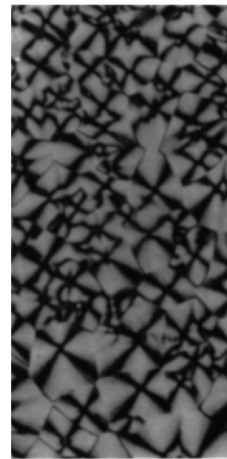
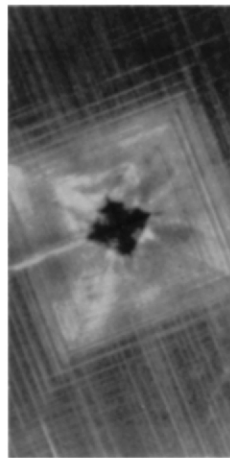
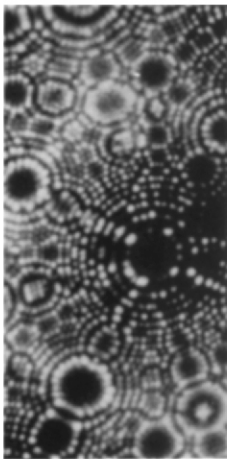


CONCISE ENCYCLOPEDIA OF
MATERIALS
CHARACTERIZATION

EDITORS
ROBERT W CAHN FRS
ERIC LIFSHIN



PERGAMON PRESS

**CONCISE ENCYCLOPEDIA OF
MATERIALS CHARACTERIZATION**

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CONCISE ENCYCLOPEDIA OF
MATERIALS CHARACTERIZATION

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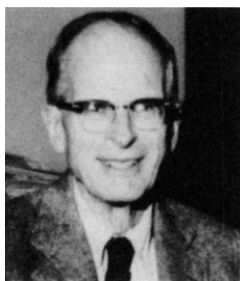
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MICHAEL BERLINER BEVER

Professor Michael Bever, Editor-in-Chief of the *Encyclopedia of Materials Science and Engineering* and Senior Advisory Editor of the Supplementary Volumes and the Concise Encyclopedias, died in Cambridge, Massachusetts, on 17 July, 1992.

He was born in Berlin in 1911 and received a degree of Doctor of Jurisprudence from the University of Heidelberg in 1934, the year in which he emigrated to the USA. In 1937 he was awarded a master's degree in business administration by Harvard University. He then studied metallurgy at the Massachusetts Institute of Technology and received his doctorate in 1944. Soon afterwards, he was appointed to the faculty of the Department of Metallurgy (now the department of Materials Science and Engineering) at MIT, where his profound knowledge and diverse interests, ranging from physical metallurgy to materials economics and management, sustained his research and teaching for nearly half a century.

Professor Bever was one of a small group of people who discussed the concept of an encyclopedia of materials in the 1950s, but the project did not get under way until 1974 when Pergamon Press decided to commit the necessary resources and to appoint Michael Bever as Editor-in-Chief. He devoted the remaining years of his life to the Encyclopedia. It is a fitting memorial to his scholarship and to the central role which he played in the evolution of materials science and engineering.

Walter S Owen
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FOREWORD

In the time since its publication, the *Encyclopedia of Materials Science and Engineering* has been accepted throughout the world as the standard reference about all aspects of materials. This is a well-deserved tribute to the scholarship and dedication of the late Editor-in-Chief, Professor Michael Bever, to the Subject Editors and to the numerous contributors.

During its preparation, it soon became clear that change in some areas is so rapid that publication would have to be a continuing activity if the Encyclopedia were to retain its position as an authoritative and up-to-date systematic compilation of our knowledge and understanding of materials in all their diversity and complexity. Thus, the need for some form of supplementary publication was recognized at the outset. The Publisher has met this challenge most handsomely: both a continuing series of Supplementary Volumes to the main work and a number of smaller encyclopedias, each covering a selected area of materials science and engineering, are in the process of publication.

Professor Robert Cahn, the Executive Editor, was previously the editor of an important subject area of the main work and many other people associated with the Encyclopedia have contributed or will contribute to its Supplementary Volumes and derived Concise Encyclopedias. Thus, continuity of style and respect for the high standards set by the *Encyclopedia of Materials Science and Engineering* are assured. They have been joined by some new editors and contributors with knowledge and experience of important subject areas of particular interest at the present time. Thus, the Advisory Board is confident that the new publications will significantly add to the understanding of emerging topics wherever they may appear in the vast tapestry of knowledge about materials.

