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国外物理名著系列 17

(影印版)

Fundamentals of  
Nanoscale Film Analysis

纳米薄膜分析基础

T.L.Alford L.C.Feldman J.W.Mayer



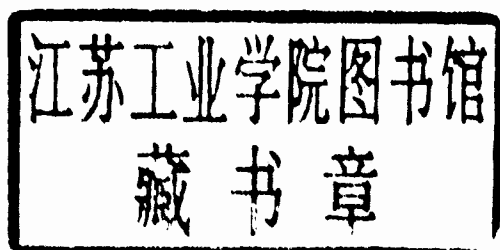
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## 国外物理名著系列序言

对于国内的物理学工作者和青年学生来讲,研读国外优秀的物理学著作是系统掌握物理学知识的一个重要手段。但是,在国内并不能及时、方便地买到国外的图书,且国外图书不菲的价格往往令国内的读者却步,因此,把国外的优秀物理原著引进到国内,让国内的读者能够方便地以较低的价格购买是一项意义深远的工作,将有助于国内物理学工作者和青年学生掌握国际物理学的前沿知识,进而推动我国物理学科研究和教学的发展。

为了满足国内读者对国外优秀物理学著作的需求,科学出版社启动了引进国外优秀著作的工作,出版社的这一举措得到了国内物理学界的积极响应和支持,很快成立了专家委员会,开展了选题的推荐和筛选工作,在出版社初选的书单基础上确定了第一批引进的项目,这些图书几乎涉及了近代物理学的所有领域,既有阐述学科基本理论的经典名著,也有反映某一学科专题前沿的专著。在选择图书时,专家委员会遵循了以下原则:基础理论方面的图书强调“经典”,选择了那些经得起时间检验、对物理学的发展产生重要影响、现在还不“过时”的著作(如:狄拉克的《量子力学原理》)。反映物理学某一领域进展的著作强调“前沿”和“热点”,根据国内物理学研究发展的实际情况,选择了能够体现相关学科最新进展,对有关方向的科研人员和研究生有重要参考价值的图书。这些图书都是最新版的,多数图书都是2000年以后出版的,还有相当一部分是2006年出版的新书。因此,这套丛书具有权威性、前瞻性和应用性强的特点。由于国外出版社的要求,科学出版社对部分图书进行了少量的翻译和注释(主要是目录标题和练习题),但这并不会影响图书“原汁原味”的感觉,可能还会方便国内读者的阅读和理解。

“他山之石,可以攻玉”,希望这套丛书的出版能够为国内物理学工作者和青年学生的工作和学习提供参考,也希望国内更多专家参与到这一工作中来,推荐更多的好书。

杨国桢

中国科学院院士  
中国物理学会理事长

To our wives and children,  
Katherine and Dylan,  
Betty, Greg, and Dana,  
and  
Betty, Jim, John, Frank, Helen, and Bill

# Preface

A major feature in the evolution of modern technologies is the important role of surfaces and near surfaces on the properties of materials. This is especially true at the nanometer scale. In this book, we focus on the fundamental physics underlying the techniques used to analyze surfaces and near surfaces. New analytical techniques are emerging to meet the technological requirements, and all are based on a few processes that govern the interactions of particles and radiation with matter. Ion implantation and pulsed electron beams and lasers are used to modify composition and structure. Thin films are deposited from a variety of sources. Epitaxial layers are grown from molecular beams and physical and chemical vapor techniques. Oxidation and catalytic reactions are studied under controlled conditions. The key to these methods has been the widespread availability of analytical techniques that are sensitive to the composition and structure of solids on the nanometer scale.

This book focuses on the physics underlying the techniques used to analyze the surface region of materials. This book also addresses the fundamentals of these processes. From an understanding of processes that determine the energies and intensities of the emitted energetic particles and/or photons, the application to materials analysis follows directly.

