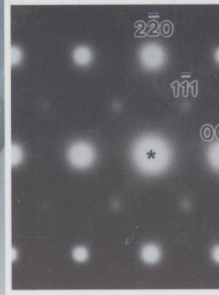
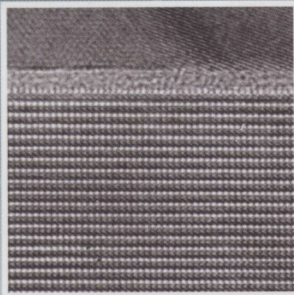
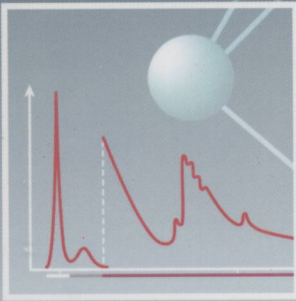


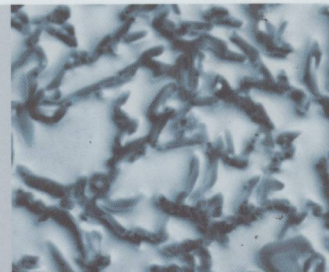
David Brandon and Wayne D. Kaplan

Microstructural Characterization of Materials



Second Edition

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Microstructural Characterization of Materials

2nd Edition

DAVID BRANDON AND WAYNE D. KAPLAN

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Microstructural Characterization of Materials

2nd Edition

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

Preface to the Second Edition	xi
Preface to the First Edition	xiii
1 The Concept of Microstructure	1
1.1 Microstructural Features	7
1.1.1 Structure–Property Relationships	7
1.1.2 Microstructural Scale	10
1.1.3 Microstructural Parameters	19
1.2 Crystallography and Crystal Structure	24
1.2.1 Interatomic Bonding in Solids	25
1.2.2 Crystalline and Amorphous Phases	30
1.2.3 The Crystal Lattice	30
Summary	42
Bibliography	46
Worked Examples	46
Problems	51
2 Diffraction Analysis of Crystal Structure	55
2.1 Scattering of Radiation by Crystals	56
2.1.1 The Laue Equations and Bragg’s Law	56
2.1.2 Allowed and Forbidden Reflections	59
2.2 Reciprocal Space	60
2.2.1 The Limiting Sphere Construction	60
2.2.2 Vector Representation of Bragg’s Law	61
2.2.3 The Reciprocal Lattice	61
2.3 X-Ray Diffraction Methods	63
2.3.1 The X-Ray Diffractometer	67
2.3.2 Powder Diffraction–Particles and Polycrystals	73
2.3.3 Single Crystal Laue Diffraction	76
2.3.4 Rotating Single Crystal Methods	78
2.4 Diffraction Analysis	79
2.4.1 Atomic Scattering Factors	80
2.4.2 Scattering by the Unit Cell	81
2.4.3 The Structure Factor in the Complex Plane	83
2.4.4 Interpretation of Diffracted Intensities	84
2.4.5 Errors and Assumptions	85
2.5 Electron Diffraction	90
2.5.1 Wave Properties of Electrons	91

2.5.2	Ring Patterns, Spot Patterns and Laue Zones	94
2.5.3	Kikuchi Patterns and Their Interpretation	96
	Summary	98
	Bibliography	103
	Worked Examples	103
	Problems	114
3	Optical Microscopy	123
3.1	Geometrical Optics	125
3.1.1	Optical Image Formation	125
3.1.2	Resolution in the Optical Microscope	130
3.1.3	Depth of Field and Depth of Focus	133
3.2	Construction of The Microscope	134
3.2.1	Light Sources and Condenser Systems	134
3.2.2	The Specimen Stage	136
3.2.3	Selection of Objective Lenses	136
3.2.4	Image Observation and Recording	139
3.3	Specimen Preparation	143
3.3.1	Sampling and Sectioning	143
3.3.2	Mounting and Grinding	144
3.3.3	Polishing and Etching Methods	145
3.4	Image Contrast	148
3.4.1	Reflection and Absorption of Light	149
3.4.2	Bright-Field and Dark-Field Image Contrast	150
3.4.3	Confocal Microscopy	152
3.4.4	Interference Contrast and Interference Microscopy	152
3.4.5	Optical Anisotropy and Polarized Light	157
3.4.6	Phase Contrast Microscopy	163
3.5	Working with Digital Images	165
3.5.1	Data Collection and The Optical System	165
3.5.2	Data Processing and Analysis	165
3.5.3	Data Storage and Presentation	166
3.5.4	Dynamic Range and Digital Storage	167
3.6	Resolution, Contrast and Image Interpretation	170
	Summary	171
	Bibliography	173
	Worked Examples	173
	Problems	176
4	Transmission Electron Microscopy	179
4.1	Basic Principles	185
4.1.1	Wave Properties of Electrons	185
4.1.2	Resolution Limitations and Lens Aberrations	187
4.1.3	Comparative Performance of Transmission and Scanning Electron Microscopy	192