The appearance of the Supplementary Volumes and the series *Advances in Materials Science and Engineering* is an event which will be welcomed by scientists and engineers throughout the world. We are sure that it will add still more luster to a most important enterprise.

Walter S Owen
Chairman
Honorary Editorial Advisory Board

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EXECUTIVE EDITOR'S PREFACE

As the publication of the *Encyclopedia of Materials Science and Engineering* approached, Pergamon Press resolved to build upon the immense volume of work which had gone into its creation by embarking on a follow-up project. This project had two components. The first was the creation of a series of Supplementary Volumes to the Encyclopedia itself. The second component of the new project was the creation of a series of Concise Encyclopedias on individual subject areas included in the Main Encyclopedia to be called *Advances in Materials Science and Engineering*.

These Concise Encyclopedias are intended, as their name implies, to be compact and relatively inexpensive volumes (typically 400–600 pages in length) based on the relevant articles in the Encyclopedia (revised where need be) together with some newly commissioned articles, including appropriate ones from the Supplementary Volumes. Some Concise Encyclopedias offer combined treatments of two subject fields which were the responsibility of separate Subject Editors during the preparation of the parent Encyclopedia (e.g., dental and medical materials).

Eleven Concise Encyclopedias have been published. These and their editors are listed below.

<i>Concise Encyclopedia of Advanced Ceramic Materials</i>	Prof. Richard J Brook
<i>Concise Encyclopedia of Building & Construction Materials</i>	Prof. Fred Moavenzadeh
<i>Concise Encyclopedia of Composite Materials</i>	Prof. Anthony Kelly CBE, FRS
<i>Concise Encyclopedia of Magnetic & Superconducting Materials</i>	Dr Jan Evetts
<i>Concise Encyclopedia of Materials Characterization</i>	Prof. Robert W Cahn FRS & Dr Eric Lifshin

<i>Concise Encyclopedia of Materials Economics, Policy & Management</i>	Prof. Michael B Bever†
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<i>Concise Encyclopedia of Medical & Dental Materials</i>	Prof. David Williams
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<i>Concise Encyclopedia of Mineral Resources</i>	Dr Donald D Carr & Prof. Norman Herz
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<i>Concise Encyclopedia of Polymer Processing & Applications</i>	Mr Patrick J Corish
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<i>Concise Encyclopedia of Semiconducting Materials & Related Technologies</i>	Prof. Subhash Mahajan & Prof. Lionel C Kimerling
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<i>Concise Encyclopedia of Wood & Wood-Based Materials</i>	Prof. Arno P Schniewind
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Many of the new or substantially revised articles in the Concise Encyclopedias have been or published in one of the three Supplementary Volumes, which are designed to be used in conjunction with the Main Encyclopedia. The Concise Encyclopedias themselves, however, are “free-standing” and are designed to be used without reference to the parent Encyclopedia.

The Executive Editor was personally responsible for the selection of topics and authors of articles for the Supplementary Volumes. In this task, he has had the benefit of the advice of the Senior Advisory Editor and of other members of the Honorary Editorial Advisory Board. The Executive Editor was responsible for appointing the Editors of the various Concise Encyclopedias and for supervising the progress of these volumes.

The entire advances in Materials Science and Engineering project will be completed at the end of 1992.

Robert W Cahn FRS
Executive Editor

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EDITORS' PREFACE

The *Concise Encyclopedia of Materials Characterization* is founded on the large group of articles on characterization and investigation of materials in the original *Encyclopedia of Materials Science and Engineering*, published in 1986, for which Eric Lifshin was responsible at the time. These articles were supplemented with a number of additional ones which had been commissioned by Robert Cahn for *Supplementary Volume 1* and *Supplementary Volume 2*, published in 1988 and 1990 respectively, with further articles that were specifically commissioned by both of us for the present volume. We believe that this gradual accretion of essays on what has eventually become an extremely wide range of techniques has proved to be an effective way of covering this enormous field.

The text begins with an introduction to the whole field, written by Eric Lifshin. The majority of the 116 alphabetical articles cover a range of techniques of characterization which are not specific to any one group of materials, although no fewer than 12 are specific to polymers and several more are specific to metals, ceramics or semiconductors. Again, a majority of the techniques covered are intrinsically bulk methods, but a substantial minority are specific to the study of the topography and composition of surfaces and near-surface regions. Some are venerable techniques (though even these are regularly modernized) and others are of very recent vintage: this latter includes, by way of example, articles such as *Atomic Force Microscopy*; *Confocal Optical Microscopy*; *Gamma-Ray Diffractometry*; *Thermal Wave Imaging*; and *X-Ray Diffraction, Time-Resolved*. The article titles guide the reader to articles covering specific techniques, but as techniques may be discussed elsewhere other than in an article devoted to them, the detailed index will aid the reader to find all the various places where a particular topic is treated.

