SECOND EDITION

Characterization of MATERIALS

Volume 1

EDITED BY
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CHARACTERIZATION OF MATERIALS

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CHARACTERIZATION OF MATERIALS

FOREWORD TO THE SECOND EDITION

The book you hold in your hands (or the web site you have navigated to) is a toolbox for the twenty-first century. Within this reference you will find starting points for understanding the materials that will create our future and play essential roles in solving many of the challenges we face.

While *Characterization of Materials* can be read to deepen one's understanding of analytical techniques, it was designed and written to be used. Because of this, it opens the door to comparisons and collaborations.

This is especially important as new knowledge and tomorrow's solutions are found, more and more, in the white spaces between disciplines. Materials science always has been interdisciplinary, taking advantage of discoveries and approaches in chemistry, physics, biology, engineering, or wherever the tools and concepts that can get the job done emerge.

The tutorial format—inventive among references—encourages exploration into unfamiliar ways to probe materials and learn their secrets, providing pointers to more in-depth studies of the methods introduced here.

This toolbox lays out the possibilities for the scientist or engineer who, more likely than not, is pushing the boundaries. These pages prompt questions, raise awareness, and, most of all, trigger conversations. As a researcher, faced with a challenge, searches to see which techniques might provide pertinent data, the answers found in these pages motivate him or her to seek colleagues who have the tools and the experience to do the needed tests or experiments. New relationships are formed; new discoveries are made; and science and engineering advance in unexpected ways.

This is all the more important since Materials Research, which, as recently at 1991, was dubbed an "orphan science" by presidential science advisor Dr. D. Allan Bromley, is now at the center of research activity. Nanotechnology, biomaterials, and biophysics have become the headline-grabbing disciplines of our era, as boundaries blur and startling new capabilities reach into our lives—based on our growing understanding of the characteristics of materials and how they might be manipulated.

Smart phones dazzle us as, with each new generation, they become more powerful (chips), more durable (metal bodies and gorilla glass), more colorful (rare earth displays), and more connected to their environments (sensors, GPS, and touchscreens). But new materials slip into our lives in more subtle ways.

The difference between a pair of eyeglasses made today and a pair made 50 years ago might not be obvious, even to the person wearing them. Two lenses fastened by metal wires, with some bits of plastic to make them comfortable as they rest in place. A closer look reveals more.

The frames may be made from a shape-memory alloy, so they will snap back into shape if they get bent. The lenses may be coated with scratch-resistant glass resin (polysiloxane polymer). The lenses themselves may be made with polymers designed to bend and focus the light—even emulating bifocals—more effectively than glass, while being lightweight. They also may be photochromic, capable of changing from clear to tinted and back again, thanks to organic dyes that break down in the presence of ultraviolet light and reform in its absence.

Every day, our lives are made better by materials that have been formed, tested, tuned, and manufactured, thanks to Materials Research. They surround us in packaging, paper, pharmaceuticals, engine components, electronic devices, paints, inks, clothing, and housewares. Materials researchers add color, durability, texture, safety, responsiveness, functionality, and affordability to the "stuff" our civilization depends upon.

I do not need to look beyond my own university to find examples. For instance, at Rensselaer, a team of researchers developed a coating that kills the lethal methicillin-resistant *Staphylococcus aureus* bacterium on contact. The new coating marries carbon nanotubes with lysostaphin, a naturally occurring enzyme that destroys the pathogen. It can be used directly or mixed with other materials, such as latex paint.

This is only the beginning of what materials can do in healthcare. We will see materials that will enable more resilient prosthetic devices with better biocompatibility. New probes will help us diagnose diseases, and medications will be formulated in ways that allow us to target cancer cells while sparing healthy tissue.

Another good example comes from the National Science Foundation (NSF) Nanoscale Science and Engineering Center, housed on our Troy campus, which allows biotechnology researchers to bring nano-capabilities to their work. Reaching across disciplines, the scientists and engineers working there already have had successes, such as the development of "nano-glues." With these glues, materials that normally do not stick together can be bonded by using a one-nanometer-high layer of self-assembling polymer chains. The extraordinarily thin "nano-glue," which gains strength as temperatures rise, could be a breakthrough for next-generation computer chips, and it also could be useful in such applications as high-temperature coatings.

Looking to the nanotechnology future, researchers have developed a new method of compacting carbon nanotubes into dense bundles that are efficient electrical conductors and could one day replace copper as the primary interconnects used on computer chips. They also may hasten the transition to next-generation 3D stacked chips.