Modern materials analysis techniques are based on the interaction of solids with interrogating beams of energetic particles or electromagnetic radiation. These interactions and their resulting radiation/particles are based upon on fundamental physics. Detection of emergent radiation and energetic particles provides information about the solid's composition and structure. Identification of elements is based on the energy of the emergent radiation/particle; atomic concentration is based on the intensity of the emergent radiation. We discuss in detail the relevant analytical techniques used to uncover this information. Coulomb scattering from atoms (Rutherford backscattering spectrometry), the formation of inner shell vacancies in the electronic structure (X-ray photoelectron spectroscopy), transitions between levels (electron microprobe and Auger electron spectroscopies), and coherent scattering (X-ray and electron diffractometry) are fundamental to materials analysis. Composition depth profiles are obtained with heavy-ion sputtering in combination with surface-sensitive techniques (electron spectroscopies and secondary ion mass spectrometry). Depth profiles are also found from energy loss of light ions (Rutherford backscattering and prompt nuclear analyses). Structures of surface layers are characterized using diffraction (X-ray, electron,

and low-energy electron diffraction), elastic scattering (ion channeling), and scanning probes (tunneling and atomic force microscopies).

Because this book focuses on the fundamentals of modern surface analysis at the nanometer scale, we have provided derivations of the basic parameters—energy and cross section or transition probability. The book is organized so that we start with the classical concepts of atomic collisions as applied to Rutherford scattering (Chapter 2), energy loss (Chapter 3), sputtering (Chapter 4), channeling (Chapter 5), and electron interactions (Chapter 6). An overview is given of diffraction techniques in both real space (X-ray diffraction, Chapter 7) and reciprocal space (electron diffraction, Chapter 8) for structural analysis. Wave mechanics is required for an understanding of photoelectric cross sections and fluorescence yields; we review the wave equation and perturbation theory in Chapter 9. We use these relations to discuss photoelectron spectroscopy (Chapter 10), radiative transitions (Chapter 11), and nonradiative transitions (Chapter 12). Chapter 13 discusses the application of nuclear techniques to thin film analysis. Finally, Chapter 14 presents a discussion of scanning probe microscopy.

The current volume is a significant expansion of the previous work, *Fundamentals of Thin Films Analysis*, by Feldman and Mayer. New chapters have been added reflecting the progress that has been made in analysis of ultra thin films and nanoscale structures.

All the authors have been engaged heavily in research programs centered on materials analysis; we realize the need for a comprehensive treatment of the analytical techniques used in nanoscale surface and thin film analysis. We find that a basic understanding of the processes is important in a field that is rapidly changing. Instruments may change, but the fundamental processes will remain the same.

This book is written for materials scientists and engineers interested in the use of spectroscopies and/or spectrometries for sample characterization; for materials analysts who need information on techniques that are available outside their laboratory; and particularly for seniors and graduate students who will use this new generation of analytical techniques in their research.

We have used the material in this book in senior/graduate-level courses at Cornell University, Vanderbilt University, and Arizona State University, as well as in short courses for scientists and engineers in industry around the world. We wish to thank Dr. N. David Theodore for his review of Chapters 7 and 8. We also thank Timothy Pennycook for proofreading the manuscript. We thank Jane Jorgensen and Ali Avcisoy for their drawings and artwork.

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# 1

## An Overview: Concepts, Units, and the Bohr Atom

### 1.1 Introduction

Our understanding of the structure of atoms and atomic nuclei is based on scattering experiments. Such experiments determine the interaction of a beam of elementary particles—photons, electrons, neutrons, ions, etc.—with the atom or nucleus of a known element. (In this context, we consider all incident radiation as *particles*, including photons.) The classical example is Rutherford scattering, in which the scattering of incident alpha particles from a thin solid foil confirmed the picture of an atom as composed of a small positively charged nucleus surrounded by electrons in circular orbits. As these fundamental interactions became understood, the scientific community recognized the importance of the inverse process—namely, measuring the interaction of radiation with targets of unknown elements to determine atomic composition. Such determinations are called *materials analysis*. For example, alpha particles scatter from different nuclei in a distinct and well-understood manner. Measurements of the intensity and energy of the scattered particles provides a direct measure of elemental composition. The emphasis in this book is twofold: (1) to describe in a quantitative fashion those fundamental interactions that are used in modern materials analysis and (2) to illustrate the use of this understanding in practical materials analysis problems.

The emphasis in modern materials analysis is generally directed toward the structure and composition of the surface and outer few tens to hundred nanometers of the materials. The emphasis comes from the realization that the surface and near-surface regions control many of the mechanical and chemical properties of solids: corrosion, friction, wear, adhesion, and fracture. In addition, one can tailor the composition and structure of the outer layers by directed energy processes utilizing lasers or electron and ion beams, as well as by more conventional techniques such as oxidation and diffusion.