Like all editors, we have been constrained by the available space to make some difficult choices concerning what to include and what to leave out. In particular, the shadowy borderland between characterization and nondestructive evaluation posed some problems: in general, we omitted techniques which are closer to the world of inspection than to that of research.

We wish to express our gratitude for the hard work, and forbearance, of the many authors who have written articles for us, sometimes against demanding deadlines. We are also greatly indebted to the professionals at Pergamon Press—in particular, Peter Frank and Helen McPherson—for all they have done to bring this volume into existence.

Robert W Cahn
Eric Lifshin
Editors

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GUIDE TO USE OF THE ENCYCLOPEDIA

This Concise Encyclopedia is a comprehensive reference work covering all aspects of materials characterization. Information is presented in a series of alphabetically arranged articles which deal concisely with individual topics in a self-contained manner. This guide outlines the main features and organization of the Encyclopedia, and is intended to help the reader to locate the maximum amount of information on a given topic.

Accessibility of material is of vital importance in a reference work of this kind and article titles have therefore been selected not only on the basis of article content but also with the most probable needs of the reader in mind. An alphabetical list of all the articles contained in this Encyclopedia is to be found on pp. xvii and xviii.

Articles are linked by an extensive cross-referencing system. Cross-references to other articles in the Encyclopedia are of two types: in text and end of text. Those in the body of the text are designed to refer the reader to articles that present in greater detail material on the specific topic under discussion at that point. They generally take one of the following forms:

...which is fully described in the article *Field-Ion Microscopy: Observation of Radiation Effects*.

...other applications of this technique (see *Laser Sampling Inductively Coupled Plasma Mass Spectrometry*).

The cross-references listed at the end of an article serve to identify broad background reading and to direct the reader to articles that cover different aspects of the same topic.

The nature of an encyclopedia demands a higher degree of uniformity in terminology and notation than many other scientific works. The widespread use of the SI system of units has determined that such units be used in this Encyclopedia. It has been recognized, however, that in some fields Imperial units are more generally used. Where this is the case, Imperial units are given with their SI equivalent quantity and unit following in parentheses. Where possible, the symbols defined in *Quantities, Units, and Symbols*, published by the Royal Society of London, have been used.

All articles in the Encyclopedia include a bibliography giving sources of further information. Each bibliography consists of general items for further reading and/or references which cover specific aspects of the text. Where appropriate, authors are cited in the text using a name/date system as follows:

...as was recently reported (Smith 1988).

Jones (1984) describes...

The contributors' names and the organizations to which they are affiliated appear at the end of all articles. All contributors can be found in the alphabetical List of Contributors, along with their full postal addresses and the titles of the articles of which they are authors or coauthors.

The article *An Introduction to Investigation and Characterization of Materials* provides an overview of the subject area and discusses in brief the issues covered in detail by the articles in the body of the work.

The most important information source for locating a particular topic in the Encyclopedia is the multilevel Subject Index, which has been made as complete and fully self-consistent as possible.

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ALPHABETICAL LIST OF ARTICLES