These advances point to new frontiers in creating a smart environment, where stressed bridges will alert us to the need for repairs, where public areas will sniff out toxins and explosives, where optimized batteries will store electricity from sunlight and wind, and where threats to the food supply will be detected before people become ill.

For many years, materials research has been an essential, if often invisible, means of meeting the major challenges the world faces. We have refined our understanding of how to characterize and manipulate the properties of polymers, glasses, alloys, and biological tissues, and, without this knowledge, our ability to meet society's needs would be degraded. It would be fair to say that advanced materials are bound to be one of the enabling technologies in our quest for a sustainable energy and environmental future.

Of course, knowing and doing are two different things. For over a decade, *Characterization of Materials*, through its accessibility and transparent, user-friendly design, has helped scientists and engineers both to share knowledge and to work together for results. I am confident that this toolbox will continue to be shared, recommended, and used by generations of materials researchers who are bringing their talent and industry to the toughest problems and biggest opportunities that will emerge over the coming years. As they reach for this book, it will reward them with ideas, insights, options, contextual information, and, perhaps, occasional provocations. I am sure they will come to treasure what it has to offer as much as the many scientists and engineers who have opened its pages or come to its web site in the past.

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FOREWORD TO THE FIRST EDITION

Whatever standards may have been used for materials research in antiquity, when fabrication was regarded more as an art than a science and tended to be shrouded in secrecy, an abrupt change occurred with the systematic discovery of the chemical elements two centuries ago by Cavendish, Priestly, Lavoisier, and their numerous successors. This revolution was enhanced by the parallel development of electrochemistry and eventually capped by the consolidating work of Mendeleyev which led to the periodic chart of the elements.

The age of materials science and technology had finally begun. This does not mean that empirical or trial and error work was abandoned as unnecessary. But rather that a new attitude had entered the field. The diligent fabricator of materials would welcome the development of new tools that could advance his or her work whether exploratory or applied. For example, electrochemistry became an intimate part of the armature of materials technology.

Fortunately, the physicist as well as the chemist were able to offer new tools. Initially these included such matters as a vast improvement of the optical microscope, the development of the analytic spectroscope, the discovery of x-ray diffraction and the invention of the electron microscope. Moreover, many other items such as isotopic tracers, laser spectroscopes and magnetic resonance equipment eventually emerged and were found useful in their turn as the science of physics and the demands for better materials evolved.

Quite apart from being used to re-evaluate the basis for the properties of materials that had long been useful, the new approaches provided much more important dividends. The ever-expanding knowledge of chemistry made it possible not only to improve upon those properties by varying composition, structure and other factors in controlled amounts, but revealed the existence of completely new materials that frequently turned out to be exceedingly useful. The mechanical properties of relatively inexpensive steels were improved by the additions of silicon, an element which had been produced first as a chemist's oddity. More complex ferrosilicon alloys revolutionized the performance of electric transformers. A hitherto all but unknown element, tungsten, provided a long-term solution in the search for a durable filament for the incandescent lamp. Eventually the chemists were to emerge with valuable families of organic polymers that replaced many natural materials.

The successes that accompanied the new approach to materials research and development stimulated an entirely new spirit of invention. What had once been dreams, such as the invention of the automobile and the airplane, were transformed into reality, in part through the modification of old materials and in part by creation of new ones. The growth in basic understanding of electromagnetic phenomena, coupled with the discovery that some materials possessed special electrical properties, encouraged the development of new equipment for power conversion and new methods of long-distance communication with the use of wired or wireless systems. In brief, the successes derived from the new approach to the development of materials had the effect of stimulating attempts to achieve practical goals which had previously seemed beyond reach. The technical base of society was being shaken to its foundations. And the end is not yet in sight.

The process of fabricating special materials for well defined practical missions, such as the development of new inventions or improving old ones, has, and continues to have, its counterpart in exploratory research that is carried out primarily to expand the range of knowledge and properties of materials of various types. Such investigations began in the field of mineralogy somewhat before the age of modern chemistry and were stimulated by the fact that many common minerals display regular cleavage planes and may exhibit unusual optical properties, such as different indices of refraction in different directions. Studies of this type became much broader and more systematic, however, once the variety of sophisticated exploratory tools provided by chemistry and physics became available. Although the groups of individuals involved in this work tended to live somewhat apart from the technologists, it was inevitable that some of their discoveries would eventually prove to be very useful. Many examples can be given. In the 1870s a young investigator who was studying the electrical properties of a group of poorly conducting metal sulfides, today classed among the family of semiconductors, noted that his specimens seemed to exhibit a different electrical conductivity when the voltage was applied in opposite directions. Careful measurements at a later date demonstrated that specially prepared specimens of silicon displayed this rectifying effect to an even more marked degree. Another investigator discovered a family of crystals that displayed surface charges of opposite polarity when placed under unidirectional pressure, so called piezoelectricity. Natural radioactivity was discovered in a specimen of a uranium mineral whose physical properties were under study. Superconductivity was discovered incidentally in a systematic study of the electrical conductivity of simple metals close to the absolute zero of temperature. The possibility of creating a light-emitting crystal diode was suggested once wave mechanics was developed and began to be applied to advance our understanding of the properties of materials further. Actually, achievement of the device proved to be more difficult than its conception. The materials involved had to be prepared with great care.

