography*, in which the ions extracted from the surface of a sharp metallic needle are chemically identified and recorded in three dimensions. This last instrument is a long way from being widely available, but a number of laboratories do offer their services commercially, bringing three-dimensional analysis and characterization well below the nanometre range, surely the ultimate in microstructural characterization.

It only remains to note the greatest difference between the present text and its predecessor: digital recording methods have all but replaced photography in every application that we have considered, and we have therefore included sections on digital recording, processing and analysis. This 'digital revolution' has crept up on us slowly, following the on-going improvements in the storage capacity and processing speed for computer hardware and software. Today, massive amounts of digital image data can be handled rapidly and reliably.

At the same time, it is still up to the microscopist and the engineer to make the critical decisions associated with the selection of samples for characterization, the preparation of suitable sections and the choice of characterization methods. This task is just as difficult today as it always was in the past. Hopefully, this new text will help rather than confuse!

Most of the data in this book are taken from work conducted in collaboration with our colleagues and students at the Technion. We wish to thank the following for their contributions: David Seidman, Rik Brydson, Igor Levin, Moshe Eizenberg, Arnon Siegmann, Menachem Bamberger, Michael Silverstein, Yaron Kauffmann, Christina Scheu, Gerhard Dehm, Ming Wei, Ludmilla Shepelev, Michal Avinun, George Levi, Amir Avishai, Tzipi Cohen, Mike Lieberthal, Oren Aharon, Hila Sadan, Mor Baram, Lior Miller, Adi Karpel, Miri Drozdov, Gali Gluzer, Mike Lieberthal, and Thangadurai Paramasivam.

D.B.
W.D.K.

Preface to the First Edition

Most logical decisions rely on providing acceptable answers to precise questions, e.g. *what, why and how?* In the realm of scientific and technical investigation, the first question is typically *what is the problem* or *what is the objective?* This is then followed by a *why* question which attempts to pinpoint priorities, i.e. the urgency and importance of finding an acceptable answer. The third type of question, *how* is usually concerned with identifying means and methods, and the answers constitute an assessment of the available resources for resolving a problem or achieving an objective. The spectrum of problems arising in materials science and technology very often depends critically on providing adequate answers to these last two questions. The answers may take many forms, but when materials expertise is involved, they frequently include a need to characterize the *internal microstructure* of an engineering material.

This book is an introduction to the expertise involved in assessing the microstructure of engineering materials and to the experimental methods which are available for this purpose. For this text to be meaningful, the reader should understand why the investigation of the internal structure of an engineering material is of interest and appreciate why the microstructural features of the material are so often of critical engineering importance, This text is intended to provide a basic grasp of both the methodology and the means available for deriving qualitative and quantitative microstructural information from a suitable sample.

There are two ways of approaching *materials characterization.* The first of these is in terms of the engineering properties of materials, and reflects the need to know the physical, chemical and mechanical properties of the material before we can design an engineering system or manufacture its components. The second form of characterization is that which concerns us in this book, namely the *microstructural characterization* of the material. In specifying the internal microstructure of an engineering material we include the chemistry, the crystallography and the structural morphology, with the term materials characterization being commonly taken to mean just this specification.

Characterization in terms of the chemistry involves an identification of the chemical constituents of the material and an analysis of their relative abundance, that is a determination of the chemical composition and the distribution of the chemical elements within the material. In this present text, we consider methods which are available for investigating the chemistry on the microscopic scale, both within the bulk of the material and at the surface.

Crystallography is the study of atomic order in the crystal structure. A crystallographic analysis serves to identify the phases which are present in the structure, and to describe the atomic packing of the various chemical elements within these phases. Most phases are highly ordered, so that they are *crystalline* phases in which the atoms are packed together in a well-ordered, regularly repeated array. Many solid phases possess no such long-range order, and their structure is said to be *amorphous* or *glassy.* Several

quasicrystalline phases have also been discovered in which classical long-range order is absent, but the material nevertheless possesses well-defined rotational symmetry.

