

# **Applied Charged Particle Optics**

Edited by A. SEPTIER

Conservatoire National des Arts et Métiers Paris, France

PART B

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# **Applied Charged Particle Optics**

PART B

#### **Advances in Electronics and Electron Physics**

#### Edited by

# L. MARTON

#### CLAIRE MARTON

Smithsonian Institution Washington, D.C.

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#### List of Contributors

Numbers in parentheses indicate the pages on which authors' contributions begin.

- YVES BALLU (257), CEN-Saclay, Service de Physique Atomique, Section d'Etudes des Interactions Gaz-Solides, 91190 Gif-sur-Yvette, France
- P. H. DAWSON (173), Division of Physics, National Research Council, Ottawa, Ontario, Canada
- ERIC MUNRO\* (73), Engineering Department and Trinity College, University of Cambridge, Cambridge, England
- GEORGES SLODZIAN (1), Laboratoire de Physique des Solides, Université de Paris-Sud, Bâtiment 510, 91405 Orsay Cedex, France
- R. G. WILSON (45), Hughes Research Laboratories, Malibu, California 90265
- H. WOLLNIK (133), II. Physikalisches Institut, Justus Liebig-Universität, 6300 Giessen, West Germany

<sup>\*</sup>Present address: Physics Department, Imperial College of Science and Technology, London SW7 2BZ, England.

#### Foreword

We are most pleased to have as a supplement to Advances in Electronics and Electron Physics these volumes edited by Professor A. Septier on Applied Charged Particle Optics. No subject better represents the value of electron physics to modern technology. This point is well elaborated in Professor Septier's own preface. We, therefore, can only add our best wishes and our thanks to the Editor and contributors for their efforts in producing so valuable a reference work.

- L. MARTON
- C. MARTON

#### **Preface**

Charged particle optics is a relatively young subject, for it was in 1926 that Busch showed that an inhomogeneous axial magnetic field exerts a focusing action on an electron beam and is thus capable of bringing electrons from an "object" point together at an "image" point. Electrostatic "lenses" appeared on the scene a few years later, around 1931–1932, barely 50 years ago.

This discovery, that lenses for charged particles are in some sense analogous to the glass lenses used to focus light, stimulated the development of this new branch of optics. Within a few years, spectacular progress had been made, both in the theory and in practical applications, particularly in the design of cathode-ray tubes, mass spectrometers, and electron microscopes.

It is surprising that so many decades should have separated the period during which the free electron was discovered and that in which the first devices for focusing and analyzing beams of charged particles appeared, for the laws governing the action of electromagnetic fields on charged particles had been known since the nineteenth century. It was doubtless the work of Louis de Broglie in 1924–1925 on the wavelike nature of particles that attracted the attention of numerous physicists and reawakened interest in particles capable of traveling freely in a sufficiently good vacuum. The progress made during that period in vacuum technology was certainly not unrelated to the growth of interest in particle optics: a good vacuum is essential if the electric fields needed to give the particles their initial acceleration are not to break down, and the same is true of any electrostatic focusing or deflecting elements.

The theory advanced by L. de Broglie in 1924 was verified experimentally for the first time in 1927 by Davisson and Germer, who observed diffraction of electrons by the regular grating formed by the atoms of a crystal. The immense possibilities for exploring solids by means of electrons immediately became apparent, if transmission electron microscopes capable of a resolution 10<sup>3</sup> or 10<sup>4</sup> times better than the diffraction limit of the best optical microscopes could be constructed.

Apart from this particular point—the calculation of the theoretical resolving power of the electron microscope—all the optical properties of lenses and deflectors for charged particles were studied from the beginning without involving the wave aspect of the particles. Once the electrical potential and magnetic field distributions are known, particle trajectories

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can be calculated from which the essential properties of the optical systems can be established; the relevant quantities are defined by analogy with those familiar from the geometrical optics of centered light optical systems and prisms, including, in particular, their geometrical aberrations.

Several large industrial firms and numerous university laboratories became interested in developing high-magnification electron microscopes. This activity was not halted by the war, for new needs became apparent with the result that fresh aspects of particle optics were investigated. Thus the design of high-frequency tubes using intense electron beams and of isotope separators capable of yielding appreciable quantities of uranium 235 became possible only after new problems concerning the mutual interaction of charged particles had been solved; these effects had hitherto been neglected in devices using beams of low-current density.

By the early 1950s, the geometrical optics of optical systems with rotational symmetry or with a plane of symmetry had few secrets from the specialists in this field. Since everyone knew that the two fundamental aberrations of lenses or prisms—aperture aberration and chromatic aberration—could not be eliminated, unlike the case of glass lenses, systems with minimum aberrations were sought. With the gradual improvement of construction techniques, affecting both the mechanical parts and the electrical supplies, high-quality electron microscopes with a resolution better than 10 Å appeared on the market. These instruments provided not only a highly magnified image of an extremely thin object but also the diffraction pattern of the zone through which the electron beam passed.