Among the many avenues explored for the sake of obtaining new basic knowledge is that related to the influence of imperfections on the properties of materials. Some imperfections, such as those which give rise to temperature-dependent electrical conductivity in semiconductors, salts and metals could be ascribed to thermal fluctuations. Others were linked to foreign atoms which were added intentionally or occurred by accident. Still others were the result of deviations in the arrangement of atoms from that expected in ideal lattice structures. As might be expected, discoveries in this area not only clarified mysteries associated with ancient aspects of materials research, but provided tests that could have a bearing on the properties of materials being explored for novel purposes. The semiconductor industry has been an important beneficiary of this form of exploratory research since the operation of integrated circuits can be highly sensitive to imperfections.

In this connection, it should be added that the ever-increasing search for special materials that possess new or superior properties under conditions in which the sponsors of exploratory research and development and the prospective beneficiaries of the technological advance have parallel interests has made it possible for those engaged in the exploratory research to share in the funds directed toward applications. This has done much to enhance the degree of partnership between the scientist and engineer in advancing the field of materials research.

Finally, it should be emphasized again that whenever materials research has played a decisive role in advancing some aspect of technology, the advance has frequently been aided by the introduction of an increasingly sophisticated set of characterization tools that are drawn from a wide range of scientific disciplines. These tools usually remain a part of the array of test equipment.

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PREFACE

Materials research is an extraordinarily broad and diverse field. It draws on the science, technology, and tools of a variety of scientific and engineering disciplines as it pursues research objectives spanning very fundamental to the highly applied. Beyond the generic idea of "material" *per se*, perhaps the single unifying element that qualifies this collection of pursuits as a field of research and study is the existence of a portfolio of characterization methods that is widely applicable irrespective of discipline or ultimate materials application. *Characterization of Materials* specifically addresses that portfolio with which researchers and educators must have working familiarity.

The immediate challenge to organizing the content for method volumes is determining how best to parse the field. By far the majority of materials researchers focus on particular classes of materials and their end uses. A comfortable choice would be to commission chapters accordingly. The objective and product of any measurement, materials properties, could also form a logical basis. Unfortunately, these approaches would require mentioning several of the measurement methods in just about every chapter. Therefore, if only to reduce redundancy, the content is arranged here according to the type of measurement "probe" upon which a method relies. Thus, separate chapters bring together specific techniques that focus on application of electrons, ions, x-rays, neutrons, heat, light, etc. Our field is too complex for this not to be an oversimplification, and indeed some logical inconsistencies are inevitable.

We have tried to maintain the distinction between a property and a method. This is easy and clear for methods based on external independent probes such as electron beams, ion beams, neutrons, or x-rays. However, many techniques rely on one and the same phenomenon for probe and property, as is the case for mechanical, electronic, and thermal methods. Many methods fall into both regimes. For example, light may be used to observe a microstructure, but may also be used to measure an optical property. From the most general viewpoint, we recognize that the properties of the measuring device and those of the specimen under study are inextricably linked. It is actually a joint property of the tool-plus-sample system that is observed. When both tool and sample each contribute their own *materials* properties, for example, electrolyte and electrode, pin and disc, source and absorber, etc., distinctions are blurred. Although these distinctions in principle ought not to be taken too seriously, keeping them in mind will aid in efficiently accessing content of interest in these volumes.

Frequently, the materials property sought is not what is directly measured. Rather it is deduced from direct observation of some other property or phenomenon that acts as a signature of what is actually of interest. These relationships take many forms. Thermal arrest, magnetic anomaly, diffraction spot intensity, relaxation rate and resistivity, to name only a few, might all serve as signatures of a phase transition and be used as "spectator" properties to determine a critical temperature. Similarly, inferred properties such as charge carrier mobility are deduced from basic electrical quantities and temperature-composition phase diagrams are deduced from observed microstructures. *Characterization of Materials*, being organized by the technique, naturally places initial emphasis on the most directly measured properties, but authors have provided many application examples that illustrate the derivative properties that techniques may address.