In modern materials analysis, one is concerned with the source beam (also referred to as the *incident beam* or the *probe beam* or *primary beam*) of radiation; the beam of particles—photons, electrons, neutrons, or ions; the interaction cross section; the emergent radiation; and the detection system. The primary interest of this book is the interaction of the beam with the material to be analyzed, with emphasis on the energies and intensities of emitted radiation. As we will show, the energy of the emitted particles provides the signature or identification of the atom, and the intensity tells the amount

of atoms (i.e., sample composition). The radiation source and the detection system are important topics in their own right; however, the main emphasis in this book is on the ability to conduct quantitative materials analysis that depends upon interactions within the target.

## 1.2 Nomenclature

Materials characterization involves the quantification of the structure, composition, amount, and depth distribution of matter with the use of energetic particles (e.g., ions, neutrons, alpha particles, protons, and electrons) and energetic photons (e.g., infrared radiation, visible light, UV light, X-rays, and gamma rays). Any materials-characterization techniques can be described in the following manner. The incident *probe beam* of energetic photons or particles *interrogates* the solid. The incident particle or photon reacts with the solid in various manners; these reactions ( $R_x$ ) induce the emission of a variety of detected beams in the form of energetic particles or photons, i.e., the detected beam (Fig. 1.1). Hence, the primary interest of this book is in using the reaction (between the beam and the solid) and the intensity and energy of the detected beam to analyze solids. Since the energy of the detected particle/photon is measured, the actual names of the various techniques have the prefix SPECTRO, meaning *energy measurement*. The suffix gives information about the relationship between the specific incidence *photon/particle* and the detected *photon/particle*. For example, if the incident species is the same as the emitted species, the technique is a SPECTROMETRY: Rutherford backscattering spectrometry and X-ray diffractometry. If the incident species is different from the emitted species, then the term SPECTROSCOPY is used: Auger electron spectroscopy and X-ray photoelectron spectroscopy.

There is an impressive array of experimental techniques available for the analysis of solids. Figure 1.2 gives the flavor of the possible combinations. In some cases, the same incident and emergent radiation is employed (we will use the general terms *radiation* and *particles* for photons, electrons, ions, etc.). Listed below are examples, with commonly used acronyms in parentheses.

Primary electron in, Auger electron out: Auger electron spectroscopy (AES)  
 Alpha particle in, alpha particle out: Rutherford backscattering spectrometry (RBS)  
 Primary X-ray in, characteristic X-ray out: X-ray fluorescence spectroscopy (XRF)

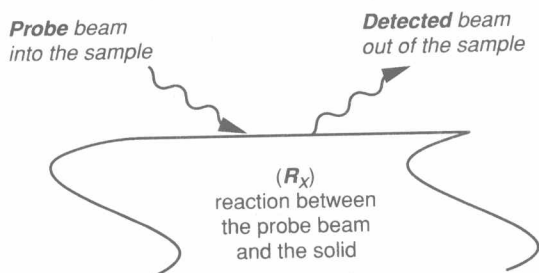


FIGURE 1.1. Schematic of the fundamentals of materials characterization. The probe beam of energetic photons or particles interrogates the solid. The incident particle or photon reacts ( $R_x$ ) and induces the emission of a variety of detected beams in the form of energetic particles or photons, i.e., the detected beam.

