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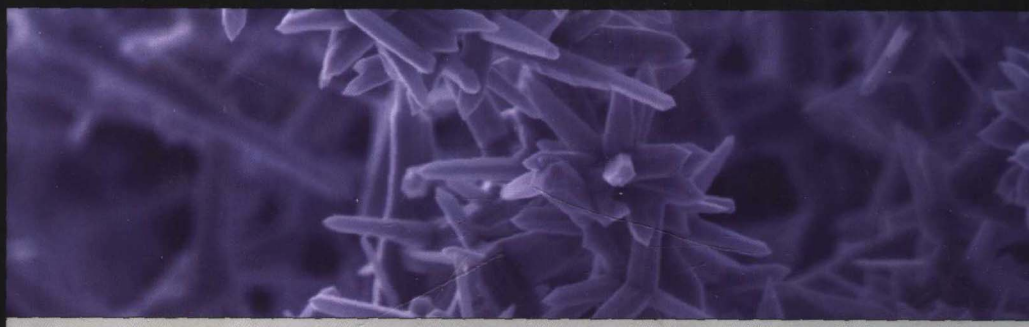
Advanced Scanning Microscopy for Nanotechnology

Techniques and Applications

扫描电子显微学及在纳米技术中的应用



Edited by Weilie Zhou and Zhong Lin Wang

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前　言

在过去的10年中，纳米技术的飞速发展使得扫描电子显微镜成为了一种分析和构建新纳米材料、结构和器件不可缺少的有力的工具。新纳米材料的发现需要通过先进的分析技术和技能来获取高质量的图片，从而帮助我们理解纳米结构，以达到改进合成方法和提高性能的目的。例如场发射枪、背散射电子的探测、X射线元素的图像化等，已经很大程度地提高了扫描电子显微镜在纳米材料分析中的应用。除了分析功能之外，扫描电子显微镜可以与最新发展起来的测控技术相结合，实行原位纳米器件的加工、制造和性能表征。这些技术包括纳米材料的操控、电子刻蚀、聚焦离子束微加工等。虽然这些技术仍在发展之中，但它们已开始广泛应用于纳米研究的各个领域。本书将重点介绍这些新发展起来的技术及其应用。

本书包含扫描电子显微学基础理论、近年发展起来的背散射电子衍射、复杂X射线分析、低电压图像获取、生物材料环境扫描电子显微观察、电子刻蚀和聚焦离子束微加工。这些章节将介绍如何运用这些技术来实施纳米材料及其结构的表征和制备。

此外本书还将讨论这些先进扫描电子显微学技术在纳米线和纳米管、光子晶体和器件、纳米颗粒和胶体粒子的组装、模板法制取纳米结构板块、一维纤锌矿半导体纳米结构、生物相关的纳米材料、纳米材料和结构的原位操控以及冷台在纳米结构观察中的应用。这些技术正被广泛用于纳米材料和结构的制造及加工中。

本书的特点是各个章节都是由本领域里世界级资深的研究组所撰写。其他贡献者则是来自这些技术和设备的国际知名供应商中的技术应用专家。本书不仅可供纳米材料研究者使用，同时也是一本面向高校师生和扫描电子显微镜工作者的教材和参考书。

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Preface

Advances in nanotechnology over the past decade have made scanning electron microscopy (SEM) an indispensable and powerful tool for analyzing and constructing new nanomaterials. Development of nanomaterials requires advanced techniques and skills to attain higher quality images, understand nanostructures, and improve synthesis strategies. A number of advancements for SEM such as, field emission guns, electron back scatter detection (EBSD), and X-ray element mapping, have improved nanomaterials analysis. In addition to being an analysis tool, SEM can be integrated with the latest technology to perform *in-situ* nanomaterial engineering and fabrication. Some examples of this integrated technology include nanomanipulation, electron beam nanolithography, and focused ion beam (FIB) techniques. Although these techniques are still being developed, they are widely applied in every aspect of nanomaterial research. This book will introduce some of the new advancements in SEM techniques and demonstrate their possible applications.