Acoustic Emission
Acoustic Microscopy
Adhesives: Tests for Mechanical Properties
Anodization Spectroscopy
Art Forgeries: Scientific Detection
Atomic Force Microscopy
Auger Electron Spectroscopy
Auger Microscopy, Angular Distribution
Ceramic Materials: Cathodoluminescence Analysis
Ceramic Powders: Packing Characterization
Channelling-Enhanced Microanalysis
Chemical Analysis of Solid Surfaces
Composite Materials: Nondestructive Evaluation
Compton Scattering
Confocal Optical Microscopy
Corrosion and Oxidation Study Techniques
Corrosion: Test Methods
Depth Profiling
Diffusion: Novel Measurement Methods
Elastomers: Spectroscopic Characterization
Elastomers: Tests for Mechanical Properties
Electron Diffraction
Electron Diffraction, Low-Energy
Electron Energy-Loss Spectrometry
Electron Microprobe Analysis
Electron Microscope Analysis of Defect Clusters,
 Voids and Bubbles
Electron Microscopy, Analytical
Electron Microscopy, High-Resolution
Electron Microscopy, High-Voltage
Electron Spectroscopy for Chemical Analysis
Electron Spin Resonance
Electron Tunnelling Spectroscopy
Field-Ion Microscopy: Atom-Probe Microanalysis
Field-Ion Microscopy: Observation of Radiation
 Effects
Fractals
Gamma Radiography
Gamma-Ray Diffractometry
Gas and Liquid Chromatography
Gas Sensors, Solid-State
Grain Size: Nondestructive Evaluation
Hardness Characterization
Hydrogen as a Metallurgical Probe
Infrared Spectroscopy
Ion Backscattering Analysis
Junction Transient Spectroscopy
Laser Microprobe Mass Spectrometry
Laser Sampling Inductively Coupled Plasma Mass
 Spectrometry
Liquid Chromatography Mass Spectroscopy
Magnetic Materials: Measurements
Mechanical Properties Microprobe
Microengineering of Materials: Characterization
Microstructural Evolution: Computer Simulation
Microtextural Analysis
Mössbauer Spectroscopy
Neutron Radiography
Nuclear Magnetic Resonance Spectroscopy
Nuclear Magnetic Resonance Spectroscopy of
 Solids
Optical Calorimetry
Optical Emission Spectroscopy
Optical Microscopy
Organic Mass Spectrometry
Paper and Paperboard: Destructive Mechanical
 Testing
Paper and Paperboard: Nondestructive Evaluation
Particle-Induced X-Ray Emission
Phase Diagrams and Phase Stability: Calculations
Pole Figures and Orientation Distribution Functions
Polymer Dielectric Properties: Test Methods
Polymers: Electron Microscopy
Polymers: Light Microscopy
Polymers: Molecular Weight and its Distribution
Polymers: Neutron Scattering
Polymers: Raman Spectroscopy
Polymers: Tests for Degradation and Stabilization
Polymers: Tests for Flammability
Polymers: Tests for Mechanical Properties
Polymers: Tests for Thermal Properties
Polymers: Thermal Analysis
Polymers: X-Ray Scattering
Porosity: Characterization and Investigation
Positron Annihilation Spectroscopy of Defects in
 Metals
Powder Characterization
Powder Mechanics
Raman Spectroscopy
Reflection Electron Microscopy
Residual Stresses: Measurement Using Neutron
 Diffraction
Residual Stresses: Nondestructive Evaluation
Scanning Electron Microscopy
Scanning Tunnelling Microscopy and Spectroscopy
Secondary-Ion Mass Spectrometry
Semiconducting Materials: Characterization by
 Etching
Semiconducting Materials: Electron Microscopy
Single-Crystal X-Ray Diffraction
Small-Angle Neutron Scattering in Metallurgy
Solid-State Nuclear Track Detectors: Applications
Solid State: Study Using Muon Beams
Spark-Source Mass Spectrography
Stress Distribution: Analysis Using Thermoelastic
 Effect
Superconducting Materials: Measurements
Texture: Nondestructive Characterization

Thermal Analysis: An Overview	Characteristics
Thermal Analysis: Recent Developments	X-Ray Absorption Spectroscopy: EXAFS and XANES Techniques
Thermal Wave Imaging	X-Ray and Neutron Diffraction Studies of Amorphous Solids
Thermodynamic Activity: Measurement	X-Ray and Neutron Diffuse Scattering of Radiation-Induced Defects
Thermoluminescence	X-Ray Diffraction, Time-Resolved
Thermophysical Measurements, Subsecond	X-Ray Fluorescence Spectrometry
Thin Films: Characterization by X Rays	X-Ray Microanalysis, Quantitative
Transmission Electron Microscopy: Convergent-Beam and Microdiffraction Techniques	X-Ray Powder Diffraction
Vibrothermography	
Wood: Acoustic Emission and Acousto-Ultrasonic	

AN INTRODUCTION TO INVESTIGATION AND CHARACTERIZATION OF MATERIALS

by Eric Lifshin

Present-day materials science depends heavily on understanding how the properties of a material relate to its composition and microstructure. However, this approach is relatively recent and for centuries before Bohr's model of the atom, skilled craftsmen and builders produced many objects of remarkable utility and beauty without any knowledge of what is now considered basic chemistry or metallurgy. They started with materials found in their native state or those requiring minimal processing. Desired properties were achieved by trial and error experimentation or the utilization of experience passed down through generations.

For thousands of years certain metals were extracted from their ores by smelting, with variations in the resulting properties often being attributed to supernatural intervention rather than to subtle differences in the materials or processes used. As time passed and printing developed, practical experience was written down. For example, in the sixteenth century Agricola's *De re metallica* described many of the methods used for the extraction, measurement and classification of metals. At about the same time empirical knowledge existed of how to use distillation to prepare a variety of simple compounds including various alcohols and oils.

