A Beginners' Guide to Scanning Electron Microscopy

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To my wife

Preface

The ability of the scanning electron microscope (SEM) to characterize materials has increased tremendously since its inception on a commercial basis at Cambridge, United Kingdom, in 1965. The tremendous prospects offered by this invention have been consistently built upon, thanks to steady advances in instrumentation and computer technology in the past few decades. Presently, surface morphology of materials ranging from biological, polymers, alloys to minerals, ceramics, and corrosion deposits is routinely studied from micrometer to nanometer scale. The SEM has emerged as a vital, powerful, and versatile tool in the advancement of modern day nanotechnology by contributing to the area of characterization of nanostructured materials. Its ease of use, typically prompt sample preparation and straightforward image interpretation combined with high resolution and high depth of field as well as the ability to undertake microchemical and crystallographic analysis, has made it one of the most popular techniques used for characterization. Presently, the SEM is being used by professionals with a diverse technical background, such as life science, materials science, engineering, forensics, and mineralogy, in various sectors of the government, industry, and academia.

A significant number of in-depth and specialized accounts of the scanning electron microscopy are available to interested readers. This book is meant to serve as a concise and brief guide to the practice of scanning electron microscopy. In this treatment, the material has been developed with the goal of providing an easily understood text for those SEM users who have little or no background in this area. It provides a solid introduction to the subject for the uninitiated. The instrumentation and working and image interpretation have been explained in a succinct practical guide to the SEM. The aim is to provide all useful information regarding SEM operation, applications, and sample preparation to the readers without them having to go through extensive reference material. Essential theory of specimen-beam interaction and image formation is treated in a manner that can be effortlessly comprehended by the readers. The SEM technique is described in simple terms to help operators and users of the SEM to get the best imaging results possible for their materials of interest. The capabilities and limitations of the SEM are also described to enable students, engineers, and materials scientists to identify and apply this technique for their work.

Necessary background to the SEM is developed in Chap. 1. Primary and secondary components of the instrument are introduced in Chap. 2. Basic concepts of electron beam-specimen interaction and contrast formation are described in Chap. 3. Chapter 4 elaborates on the mechanisms of image formation in the SEM. The working of the SEM is introduced, and the factors affecting the quality of images are discussed. Specialized SEM techniques are described briefly in Chap. 5. Chapters 6 and 7 elaborate the characteristics of x-rays and principles of EDS/WDS microchemical analysis, respectively. Chapter 8 includes sample preparation techniques used for various classes of materials. Images, illustrations, and photographs are used to explain concepts, provide information, and aid in data interpretation. The effect of various imaging conditions on the quality of images is described to help users get the best results for their materials of interest. The book is structured in a way that can help a novice find necessary information quickly.

The support of the King Fahd University of Petroleum & Minerals (KFUPM), Dhahran, through project number BW161001 is gratefully acknowledged. I am utterly indebted to Mr. Abuduliken Bake for drawing with great skill almost all of the illustrations appearing in this book. In addition, he has taken a number of SEM images and photographs. His help has been instrumental in the timely completion of the manuscript. I am also grateful to personnel working in various organizations who permitted the use of relevant material. I especially thank Mr. Tan Teck Siong from JEOL Asia Pte Ltd. for providing me with a number of wonderful images. I also extend my appreciation to my colleagues at the Materials Characterization Laboratory, Center for Engineering Research, KFUPM, for their continued support.

In the end, I am grateful to have been blessed with family and friends who make life truly worthwhile.

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Contents

Abbreviations

BSE	Backscattered electron(s)
CAD	Computer-aided design
CCD	Charge-coupled device
CL	Cathodoluminescence
DBS detector	Distributed backscattered detector
E-beam lithography	Electron beam lithography
EBID	Electron beam-induced deposition
EBSD	Electron backscatter diffraction
EBSP	Electron backscatter pattern
EDS	Energy dispersive x-ray spectroscopy
EPMA	Electron probe microanalyzer
EsB detector	Energy selective Backscatter detector
ESEM	Environmental SEM
E-T detector	Everhart-Thornley detector
FEG	Field emission gun
FET	Field-effect transistor
FIB	Focused ion beam
FWHM	Full width at half maximum
FWTM	Full width at tenth maximum
GFIS	Gas field ion source
GPL	Gas path length
GSED	Gaseous secondary electron detector
GUI	Graphical user interface
HAADF	High-angle annular dark field
HSQ	Hydrogen silsequioxane
IBID	Ion beam-induced deposition
ICC	Incomplete charge collection
IPF map	Inverse pole figure map
LaB_6	Lanthanum hexaboride
LABe detector	Low-angle backscattered electron detector
LG	Light guide
LMIS	Liquid metal ion source
LV	Low vacuum