The *microstructure* of the material also includes those *morphological* features which are revealed by a microscopic examination of a suitably prepared specimen sample. A study of the microstructure may take place on many levels, and will be affected by various parameters associated with specimen preparation and the operation of the microscope, as well as by the methods of data reduction used to interpret results. Nevertheless, *all* microstructural studies have some features in common. They provide an image of the internal structure of the material in which the image contrast depends upon the interaction between the specimen and some incident radiation used to probe the sample morphology. The image is usually magnified, so that the region of the specimen being studied is small compared with the size of the specimen. Care must be exercised in interpreting results as being 'typical' of the bulk material. While the specimen is a three-dimensional object, the image is (with few exceptions) a two-dimensional projection. Even a qualitative interpretation of the image requires an understanding of the spatial relationship between the two-dimensional imaged features and the three-dimensional morphology of the bulk specimen.

Throughout this book we are concerned with the interpretation of the interaction between the probe and a sample prepared from a given material, and we limit the text to probes of X-rays, visible light or energetic electrons. In all cases, we include three stages of investigation, namely specimen preparation, image observation and recording, and the analysis and interpretation of recorded data. We will see that these three aspects of materials characterization interact: the microstructural morphology defines the phase boundaries, and the shape and dimensions of the grains or particles, the crystallography determines the phases present and the nature of the atomic packing within these phases, while the microchemistry correlates with both the crystallography of the phases and the microstructural morphology.

This text is intended to demonstrate the versatility and the limitations of the more important laboratory tools available for microstructural characterization. It is *not* a compendium of all of the possible methods, but rather a teaching outline of the most useful methods commonly found in student laboratories, university research departments and industrial development divisions.

Most of the data in this book are taken from work conducted in collaboration with our colleagues and students at the Technion. We wish to thank the following for their contributions: Moshe Eizenberg, Arnon Siegmann, Menachem Bamberger, Christina Scheu, Gerhard Dehm, Ming Wei, Ludmilla Shepelev, Michal Avinun, George Levi, Mike Lieberthal, and Oren Aharon.

D.B.
W.D.K.

Contents

1

The Concept of Microstructure

This text provides a basic introduction to the most commonly used methods of microstructural characterization. It is intended for students of science and engineering whose course requirements (or curiosity) lead them to explore beyond the accepted causal connection between the engineering properties of materials and their microstructural features, and prompt them to ask how the microstructures of diverse materials are characterized in the laboratory.

Most introductory textbooks for materials science and engineering emphasize that the processing route used to manufacture a component (shaping processes, thermal treatment, mechanical working, etc.) effectively determines the microstructural features (Figure 1.1). They note the interrelation between the microstructure and the chemical, physical, and/or mechanical properties of materials, developing expressions for the dependence of these properties on such microstructural concepts as *grain size* or *precipitate volume fraction*. What they do *not* usually do is to give details of either the methods used to identify microstructural features, or the analysis required to convert a microstructural observation into a parameter with some useful engineering significance.

This book covers three aspects of microstructural characterization (Table 1.1). First, the different crystallographic phases which are present in the sample are identified. Secondly, the morphology of these phases (their size, shape and spatial distribution) are characterized. Finally, the local chemical composition and variations in chemical composition are determined.

In all three cases we will explore the characterization of the microstructure at both the qualitative and the quantitative level. Thus, in terms of crystallography, we will be concerned not only with qualitative phase identification, but also with the elementary aspects of applied crystallography used to determine crystal structure, as well as with the quantitative determination of the volume fraction of each phase. As for the microstructure, we will introduce *stereological relationships* which are needed to convert a qualitative observation of morphological features, such as the individual grains seen in a cross-section, into a clearly defined microstructural parameter, the grain size. Similarly, we shall not be

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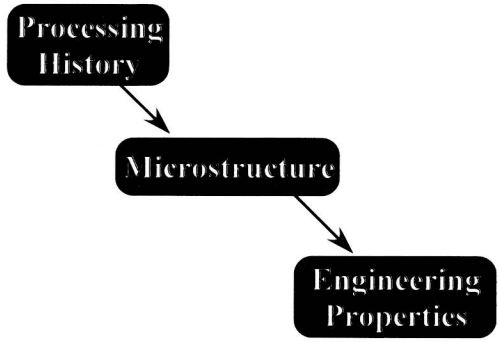

Figure 1.1 *The microstructure of an engineering material is a result of its chemical composition and processing history. The microstructure determines the chemical, physical and mechanical properties of the material, and hence limits its engineering performance.*

satisfied with the microanalytical identification of the chemical elements present in a specific microstructural feature, but rather we shall seek to determine the local chemical composition through microanalysis. Throughout the text we shall attempt to determine both the sensitivity of the methods described (the limits of detection) and their accuracy (the spatial or spectral resolution, or the concentration errors).