4.2	Specimen Preparation	194
4.2.1	Mechanical Thinning	195
4.2.2	Electrochemical Thinning	198
4.2.3	Ion Milling	199
4.2.4	Sputter Coating and Carbon Coating	201
4.2.5	Replica Methods	202
4.3	The Origin of Contrast	203
4.3.1	Mass-Thickness Contrast	205
4.3.2	Diffraction Contrast and Crystal Lattice Defects	205
4.3.3	Phase Contrast and Lattice Imaging	207
4.4	Kinematic Interpretation of Diffraction Contrast	213
4.4.1	Kinematic Theory of Electron Diffraction	213
4.4.2	The Amplitude-Phase Diagram	213
4.4.3	Contrast From Lattice Defects	215
4.4.4	Stacking Faults and Anti-Phase Boundaries	216
4.4.5	Edge and Screw Dislocations	218
4.4.6	Point Dilatations and Coherency Strains	219
4.5	Dynamic Diffraction and Absorption Effects	221
4.5.1	Stacking Faults Revisited	227
4.5.2	Quantitative Analysis of Contrast	230
4.6	Lattice Imaging at High Resolution	230
4.6.1	The Lattice Image and the Contrast Transfer Function	230
4.6.2	Computer Simulation of Lattice Images	231
4.6.3	Lattice Image Interpretation	232
4.7	Scanning Transmission Electron Microscopy	234
	Summary	236
	Bibliography	238
	Worked Examples	238
	Problems	247
5	Scanning Electron Microscopy	261
5.1	Components of The Scanning Electron Microscope	262
5.2	Electron Beam-Specimen Interactions	264
5.2.1	Beam-Focusing Conditions	265
5.2.2	Inelastic Scattering and Energy Losses	266
5.3	Electron Excitation of X-Rays	269
5.3.1	Characteristic X-Ray Images	271
5.4	Backscattered Electrons	277
5.4.1	Image Contrast in Backscattered Electron Images	279
5.5	Secondary Electron Emission	280
5.5.1	Factors Affecting Secondary Electron Emission	283
5.5.2	Secondary Electron Image Contrast	286
5.6	Alternative Imaging Modes	288
5.6.1	Cathodoluminescence	288
5.6.2	Electron Beam Induced Current	288
5.6.3	Orientation Imaging Microscopy	289

5.6.4	Electron Backscattered Diffraction Patterns	289
5.6.5	OIM Resolution and Sensitivity	291
5.6.6	Localized Preferred Orientation and Residual Stress	292
5.7	Specimen Preparation and Topology	294
5.7.1	Sputter Coating and Contrast Enhancement	295
5.7.2	Fractography and Failure Analysis	295
5.7.3	Stereoscopic Imaging	298
5.7.4	Parallax Measurements	298
5.8	Focused Ion Beam Microscopy	301
5.8.1	Principles of Operation and Microscope Construction	302
5.8.2	Ion Beam–Specimen Interactions	304
5.8.3	Dual-Beam FIB Systems	306
5.8.4	Machining and Deposition	306
5.8.5	TEM Specimen Preparation	310
5.8.6	Serial Sectioning	314
	Summary	315
	Bibliography	318
	Worked Examples	318
	Problems	326
6	Microanalysis in Electron Microscopy	333
6.1	X-Ray Microanalysis	334
6.1.1	Excitation of Characteristic X-Rays	334
6.1.2	Detection of Characteristic X-Rays	338
6.1.3	Quantitative Analysis of Composition	343
6.2	Electron Energy Loss Spectroscopy	357
6.2.1	The Electron Energy-Loss Spectrum	360
6.2.2	Limits of Detection and Resolution in EELS	361
6.2.3	Quantitative Electron Energy Loss Analysis	364
6.2.4	Near-Edge Fine Structure Information	365
6.2.5	Far-Edge Fine Structure Information	366
6.2.6	Energy-Filtered Transmission Electron Microscopy	367
	Summary	370
	Bibliography	375
	Worked Examples	375
	Problems	386
7	Scanning Probe Microscopy and Related Techniques	391
7.1	Surface Forces and Surface Morphology	392
7.1.1	Surface Forces and Their Origin	392
7.1.2	Surface Force Measurements	396
7.1.3	Surface Morphology: Atomic and Lattice Resolution	397
7.2	Scanning Probe Microscopes	400
7.2.1	Atomic Force Microscopy	403
7.2.2	Scanning Tunnelling Microscopy	410
7.3	Field-Ion Microscopy and Atom Probe Tomography	413