Until the eighteenth century only a small number of elements were known, and physical attributes like density, color and hardness were sufficient to tell one from another. As chemistry and physics developed in the nineteenth century, the growth of more basic understanding led to the discovery of a number of elements and the extraction or synthesis of thousands of compounds. These new substances were applied to many areas including medicine, construction, transportation, weaponry and agriculture. By the mid-nineteenth century the amount of scientific information available had grown so large that various scientific disciplines began to split into specialties. In chemistry, general chemists were replaced by organic, inorganic, physical, physiological, agricultural and analytical chemists. In 1848 Fresenius founded the first analytical laboratory which, in addition to teaching, provided government agencies and industry with checks on material specifications and the analysis of water, food and physiological specimens. The idea caught on and today there are hundreds of independent analytical laboratories, as well as those associated with hospitals, industrial concerns, universities and government agencies. Following the specialization of analytical chemistry there has remained a strong interplay between the analysis and development of materials. Better analytical methods lead to a

more systematic and complete classification of existing materials and provide the insight required for the development of new ones.

1. Historical Background

The most significant event in laying the foundation for all phases of modern materials science took place at the very beginning of the nineteenth century when Dalton postulated that matter consisted of aggregates of "ultimate particles," or atoms. Atoms of any given element were indistinguishable from other atoms of the same element with respect to mass and all properties, while atoms of different substances could combine to form "atoms" (molecules) of a third substance. Dalton further recognized that elemental atoms combine only in certain ratios, so as to form products with unique stoichiometry. Knowing that he could not weigh individual atoms, he used this concept to determine the relative weights of atoms and molecules based on hydrogen. Thus, the formation of a given amount of water (assumed to be HO) with an atomic weight of 6.5 required the combination of oxygen with an atomic weight of 5.5 with hydrogen with an atomic weight of 1.0. Although Dalton did not recognize that two atoms of hydrogen combine with one atom of oxygen to form one molecule of water (a point which was later clarified by Avogadro) and his atomic weights were not particularly accurate, his ideas are of fundamental importance to modern chemistry.

The concept of definite proportions and atomic weights combined with a fairly consistent symbolic nomenclature for the elements, introduced by Berzelius, led to the description of matter and chemical reactions in the form with which we are now familiar. This approach also led to the emergence of analysis techniques based on chemical behavior rather than physical property determinations. For example, the method known as gravimetric analysis was widely used in the nineteenth century. A chemical reaction between a solution of the material to be analyzed and a second reagent results in the formation of an insoluble compound containing the element of interest. The precipitate is isolated, dried and weighed, and the quantity of the element is determined from its stoichiometric fraction.

A second technique, known as volumetric analysis (titration), was developed at about the same time. It involves measuring the volume of a reagent necessary to react completely with a fixed quantity of a sample in a predetermined chemical reaction. At first it was not as commonly used as gravimetric analysis,

because of difficulty in measuring reaction end points, but as more indicators became available the method grew in popularity. A third technique, known as colorimetry, was also known in the nineteenth century. Selected reactions are performed to produce colored compounds containing the element of interest. Color intensity is then measured, relative to standard solutions, to estimate the amount of that element present. Although this approach was initially limited to a few elements and quantitative analysis was fairly subjective, its use increased as more colorimetric reagents were identified and instrumentation was developed to make measurements more accurate.

The nineteenth century also saw the initial development of electroanalytical chemistry and the use of polarized light to discriminate between materials, but the most significant instrumental method developed was the spectroscope. Its operation is based on the ability of a prism to disperse white light into its various constituents, a fact demonstrated by Newton in the seventeenth century. In 1814 Fraunhofer, a lens manufacturer, recognized that a number of both bright and dark lines could be observed in both sunlight and the flames produced by burning different fuels. The idea of using absorption spectra for more general chemical analysis was proposed by Brewster a few years later. In the 1850s Bunsen and Kirchhoff developed a spectroscope with an improved source and viewing optics which is the forerunner of instruments used today. With it they discovered both the elements cesium and rubidium.

At the beginning of the nineteenth century only about 30 elements were known; by the end this number had more than doubled. During this period it also became apparent that some type of systematic classification of the elements was needed. The concept of the periodic table was independently developed by Mendeleev in Russia and Meyer in Germany using schemes based on atomic weights and various other physical properties. Although ordering of the elements is now related to atomic number and the electronic structure of the elements, most of the changes made over the years have been to add new elements rather than to reorder known elements.