First among our objectives is to help the researcher discriminate among alternative measurement modalities that may apply to their property of interest. The field of possibilities is often very wide, and although excellent texts treating each possible method in great detail exist, identifying the most appropriate method before delving deeply into any one seems the most efficient approach. *Characterization of Materials* serves to sort the options at the outset, with individual articles affording the researcher a description of the method sufficient to understand its applicability, limitations, and relationship to competing techniques, while directing the reader to more extensive resources that fit specific measurement needs.

Whether one plans to perform such measurements oneself or simply needs to gain sufficient familiarity to effectively collaborate with experts in the method, *Characterization of Materials* will be a useful reference. Although our expert authors were given great latitude to adjust their presentations to the "personalities" of their specific methods, some uniformity was sought.

Thus, you will find most units organized in a similar fashion. First, an introduction serves to succinctly describe for what properties the method is useful and what alternatives may exist. Underlying physical principles of the method and practical aspects of its implementation follow. Most articles will offer examples of data and their analyses as well as warnings about common problems of which one should be aware. Preparation of samples and automation of the methods are also covered when appropriate.

As implied above, the level of presentation of these volumes is intended to be intermediate between cursory overview and detailed instruction. Readers will find that, in practice, the level of coverage is also very much dictated by the character of the technique described. Many are based on quite complex concepts and devices. Others are less so, but still, of course, demand precision of understanding and execution. What is or is not included in a presentation also depends on the assumed technical background of the reader. Concepts that are part of common technical curricula are therefore omitted, while less common, more specialized topics are included.

As much as possible, we have avoided extended discussion of the science and application of the materials properties themselves, which, although very interesting and clearly the motivation for research in the first place, do not generally speak to efficacy of a method or its implementation.

These are *materials-oriented* volumes, and as such must overlap fields such as physics, chemistry, metallurgy, and engineering. There is no sharp delineation possible between a "physics" property (e.g., the band structure of a solid) and the materials consequences (e.g., conductivity, mobility, etc.). At the other extreme, it is not at all clear where a materials property such as toughness ends and an engineering property associated with performance and life-cycle begins. The very attempt to assign such concepts to only one disciplinary category serves no useful purpose. Suffice it to say that *Characterization of Materials* has focused its coverage on a core of materials topics while trying to remain inclusive at the boundaries of the field.

Processing and fabrication are also important aspects of materials research. *Characterization of Materials* does not deal with these methods *per se*, because they are not strictly measurement methods. However, here again no clear line is found and in such methods as electrochemistry, tribology, mechanical testing, and even ion-beam irradiation, where the processing can *be* the measurement, these aspects are perforce included.

The second chapter is unique in that it collects methods that are not, literally speaking, measurement methods; these articles do not follow the format found in subsequent chapters. As theory or simulation or modeling methods, they certainly serve to augment experiment. They may be a necessary corollary to an experiment to understand the result after the fact or to predict the result and thus help direct an experimental search in advance. More than this, as equipment needs of many experimental studies increase in complexity and cost, as the materials themselves become more complex and multicomponent in nature, and as computational power continues to grow, simulation of properties and behaviors *will* in fact become the measurement method of choice in many cases.

Another unique chapter is the first, covering "common concepts." It collects some of the ubiquitous aspects of measurement methods that would have had to be described repeatedly and in more detail in later articles. Readers may refer back to this chapter as related topics arise around specific methods, or they may use this chapter as a general tutorial. The Common Concepts chapter, however, does not and should not eliminate *all* redundancies in the remaining chapters. Expositions within individual articles attempt to be somewhat self-contained and the details as to how a common concept actually relates to a given method are bound to differ from one to the next. Although *Characterization of Materials* is directed more toward the research laboratory than the classroom, all articles treat their topics in a tutorial style and should prove useful as adjuncts to any materials science curriculum.

The content of the first edition of *Characterization of Materials* had previously appeared as *Methods in Materials Research*, a loose-leaf compilation amenable to updating. To retain the ability to keep content as up to date as possible, *Characterization of Materials* was also published online where several additional topics appeared.

THE SECOND EDITION

Over the last decade, materials research and its characterization tools have seen many advances. The second edition of *Characterization of Materials* attempts to capture those advances related to measurement methods in two ways. Each of the topics of the first edition was considered for updating. Whereas a few quite basic and classical topics stood the test of time and are reproduced without change, the vast majority of topics have been updated by their original authors when available or by a newly identified expert in the relevant field.