7.3.1	Identifying Atoms by Field Evaporation	414
7.3.2	The Atom Probe and Atom Probe Tomography	416
	Summary	417
	Bibliography	420
	Problems	420
8	Chemical Analysis of Surface Composition	423
8.1	X-Ray Photoelectron Spectroscopy	424
8.1.1	Depth Discrimination	426
8.1.2	Chemical Binding States	428
8.1.3	Instrumental Requirements	429
8.1.4	Applications	431
8.2	Auger Electron Spectroscopy	431
8.2.1	Spatial Resolution and Depth Discrimination	433
8.2.2	Recording and Presentation of Spectra	434
8.2.3	Identification of Chemical Binding States	435
8.2.4	Quantitative Auger Analysis	436
8.2.5	Depth Profiling	437
8.2.6	Auger Imaging	438
8.3	Secondary-Ion Mass Spectrometry	440
8.3.1	Sensitivity and Resolution	442
8.3.2	Calibration and Quantitative Analysis	444
8.3.3	SIMS Imaging	445
	Summary	446
	Bibliography	448
	Worked Examples	448
	Problems	453
9	Quantitative and Tomographic Analysis of Microstructure	457
9.1	Basic Stereological Concepts	458
9.1.1	Isotropy and Anisotropy	459
9.1.2	Homogeneity and Inhomogeneity	461
9.1.3	Sampling and Sectioning	463
9.1.4	Statistics and Probability	466
9.2	Accessible and Inaccessible Parameters	467
9.2.1	Accessible Parameters	468
9.2.2	Inaccessible Parameters	476
9.3	Optimizing Accuracy	481
9.3.1	Sample Size and Counting Time	483
9.3.2	Resolution and Detection Errors	485
9.3.3	Sample Thickness Corrections	487
9.3.4	Observer Bias	489
9.3.5	Dislocation Density Revisited	490
9.4	Automated Image Analysis	491
9.4.1	Digital Image Recording	494
9.4.2	Statistical Significance and Microstructural Relevance	495

9.5	Tomography and Three-Dimensional Reconstruction	495
9.5.1	Presentation of Tomographic Data	496
9.5.2	Methods of Serial Sectioning	498
9.5.3	Three-Dimensional Reconstruction	499
	Summary	500
	Bibliography	503
	Worked Examples	503
	Problems	514
Appendices		517
Appendix 1:	Useful Equations	517
	Interplanar Spacings	517
	Unit Cell Volumes	518
	Interplanar Angles	518
	Direction Perpendicular to a Crystal Plane	519
	Hexagonal Unit Cells	520
	The Zone Axis of Two Planes in the Hexagonal System	521
Appendix 2:	Wavelengths	521
	Relativistic Electron Wavelengths	521
	X-Ray Wavelengths for Typical X-Ray Sources	521
Index		523

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

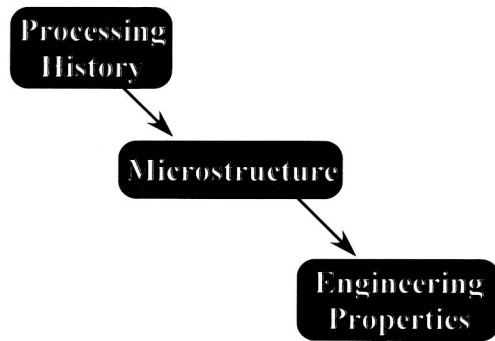


Figure 1.1 The microstructure of an engineering material is a result of its chemical composition and processing history. The microstructure determines the chemical, physical and mechanical properties of the material, and hence limits its engineering performance.

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

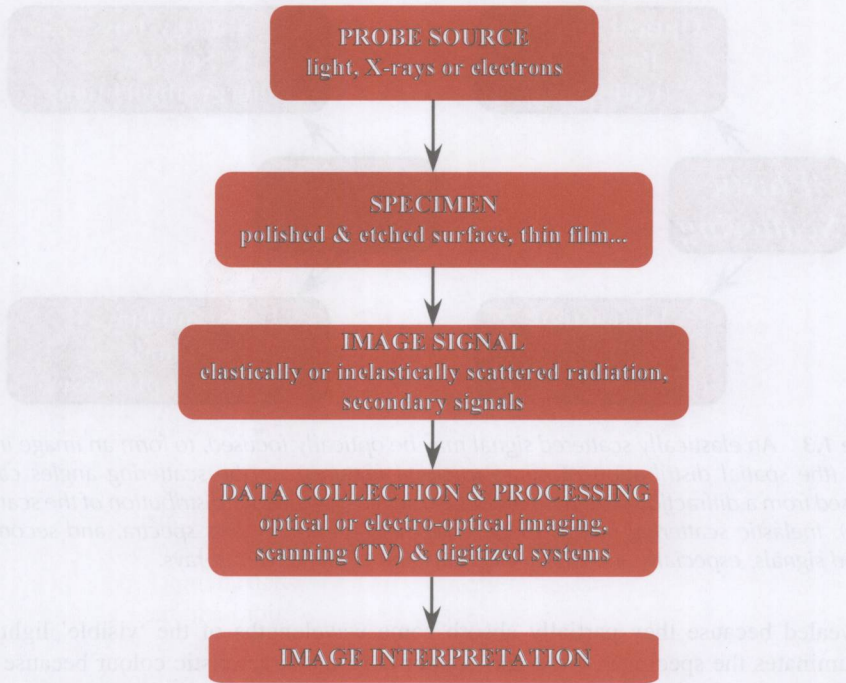


Figure 1.2 Microstructural characterization relies on the interaction of a material sample with a probe. The probe is usually visible light, X-rays or a beam of high energy electrons. The resultant signal must be collected and interpreted. If the signal is elastically scattered an image can be formed 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