MCA	Multichannel x-ray analyzer
MCP	Microchannel plate
MDL	Minimum detectability limit
MFP	Mean free path
OM	Orientation map
PHA	Pulse-height analyzer
PLA	Pressure-limiting aperture
PMMA	Poly methyl methacrylate
PMT	Photomultiplier tube
Pre-Amp	Preamplifier
SACP	Selected area channeling pattern
SDD	Silicon drift detector
SE	Secondary electron(s)
SEM	Scanning electron microscope
Si(Li)	Lithium-drifted silicon
SNR	Signal-to-noise ratio
STEM	Scanning transmission electron microscope
t-EBSD	Transmission EBSD
TEM	Transmission electron microscope
TES	Transition edge x-ray sensor
TKD	Transmission Kikuchi diffraction
TTL	Through-the-lens
UED	Upper electron detector
UTW	Ultra-thin window
VPS	Volume plasma sources
WD	Working distance
WDS	Wavelength dispersive x-ray spectroscopy
XRD	X-ray diffractometer

Symbols List

σ_A	Standard deviation
$\left(\frac{\mu}{\rho}\right)$	Mass absorption coefficient
C_i	Mass fraction of element i
n_B	Number of incident beam electrons
n_{SE}	Number of secondary electrons
p_g	Pressure of the gas
σ_g	Total scattering cross section of gas molecule for electrons
ΔE	Energy spread
A	Area, Atomic weight
a, b, and c	Constants
A, C	Constants
A_A	Maximum intensity
B	Magnetic field
C	Contrast
C_c	Chromatic aberration coefficient
$C_{i\ (\mathrm{standard})}$	Weight percent concentration of element i in the standard
$C_{i\ (\mathrm{unknown})}$	Weight percent concentration of element i in unknown bulk specimen
C_s	Spherical aberration coefficient
C_Z	Z contrast
d	Escape depth of SE
d	Lattice plane spacing
d	Resolution
d	Beam diameter
d_0	Crossover diameter
d_A	Diameter of astigmatism disc
d_c	Diameter of the chromatic aberration disc
d_d	Diameter of diffraction disc
d_{opt}	Optimum probe diameter
d_p	Probe diameter
$d_{p,min}$	Minimum probe size
d_s	Diameter of the spherical aberration disc

E	Energy of the x-ray line
e	Electric charge in Coulomb
E	Kinetic energy
E_0	Incident beam energy
E_A	Average energy for the x-ray peak
E_{BSE}	Energy of the BSE
E_c	Critical energy of ionization
E_{exc}	Mean energy per excitation
E_K	Binding energy of K shell
E_L	Binding energy of L shell
E_m	Mean number of electron-hole pairs
E_v	Continuum x-ray photon energy at some point in the spectrum
F	Force
h	Planck's constant
I	Intensity of x-ray photons when leaving the specimen surface
I	Width of the intrinsic line of the detector
I_0	Original intensity of x-ray photons
i_B	Electron beam current entering the specimen
i_b	Beam current
I_b	Background intensity
i_{BSE}	Backscattered electron current moving out of the specimen
I_{cm}	Intensity of continuum x-ray
i_e	Emission current
i_f	Filament heating current
$I_{i\ (standard)}$	Intensity of characteristic x-ray peak emanating from element i in the standard
$I_{i\ (unknown)}$	Intensity of characteristic x-ray peak emanating from element i in unknown specimen
i_p	Probe current
$I_{p,max}$	Maximum probe current
IR	Infrared
J	Average loss in energy per event
J_b	Current density of electron beam
J_c	Current density of electron source
k	Boltzmann constant
LED	Light-emitting diode
$L_{monitor}$	Scan length on monitor
$L_{picture\ element}$	Length of picture element
L_{pixel}	Length of pixel
$L_{specimen}$	Scan length on specimen
m	Mass
n	Order of diffraction
n	Refractive index
N	Total number of atoms present in the irradiated volume

n	Total number of ionization events
nm	Nanometer
N_v	Avogadro's number
p	Probability
P	Quality indicator of the electronics used
Q	Cross section of ionization
R	Detector's energy resolution
R	Electron range
R	Extent of backscattering
r_s	Skirt radius
R_x	X-ray range
s	Distance travelled by an electron in the specimen
S	Stopping power
S_A	Signal emitted by the feature A
S_B	Signal emitted by the feature B
t	Thickness of specimen travelled
T	Absolute temperature (K)
t	Acquisition time
U	Overvoltage
v	Electromagnetic radiation frequency
v	Velocity of the particle
V_0	Accelerating voltage
W	Work function
X	The equivalent FWHM related to incomplete charge collection and leakage current of the detector
X_k	Number of emitted x-ray photons
Y	X-ray peak intensity
Z	Atomic number
α	Convergence angle of the beam
η	Backscatter coefficient
η_B	Number of incident beam electrons
η_{BSE}	Number of backscattered electrons
λ_{SWL}	Short wavelength limits
μ	Absorption coefficient
ρ	Density
Φ	Depth distribution function
φ	Work function
ψ	Take-off angle
ω	Fluorescence yield
Ω	Solid angle
α_{opt}	Optimum convergence angle
β	Brightness of gun
β_{max}	Maximum brightness of gun
δ	Secondary yield

λ	Wavelength, mean free path (escape depth of SE)
μm	Micron
θ	Bragg angle of diffraction
F	Frame scan time
q	Detector efficiency