The first section covers basic theory, newly developed EBSD techniques, advanced X-ray analysis, low voltage imaging, environmental microscopy for biomaterials observation, e-beam nanolithography patterning, FIB nanostructure fabrication, and scanning transmission electron microscopy (STEM). These chapters contain practical examples of how these techniques are used to characterize and fabricate nanomaterials and nanostructures.

The second section discusses the applications of these SEM based techniques, including nanowires and carbon nanotubes, photonic crystals and devices, nanoparticles and colloidal self-assembly, nano-building blocks fabricated through templates, one dimensional wurtzite semiconducting nanostructures, bio-inspired nanomaterials, *in-situ* nanomanipulation, and cry-temperature stage in nanostructure research. These applications are widely used in fabricating and engineering new nanomaterials and nanostructures.

The unique feature of this book is that it is written by experts from leading research groups, who specialize in the development of nanomaterials using these SEM based techniques. Additional contributions are made by application specialists from several popular instrument vendors concerning their techniques to characterize, engineer and manipulate nanomaterials *in-situ* SEM. This book should be a useful and practical guide for nanomaterial researchers, as well as a valuable reference book for students and SEM specialists.

Weilie Zhou and Zhong Lin Wang

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郑重声明

Contents

Chapter 1

Fundamentals of Scanning Electron Microscopy

Weilie Zhou, Robert P. Apkarian, Zhong Lin Wang, and David Joy

1.1 Introduction

The scanning electron microscope (SEM) is one of the most versatile instruments available for the examination and analysis of the microstructure morphology and chemical composition characterizations. It is desirable to understand several of the basic principles of light optics in order to understand the fundamentals of electron microscopy. The unaided eye can discriminate objects subtending about 1/60° visual angle, corresponding to a resolution of ~0.1 mm (at the optimum viewing distance of 25 cm). Optical microscopy has the limit of resolution of ~2,000 Å by enlarging the visual angle through optical lens. Light microscopy has been, and continues to be, of great importance to scientific research. Since the discovery that electrons can be deflected by the magnetic field in numerous experiments in 1890's[1], electron microscopy has been developed by replacing the light source with high energy electron beam. In this section, we will for a split second go over the theoretical basics of scanning electron microscopy including the resolution limitation, electron beam interactions with specimens, and signal generation.

1.1.1 Resolution and Abbe's equation

The limit of resolution is defined as the minimum distances by which two structures can be separated and still appear as two distinct objects. Ernst Abbe[1] proved that the limit of resolution depends on the wavelength of the illumination source. At certain wavelength, when resolution exceeds the limit, the magnified image blurs.

Because of diffraction and interference, a point of light cannot be focused as a perfect dot. Instead, the image will have the appearance of a larger diameter than the source, consisting of a disc composed of concentric circles with diminishing intensity. This is known as an Airy disc and is represented in Fig. 1.1(a). The primary wavefront contains approximately 84% of the light energy, and the intensity of secondary and tertiary *etc.* wavefronts decay rapidly at higher

orders. Generally, the radius of Airy disc is defined as the distance between the first order peak and the first order trough, as shown in Fig. 1.1(a). When the center of two primary peaks are separated by a distance equal to the radius of Airy disc, the two objects can be distinguished from each other, as shown in Fig. 1.1(b). Resolution in a perfect optical system can be described mathematically by Abbe's equation. In this equation:

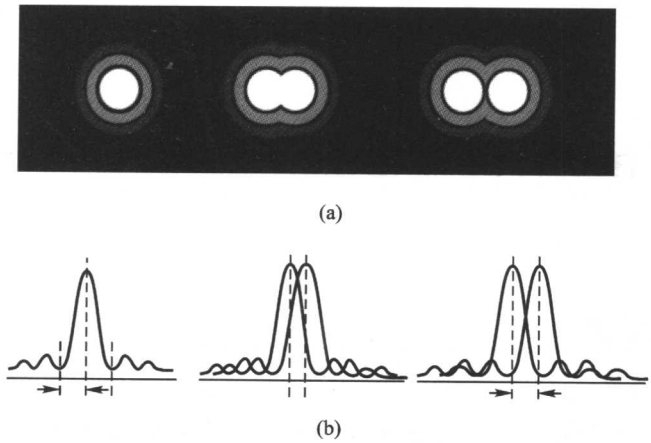

Fig. 1.1 Illustration of resolution in Airy disc (a) and wavefront(b).

$$d = 0.612\lambda / n\sin\alpha$$

where: d is resolution; λ is wavelength of imaging radiation; n is index of refraction of medium between point source and lens, relative to free space; α is half the angle of the cone of light from specimen plane accepted by the objective (half aperture angle in radians); $n\sin\alpha$ is often called NA (numerical aperture).