New fields of application were emerging, however: large high-energy particle accelerators, which required the development of RF or microwave generators capable of furnishing several tens of megawatts in pulsed operation, and a new family of instruments for the quantitative analysis of solid specimens, which would thus go beyond mere qualitative observation. The first of these applications provoked a spurt of intense activity, devoted to strong focusing optical systems, as a result of which it became possible to accelerate and focus electron or proton beams to energies in the gigaelectron-volt range (1 GeV = 109 eV). During the same period, the 1950s, a new generation of instruments of more modest size was born, which were to revolutionize analytical techniques. The earliest of these was the electron microprobe, in which the characteristic X rays emitted by a solid bombarded with electrons were used to investigate the distribution of a particular chemical element at the surface of the solid; in the earliest models, the specimen was moved under a static probe, but the advantage of scanning the probe over a stationary specimen was soon recognized and the necessary scanning unit incorporated. The concentration of the element in question within a volume element of the order of 1 µm<sup>3</sup> could be estimated to a good approximation.

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The effort that went into producing electron microprobes—electron beams converging to a very small spot—naturally led to the commercial development of scanning microscopes, which gave an image of the surface of a massive specimen on a cathode-ray tube; the secondary electrons emitted by the specimen were used to create the signal, and the resulting image had almost unlimited depth of field and a resolution of the order of a few hundred angstroms.

Over the years, these microprobe instruments were improved, in order to reduce the spot size while keeping the probe current as high as possible. The brightness of the electron source, already known to be an important parameter in transmission electron microscopes, now became the vital quantity to be increased, and field emission tips began to be incorporated into microprobe instruments not long after the appearance of the equipment needed to provide the very high vacuum (UHV) required. These developments also acted as a stimulus in the design of energy analyzers and filters.

The constant efforts to ameliorate both the optical system and the technological aspects of the transmission electron microscope (TEM), associated with a better understanding of the way in which the image is formed, gradually pushed the resolution limit down to 2–3 Å with electrons of 100–200 keV. The age-old dream of seeing individual atoms, or at least an "image" of atoms, however imperfect, seemed to be on the brink of becoming a reality. In order to improve the resolution still further, and also to enable thicker specimens to be examined (of the order of micrometers in thickness, rather than a few hundred angstroms), very high-voltage microscopes were built (first at 1 MeV and later at 3 MeV). Innumerable problems, connected with the optical system, parasitic radiation, and mechanical vibration, had to be overcome before these cumbersome instruments reached practical resolutions in the angstrom range.

It was against this background that the idea of constructing a scanning microscope with a probe only a few angstroms in diameter provided by a field emission gun was born, in the mid-1960s. Such a microscope would have much the same resolution as a traditional TEM, for thin specimens, and the image would be displayed using information conveyed by the transmitted electrons. It was some years before the partisans of the TEM came to recognize the advantages of the scanning transmission electron microscope (STEM), but commercial models are now finding their way into many laboratories and a number of high-voltage STEMs are now being constructed or planned. It was in fact with a STEM that individual atoms of a specimen were first observed directly, in 1970, and similar TEM micrographs appeared shortly after, in 1971.

All these instruments—electron microscopes, both TEM and STEM, X-ray microanalyzers, particle accelerators at medium and high energies,

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mass spectrometers, and even their more humble relations such as television cameras and tubes—are indispensable tools, not only for physicists, chemists, and biologists in the field of pure research, but also for applied scientists and engineers striving to develop new materials and new devices for use in all the so-called "applied" sciences, electronics in particular. Numerous international congresses, and the spectacular results that have been obtained with such instruments, have made the latter familiar throughout the scientific world and indeed to an even wider public, thanks to popular texts and to television.

Although their merits are beyond discussion, these high-prestige instruments have somewhat obscured the existence of a wide variety of other devices using low-current beams of particles. These are used to obtain a better knowledge of the physical, electronic, or chemical properties of surfaces, for quantitative analysis of thin films or chemical compounds, for microfabrication of electronic components smaller than a micrometer in dimension, or in optoelectronics as image converters for images obtained with radiation imperceptible to the unaided eye. Likewise, turning to beams of high-power density, we draw attention to electron tubes for microwave generation already mentioned, electron beam welders, sources and injectors of electron and ion beams for accelerators, and the electron beams in the gigawatt range with which controlled fusion could be achieved.

All of these devices are the outcome of 50 years of research and effort toward what we might call the domestication of charged particles in the service of mankind.