At the end of the nineteenth century and the beginning of the twentieth century a series of major events took place which revolutionized understanding of both the atom and the electromagnetic radiation. Studies of the electrical conductivity of gases at reduced pressure led to the discovery of cathode rays by Hittorf in 1869, confirmed by Goldstein in 1876. Although initially the nature of these rays was unclear, work by Thomson, published in 1897, showed that they consisted of negatively charged particles, from their deflection in magnetic and electrostatic fields. He also demonstrated that these particles (later called electrons) had the same charge-to-mass ratio as those given off from hot filaments or released during the exposure of certain metals to

electromagnetic radiation (the photoelectric effect, discovered by Hertz in 1887). Thus they appeared to be a key component of all matter, and when removed would leave atoms as positive ions.

The nature of this positive ion core was studied in the first decade of the twentieth century by Rutherford, who experimented on the scattering of α particles from thin metal foils. From this work came a model of the atom in which electrons revolve around a positively charged nucleus. Although this model was consistent with his observations, it was unacceptable from the point of view of classical physics, since the electrons would continuously radiate energy and should, therefore, spiral into the nucleus. However, this dilemma was soon reconciled by Bohr, who postulated that the electron orbits corresponded to discrete energy states and as long as an electron remained in its orbit no radiation would occur. The idea of discrete energy states was based on the principles of quantum mechanics introduced by Planck in 1900 to describe radiation emitted from a black body. Using these states, Bohr was able to describe correctly the line spectra of hydrogen observed in the 1880s by Balmer and mathematically formulated by Rydberg in 1890. Although the Bohr model was significantly modified by Sommerfeld and Schrödinger to describe multielectron atoms more accurately, the concept of electron transitions between discrete levels and the accompanying release or absorption of energy is of central importance to many of the different types of photon and electron spectrometries used today.

Thomson's use of magnetic and electric fields to determine charge-to-mass ratios was also applied to the characterization of positive ion beams (called Kanalstrahlen by Goldstein), which were also known to exist along with cathode rays, as shown in experiments on the electrical conductivity of gases. Thomson's apparatus, known as a parabolic mass spectrometer, produced a series of photographically recorded parabolas, one for each charge-to-mass ratio. The system lacked mass resolution, however, because it had no energy selectivity. This difficulty was overcome several years later (1919) when Aston developed a velocity focusing instrument which showed that the spectrum of neon actually contained two peaks, one at mass 20 and the other at mass 22. This demonstrated that an element of a given number can have more than one form (isotopes) and thus revealed the power of the mass spectrometer as an important analytical instrument. In the decades that followed, resolution and sensitivity continued to improve as a result of the efforts of Dempster, Mattauch and Herzog, and Nier and Johnson. Some commercially available instruments today have mass resolutions of 1 part in 100 000, compared with the 1 part in 12 of the first parabolic spectrometers.

Experiments on the electrical conductivity of gases also led to the accidental discovery of x rays by Roentgen in 1896. He recognized that their unusual

penetrating power was influenced by the tube operating voltage as well as by the thickness and type of absorbers used. Since x rays were not deflected by magnetic or electrostatic fields, it was believed that they were part of the electromagnetic spectrum, but that they must have a very short wavelength. Determining their wavelength distribution presented something of a problem, because although diffraction gratings had previously been developed for dispersing visible spectra, they were not fine enough to separate x rays. The solution was proposed by Laue in 1912, who believed that the interatomic spacing of crystals should be of the right order of magnitude to act as suitable diffraction gratings. He proved this hypothesis by passing a beam of x rays through a crystal of zinc sulfide and obtaining a pattern of spots on a photographic plate.

This work stimulated further studies by W. H. Bragg and W. L. Bragg (father and son), who built the first crystal diffraction spectrometer. Using a crystal of rock salt with a known interplanar spacing, they were able to characterize the spectral distribution of a platinum x-ray target and also develop the simple equation which relates interplanar spacing, x-ray wavelength and diffraction angle. The spectra they observed consisted of both a broad band of general radiation (the x-ray continuum) and discrete lines characteristic of the tube anode. Investigations by Moseley described in 1913 showed that there also existed a simple relationship between the wavelengths of the characteristic lines emitted from an element and its atomic number. This unique relationship forms the basis for elemental analysis by a variety of x-ray, electron and ion-induced x-ray spectroscopies. The work of the Braggs also provided the key to crystal structure determination by using a selected characteristic x-ray wavelength to determine a set of interplanar spacings for a single crystal.