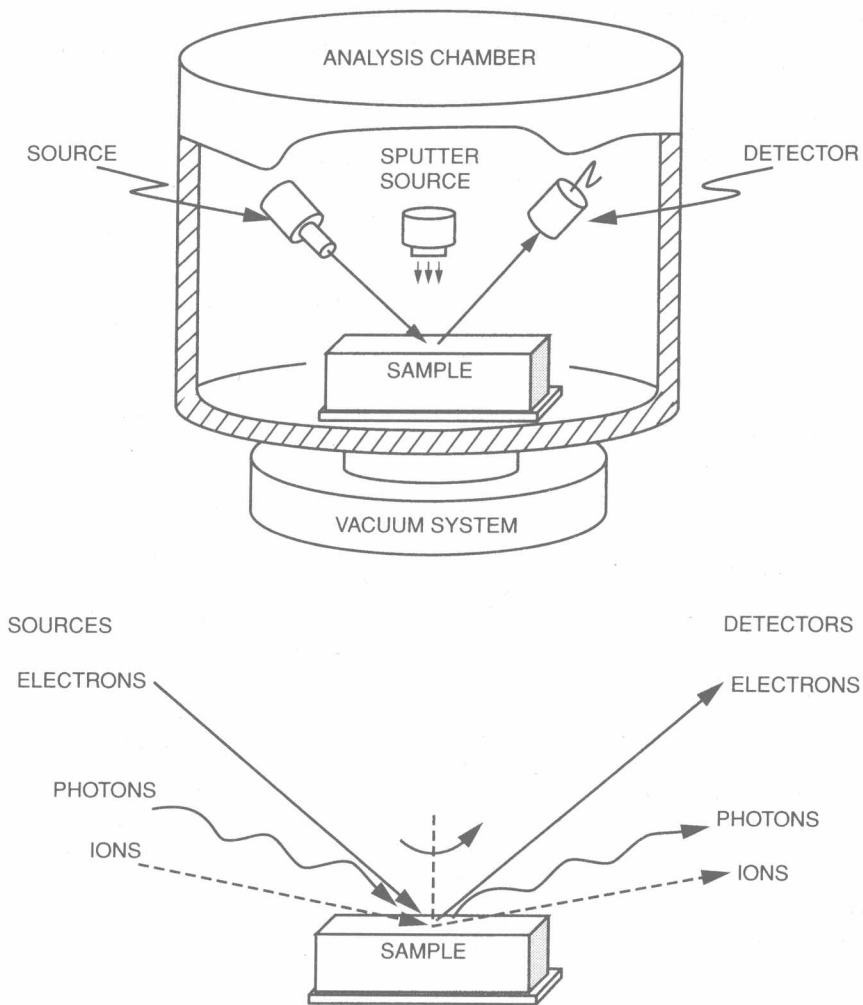


FIGURE 1.2. Schematic of radiation sources and detectors in thin film analysis techniques. Analytical probes are represented by almost any combination of source and detected radiation, i.e., photons in and photons out or ions in and photon out. Many chambers will also contain sample erosion facilities such as an ion sputtering as well as an evaporation apparatus for deposition of materials onto a clean substrate under vacuum.

In other cases, the incident and emergent radiation differ as indicated below:

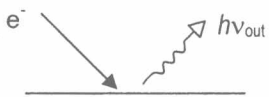
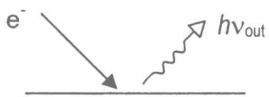
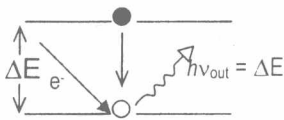
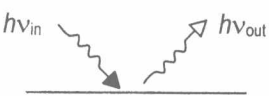
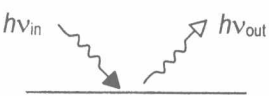
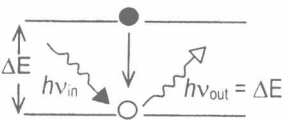
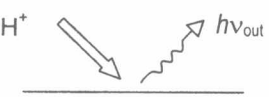
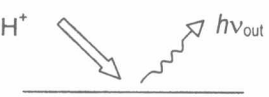
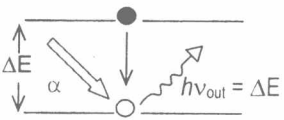
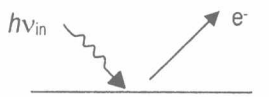
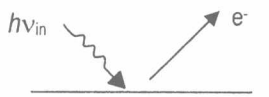
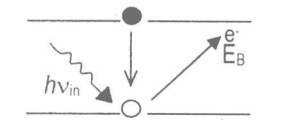
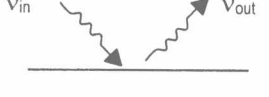
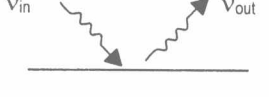






X-ray in, electron out: X-ray photoelectron spectroscopy (XPS)

Electron in, X-ray out: electron microprobe analysis (EMA)

Ion in, target ion out: secondary ion mass spectroscopy (SIMS)

A beam of particles incident on a target either scatters elastically or causes an electronic transition in an atom. The scattered particle or the energy of the emergent radiation contains the signature of the atom. The energy levels in the transition are

TABLE 1.1. Nomenclature of many techniques available for the analysis of materials. The name of a given technique often provides a complete or partial description of the technique.