The second edition also includes a number of new topics—topics that did not exist or at least were not commonplace among measurement techniques a decade or so ago. Over 50 new articles have been added, several of which are collected in a new fourteenth chapter on scanning probe methods. Advances in instrumentation account for many new and updated topics. The aberration-corrected transmission electron microscope is one good example. Another is an ever expanding set of powerful techniques using x-rays and neutrons at major user facilities.

Also, the role of high-performance computing cannot be overstated. Faster processors, now at the petascale and still advancing; thousands of cores supporting parallel processing algorithms; and massive data storage capacities have all contributed to making feasible very large computations not within reach just a few years ago.

Perhaps the most exciting aspect of advances in the field is the number of new materials systems that are being subjected to characterization—the mandatory step on the path toward understanding the fundamentals and to eventual applications. These new systems force us to expand and blur our boundaries as we reexamine the scope of a reference work such as *Characterization of Materials*. We include studies at the nanoscale that border on condensed matter and even atomic physics; techniques more often associated with biology for measurements on soft and hybrid materials; and more electrochemical methods of great relevance to today's energy storage needs.

Finally, we can see some characterization challenges that are still near their origins. Multiscale modeling that takes full advantage of advances in computing power as it endeavors to link results at the microscale to real-world macrosystems and to bridge timescales from femtoseconds to seconds to hours and days is one such area. Another is the "simultaneous" application of multiple measurement methods to synergistically determine materials properties. The latter challenge takes many forms: *in situ* experiments in microscopes or at beam lines; joint refinement and analysis of disparate data sets; and real-time coupled data analysis, simulation, and experimental parameter adjustment; to name just a few.

This second edition of *Characterization of Materials* like the first appears in print and online. The online version affords the opportunity to add new developments over time. Also of interest to some readers may be earlier versions of individual articles that will also be accessible via the online version. As advantageous as that online version is and will continue to be, this editor would like to raise one caveat. Just as a student cannot hope to grasp the big picture, the interrelationships among the various aspects of a multifaceted topic, by attending only a few lectures plucked from a semester-long syllabus, the online reader of a few isolated articles will not benefit from the synergistic tutorial arrangement of articles, chapters and indeed the complete *Characterization of Materials* work. The essence of materials research as a distinctive field in its own right *is* that cross-disciplinary synergy of many complimentary tools. One hopes that the miraculous efficiency of the modern Internet and online access to tidbits of data on all possible subjects will, in our case, not be perceived as a substitute for delving into the character and value of materials characterization as a unified coherent component of the materials research enterprise.

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Development of the second edition presented its own unique challenges. Throughout the entire proposal, acquisition and production process, the hands-on management of Executive Editor, Dr. Arza Seidel was again invaluable. Day-to-day interactions with editors and authors were also expertly supported by Development Editors Erin Arndt and Dr. Mihai Peterca. Appreciation is perhaps most deserved by authors of articles in the first edition, who, after more than a decade, were able to bring their contributions up-to-date and to new authors who stepped in when original authors could not and built updated versions of those first edition articles. The opportunity to add new topics in the second edition also relied on authors and on some chapter editors new to *Characterization of Materials*. We thank them for contributing their considerable expertise to the second edition that would not have been as comprehensive in scope and reflective of advances in the field without their help.

For the editors,
Elton N. Kaufmann, Editor-in-Chief
2003 and 2012

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Challenges to Structure Prediction and Structure Characterization at the Nanoscale

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High-Strain-Rate Testing of Materials: The Split-Hopkinson Pressure Bar

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Atomic Excitation Exploited by Energetic-Beam Characterization Methods

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Carrier Lifetime: Free Carrier Absorption, Photoconductivity, and Photoluminescence

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Principles and Practices of Thermal Analysis and Calorimetry

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Ultraviolet Photoelectron Spectroscopy

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Nuclear Magnetic Resonance: Basic Principles and Liquid State Spectroscopy

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Atomic Force Microscopy and Spectroscopy

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X-Ray and Neutron Diffuse Scattering Measurements
X-Ray Microprobe for Fluorescence and Diffraction Analysis

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Characterizing Micro and Nanomaterials Using MEMS Technology

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Challenges to Structure Prediction and Structure Characterization at the Nanoscale

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Atomic Excitation Exploited by Energetic-Beam Characterization Methods
"Total" Ion Beam Analysis—3D Imaging of Complex Samples Using MeV Ion Beams
Elastic Backscattering of Ions for Compositional Analysis
Ion Beam Methods, Introduction

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