The numerous scientific discoveries that took place at the beginning of the twentieth century provided the physical foundation for the hundreds of instrumentally oriented analytical methods which are widely used today. It is astonishing that most of what we now consider standard laboratory instruments were developed after 1930. The following techniques and key contributors are examples:

- transmission electron microscopy, Ruska (1934)
- scanning electron microscopy, von Ardenne (1938)
- liquid chromatography, Martin and Syngé (1941)
- carbon-14 dating, Libby (1946)
- nuclear magnetic resonance, Block and Purcell (1946)
- electron microprobe analysis, Castaing (1951)
- gas chromatography, Martin and James (1952)
- secondary-ion mass spectrometry, Castaing and Slodzian (1960)

The names, events and dates mentioned so far tell only a small part of the chronological development of some of the methods described. Practical refinement

of many of these instrumental methods often took many years and considerable ingenuity on the part of other key contributors. For example, the first commercially available scanning electron microscope did not appear until 27 years (1965) after von Ardenne's first instrument, and it has been only during the last few years that relatively easy-to-use computerized complete single-crystal structure determination equipment has been readily available. At present one of the most active areas of analysis is the study of surfaces by x-ray photoelectron spectroscopy but, as mentioned previously, the photoelectric effect was known before the end of the nineteenth century. Relatively recent developments in electronics, computers and high-vacuum systems have all had an enormous impact on analytical instrumentation in terms of ease of operation, sophistication of the analysis, quality of results and speed. Industrial laboratories now routinely undertake types of analysis that would formerly have required the effort for a Ph.D. thesis.

With the great diversity of instrumentation available, the role of the traditional analytical laboratory has now been expanded to include determinations of morphology, microstructure, crystallography and a variety of physical properties. The term often associated with the combination of analytical chemistry and these other methods of analysis is materials characterization. The concept of a materials characterization group goes hand in hand with the evolution of interdisciplinary materials science and engineering centers which bring together experts in such areas as metallurgy, electronic materials, ceramics, solid-state physics, polymers and surface chemistry. Not only have the variety of materials and the types of instrumentation available increased, but the reasons for looking at these materials have also multiplied. Materials characterization specialists must now be concerned with basic research, product and process development, failure analysis, regulatory compliance, patent issues, quality control, manufacturing productivity and the examination of competitive products.

It is also important to recognize the degree of analytical specialization which has been taking place. Individuals who are experts in nuclear magnetic resonance spectroscopy may not have expertise in single-crystal x-ray structure determination, even though information gained by the two techniques may be highly complementary and important to a polymer chemist. An expert in transmission electron microscopy may have only an incidental knowledge of secondary-ion mass spectrometry, even though both chemical and structural information may be the key to understanding the behaviour of an electronic device. As the demand for materials with unique properties grows, so also does the complexity of the materials themselves and the methods needed to analyze them. Examples are the chemical identification of submicrometer second-phase particles which can sig-

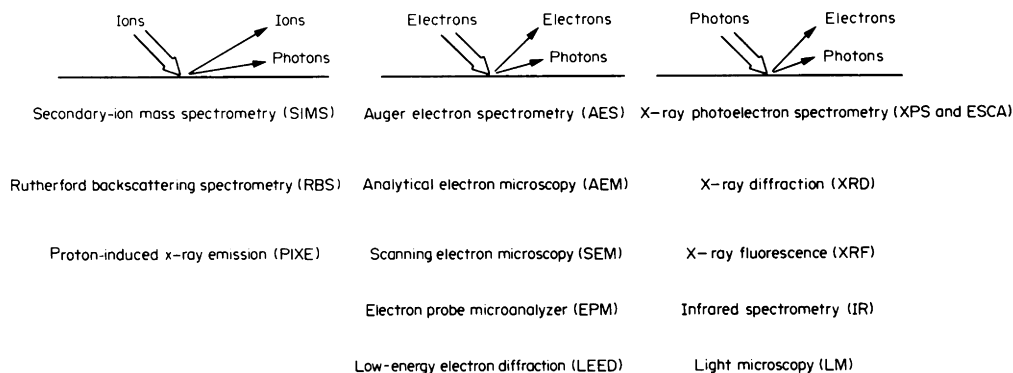


Figure 1
Analytical methods: probes and responses

nificantly influence the properties of high-temperature alloys, the determination of pollutants at the parts per billion level and the characterization of a few atomic layers on the surface of a catalyst. Clearly, scientists and engineers have enough difficulty in keeping up with advances in their own fields without having to be materials characterization experts. However, it is essential to have enough basic understanding of currently used analytical methods to be able to interact effectively with such experts.