In general terms, microstructural characterization is achieved by allowing some form of probe to interact with a carefully prepared specimen sample. The most commonly used probes are visible light, X-ray radiation and a high energy electron beam. These three types of probe, taken in the same order, form the basis for optical microscopy, X-ray diffraction and electron microscopy. Once the probe has interacted with the sample, the scattered or excited signal is collected and processed into a form where it can be interpreted, either qualitatively or quantitatively. Thus, in microscopy, a two-dimensional image of the specimen is obtained, while in microanalysis a *spectrum* is collected in which the signal intensity is recorded as a function of either its energy or wavelength. In diffraction the signal may be displayed as either a diffraction pattern or a diffraction spectrum.

All the instrumentation that is used to characterize materials microstructure includes components that have five distinct functions (Figure 1.2). First, the probe is generated by a

Table 1.1 *On the qualitative level, microstructural characterization is concerned with the identification of the phases present, their morphology (size and shape), and the identification of the chemical constituents in each phase. At the quantitative level, it is possible to determine the atomic arrangements (applied crystallography), the spatial relationships between microstructural features (stereology), and the microchemical composition (microanalysis).*

Qualitative analysis	Phase identification	Microstructural morphology	Microchemical identification
Quantitative analysis	Applied crystallography	Stereology	Microanalysis

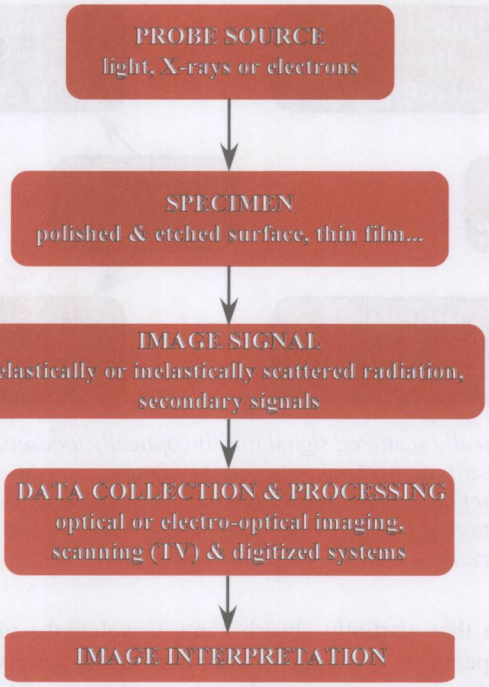

Figure 1.2 *Microstructural characterization relies on the interaction of a material sample with a* probe. *The probe is usually visible light, X-rays or a beam of high energy electrons. The resultant signal must be collected and interpreted. If the signal is elastically scattered an image can be formed by an optical system. If the signal is inelastically scattered, or generated by secondary emission the image is formed by a scanning raster (as in a television monitor).*

source that is filtered and collimated to provide a well-defined beam of known energy or wavelength. This probe beam then interacts with a prepared sample mounted on a suitable object stage. The signal generated by the interaction between the probe and the sample then passes through an optical system to reach the image plane, where the signal data are collected and stored. Finally, the stored data are read out, processed and recorded, either as a final image, or as diffraction data, or as a chemical record (for example, a composition map). The results then have to be interpreted!

In all the methods of characterization which we shall discuss, two forms of interaction between the probe and the specimen occur (Figure 1.3):

1. *Elastic scattering*, which is responsible for the intensity peaks in X-ray diffraction spectra that are characteristic of the phases present and their orientation in the sample. Elastic scattering also leads to diffraction contrast in transmission electron microscopy (TEM), where it is directly related to the nature of the crystal lattice defects present in the sample (grain boundaries, dislocations and other microstructural features).
2. *Inelastic scattering*, in which the energy in the probe is degraded and partially converted into other forms of signal. In optical microscopy, microstructural features may be