Method	In	Out	$R_x$
Energy Dispersive X-rays (EDX) Spectroscopy			
X-ray Fluorescence (XRF) Spectroscopy			
Particle Induced X-ray Emission (PIXE) Spectroscopy			
X-ray Photoelectron Spectroscopy (XPS)			
X-ray Diffractometry (XRD)			Coherent Scattering $v_{in} = v_{out}$ (particle characteristics)
Electron Diffractometry (ED)			Coherent Scattering $v_{in} = v_{out}$ (wave characteristic)
Rutherford Backscattering Spectrometry (RBS)			Elastic Scattering
Secondary Ion Mass Spectroscopy (SIMS)			Sputtered Ion (erosion due to momentum transfer)

characteristic of a given atom; hence, measurement of the energy spectrum of the emergent radiation allows identification of the atom. Table 1.1 gives a summary of various techniques based on the nomenclature of the incident *probe beam*, the induced emission, and the detected beam.

The number of atoms per  $\text{cm}^2$  in a target is found from the relation between the number,  $I$ , of incident particles and the number of interactions. The term *cross section* is used as a quantitative measure of an interaction between an incident



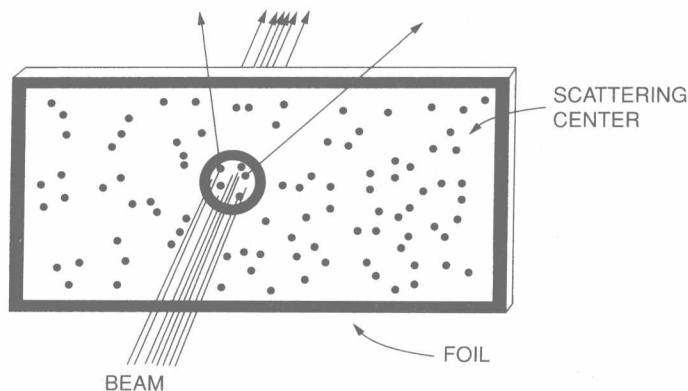


FIGURE 1.3. Illustration of the concept of cross section and scattering. The central circle defines a unit area of a foil containing a random array of scattering centers. In this example, there are five scattering centers per unit area. Each scattering center has an area (the cross section for scattering) of  $1/20$  unit area; therefore, the probability of scattering is  $5/20$ , or  $0.25$ . Then a fraction ( $0.25$  in this example) of the incident beam will be scattered, i.e., 2 out of 8 trajectories in the drawing. A measure of the fraction of the scattered beam is a measure of the probability ( $P = Nt\sigma$ , Eq. 1.1). If the foil thickness and density are known,  $Nt$  can be calculated, yielding a direct measure of the cross section.

particle and an atom. The cross section  $\sigma$  for a given process is defined through the probability,  $P$ :

$$P = \frac{\text{Number of interactions}}{\text{Number of incident particles}}. \quad (1.1)$$

For a target containing  $Nt$  atoms per unit area perpendicular to an incident beam of  $I$  particles, the number of interactions is  $I\sigma Nt$ . From knowledge of detection efficiency for measuring the emergent radiation containing the signature of the transition, the number of atoms and ultimately the target composition can be found (Fig. 1.3).

The information required from analytical techniques is the species identification, concentration, depth distribution, and structure. The available analytical techniques have different capabilities to meet these requirements. The choice of analysis method depends upon the nature of the problem. For example, chemical bonding information can be obtained from techniques that rely upon transitions in the electronic structure around the atoms—the electron spectroscopies. Structural determination is found from diffraction or particle channeling techniques.

In the following chapters, we are mostly concerned with materials analysis in the outer microns of the sample's surface and near-surface region. We emphasize the energy of the emergent radiation as an identification of the element and the intensity of the radiation as a measure of the amount of material. These are the basic principles that provide the foundation for the different analytical techniques.