A vast literature has grown up around the better-known charged particle instruments. In these new volumes, which may be regarded as a continuation of "Focusing of Charged Particles," Vols. 1 and 2 (A. Septier, ed., Academic Press, New York, 1967), we have tried to cover the many devices that have proved so useful in surface studies, in the analysis of solids and gases, and in the microfabrication of electronic components, but without going beyond the bounds of geometrical optics.

The object of the first three articles in Supplement 13A is to bring up to date the general methods, described in "Focusing of Charged Particles," for calculating the properties of optical systems or particle beams. In the first article, newly developed numerical methods of solving the equations of Laplace and Poisson are described, whereby potentials and magnetic field distributions can be calculated very accurately. Techniques for studying the trajectories and aberrations of optical systems are then considered, with particular attention given to the practical formulas from which they can be evaluated in concrete situations and to procedures for optimizing complex systems. In the second of this group of articles,

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the fundamental characteristics of real beams with nonvanishing emittance are examined, together with a critical account of the various diagnostic methods by which these characteristics can be investigated.

The last two articles in Supplement 13A and the first in Supplement 13B provide descriptions of ion microprobes operating in very different energy ranges and for quite different purposes. First, scanning transmission ion microscopes (STIM) are considered, a prototype of which is already functioning with 50-keV protons; these microscopes use point and field emission ion sources, which will shortly be ready for incorporation into any high-resolution ion microprobe system for the inspection or fabrication of microcircuits. The next article is devoted to ion microprobes, intended essentially for the local chemical analysis of solids using the X rays emitted when the specimen is bombarded with 2-3-MeV ions; this recent technique is more sensitive than its electron counterpart. Finally in this group, low-energy scanning and fixed-beam ion microprobes are considered: by analyzing the secondary ions emitted, the surface distribution of a particular element can be established with a resolution of the order of micrometers. We felt that it would be useful to compare the methods of obtaining the image and the performances of the two types of microprobe systems.

Two pieces of equipment used in the electronics industry for the production of components and in particular for microminiaturization are then described. First, ion implanters are considered, with which semiconductors can be locally doped; numerous problems concerning ion production, purification, and transport of the beam and focusing have arisen and the solutions are described. The various instruments, derived from the SEM, that use electron microprobes to etch electron-sensitive resins directly are then described; these are employed when the size of the components required is so small that masks can no longer be made. The scanning pattern of the probe is then controlled by computer.

In the three final articles of Supplement 13B, devices for analyzing mass or energy are described. Mass analysis is required when the substance to be analyzed, which may be a solid or a gas, is converted into ions, whereas energy analysis is required when an electron beam is used as an active element to excite or detect atoms or molecules at a surface or in a gas cell. Thus in the first of these articles, it seemed appropriate to describe recent progress in the field of mass spectrometers and isotope separators, particularly for applications involving radioactive substances with very short lifetimes, created artificially by bombarding a solid target with particles from an accelerator. For surface analysis by secondary ion mass spectrometry (SIMS) and the analysis of residual gases in ultrahigh vacuum chambers, quadrupole mass filters are now used extensively; the

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next article therefore describes the improvements that are continually being made to these, to provide even better resolution and transmission. Finally, in the last article, we turn to low-energy electron studies. Energy analysis of beams of slow electrons and the need to render such beams monochromatic have created a profusion of devices, each with its own particular domain of application. We therefore felt that a critical comparison of the properties of these analyzers would be welcomed by users confronted with a bewilderingly wide choice. Among the most common applications, we may mention Auger analysis, ESCA, and low-energy electron spectroscopy.

Not all devices using particle beams are discussed in these volumes but even so, the amount of material is too great to be contained in a single volume and we have therefore made a somewhat arbitrary division between Supplements 13A and B.

It is a pleasure to thank all the authors who have contributed articles for these volumes, despite all the other calls of a busy professional life on their time, and thus to share with the reader their personal reflections and the fruit of their wide experience.

A. SEPTIER

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### Microanalyzers Using Secondary Ion Emission

#### GEORGES SLODZIAN

Laboratoire de Physique des Solides Université de Paris-Sud Orsay Cedex, France

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#### I. INTRODUCTION

The analysis of a solid sample using mass spectrometry implies that we should have the means of extracting atoms from the target and ionizing them, before sending them into the spectrometer to be identified according to their mass. Extraction and ionization of atoms can be obtained simultaneously by cathodic sputtering. When a beam of particles, possessing several kiloelectron volts of energy, hits a target, it triggers off cascades of atomic collisions, which may result in the ejection of one or more atoms. Clusters of atoms, single, polyatomic, positive and negative ions, electrons, and photons are found among the particles emitted as well as single atoms. The ions emitted form the secondary ion emission that is used in mass spectrometry analysis. Although the angle and energy dispersions are quite large in the case of secondary ions, the maximum of the energy curve peaks