Substituting the illumination source and condenser lens with electron beam and electromagnetic coils in light microscopes respectively, the first transmission electron microscope (TEM) was constructed in 1930's[2], in which electron beam was focused by an electromagnetic condenser lens onto the specimen plane. The scanning electron microscope (SEM) utilizes a focused electron beam to scan across the surface of the specimen systematically, producing large numbers of signals, which will be discussed in detail later. These electron signals are eventually converted to a visual signal displayed on a cathode ray tube.

1.1.2 Interaction of electron with samples

Image formation in the SEM is dependent on the acquisition of signals produced from the electron beam and specimen interactions. These interactions can be divided into two major categories: elastic interactions and inelastic interactions. Elastic scattering results from the deflection of the incident electron by the specimen atomic nucleus or by outer shell electrons of similar energy. This kind of interaction is characterized by negligible energy loss during the collision and by a wide-angle directional change of the scattered electron. Incident electrons which are

elastically scattered through an angle of more than 90° are called backscattered electrons, and yield a useful signal for imaging the sample. Inelastic scattering occurs through a variety of interactions between the incident electrons and the electrons and atoms of the sample, and result in the primary beam electron transferring substantial energy to that atom. The amount of energy loss depends on whether the specimen electrons are excited singly or collectively and on the binding energy of the electron to the atom. As a result, the excitation of the specimen electrons during the ionization of specimen atoms leads to the generation of secondary electrons, which are conventionally defined as possessing energies of less than 50 eV and can be used to image or analyze the sample. In addition to those signals that are utilized to form an image, a number of other signals are also produced when an electron beam strikes a sample, including the emission of characteristic X-rays, Auger electrons, and cathodoluminescence. We will discuss these signals in the later sections. Fig. 1.2 shows the regions from which different signals are detected.

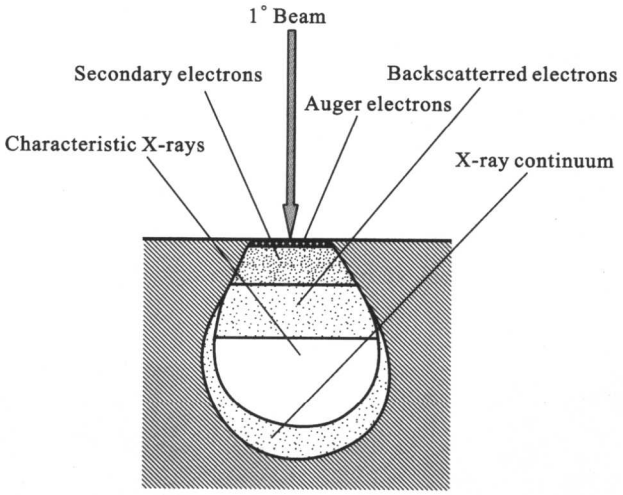

Fig. 1.2 Illustration of several signals generated by the electron beam-specimen interaction in the scanning electron microscope and the regions from which the signals can be detected.

In most cases when incident electrons strike the specimen surface, instead of being bounced off immediately, the energetic electrons penetrate into the sample for some distance before they encounter and collide with a specimen atom. In doing so the primary electron beam produces what is known as a region of primary excitation, from which a variety of signals are produced. The size and shape of this zone is largely dependent upon the beam electron energy and the atomic number, and hence the density, of the specimen. Fig. 1.3 illustrates the variation of interaction volume with respect to different accelerating voltage and atomic number. At certain accelerating voltage, the shape of interaction volume is "tear drop" for low atomic number specimen and hemi-sphere for specimens of high atomic number. The volume and depth of penetration increase with an increase of the beam energy and fall with the increasing specimen atomic number because specimens with higher atomic number have more particles to stop elec-