2. Methods Available in the Modern Materials Characterization Laboratory

This Concise Encyclopedia contains a series of articles describing various methods for the investigation and characterization of materials. While not every one is covered, most of the main ones in use today are represented. Each article includes a description of the underlying physical principles, the type of samples required and typical data output and some discussion of strengths and weaknesses. As described above, the structure and composition of matter can be studied by examining how it interacts with radiation, electrons and ions. A summary of some of these interactions and the related analytical techniques is given in Fig. 1. The approach that is best suited for a particular problem depends on a variety of factors, many of which are strongly interrelated.

Chemical or microstructural analysis in most laboratories of any size can be carried out by a centralized analytical facility which maintains core analytical instruments staffed by personnel who are experts in each area. The following methods are fairly essential in any type of multidisciplinary research or engineering laboratory:

- classical chemical analysis techniques
- thermal analysis techniques
- optical microscopy and metallography
- ultraviolet, visible and infrared spectroscopy

- mass spectrometry
- gas and liquid chromatography
- scanning and transmission electron microscopy
- nuclear magnetic resonance spectrometry
- optical emission and absorption spectrometry
- x-ray diffraction and fluorescence spectrometry
- Auger and x-ray photoelectron spectrometry
- scanning tunnelling and scanning force microscopy

Other techniques employing commercially available equipment which are somewhat less commonly used although often important include:

- low-energy electron diffraction
- secondary-ion mass spectrometry
- Raman spectrometry
- low-energy ion scattering spectrometry
- field-ion microscopy and atom-probe analysis
- electron spin resonance spectrometry
- Mössbauer spectrometry
- laser microprobe analysis
- acoustic microscopy
- quantitative image analysis

What must be recognized also is that even if a well-equipped analytical laboratory has all the specialties listed in the first category, each of the methods can be substantially subdivided, requiring separate instruments and often separate skills. Taking x-ray diffraction as an example, the following subdivision can easily be made:

- conventional diffractometry
- high- and low-temperature diffractometry
- back-reflection and transmission Laue photography
- residual stress measurement
- single-crystal structure determination
- pole figure determination
- double-crystal diffractometry
- microdiffraction
- Debye-Scherrer and Guinier powder photography
- Berg-Barrett and Lang topography measurements

Services not readily available in-house can often be purchased externally from general analytical laboratories or specialists in some of the above methods.

This approach is particularly useful when the number of samples does not justify setting up an in-house facility or if in-house facilities are severely overloaded. No single laboratory can possess everything and the use of external analysts often provides an effective way of providing needed resources. In some cases, the use of external laboratories is the only approach when the instrumentation required is in the multimillion dollar price range and large support staffs are needed. Examples of such specialized facilities include:

- extended x-ray absorption fine structure measurements (using synchrotron radiation)
- neutron activation analysis
- carbon-14 dating
- picosecond laser fluorescence spectrometry
- neutron activation spectrometry
- high-voltage transmission electron microscopy
- Rutherford backscattering and ion-channelling spectrometry
- proton-excited x-ray fluorescence

There are other factors in addition to cost and speed of analysis that should be considered in the decision to use in-house or external facilities. In an industrial environment, proprietary concerns often limit sample distribution. Another important factor is the nature of the interaction between the analyst and the research scientist or engineer. In those cases where the specific type of analysis needed is known and the right sample has been selected, it may be quite satisfactory to send a sample several thousand kilometers and receive the results in a few weeks. However, in other cases a much higher degree of interaction may lead more rapidly to meaningful results. This is particularly true if the technique or the sample selected has to be changed because the initial approach is unsuccessful. It can also be true if detailed data interpretation relative to the objectives of the overall investigation is required.

Sometimes the question of who should do the analysis becomes an internal one. Should scientists or engineers have their own characterization tools or should they use a centralized service? While there is no single answer to this question, it is clearly an issue of optimizing the utilization of resources. It is not uncommon for every chemist in a laboratory to have his own gas or liquid chromatograph and every metallurgist to have his own light optical microscope. In such cases an investment of a few thousand dollars could improve research productivity by providing quick answers expediting the next experiment. It is highly unlikely, however, that each chemist would have a private nuclear magnetic resonance spectrometer and each metallurgist a separate transmission electron microscope. These require very large initial capital as well as the expenditure of both money and time to acquire the needed expertise and to maintain a piece of equipment that may not even have a long-term use. Obviously the overall impact

could be a serious decrease in research productivity.

Occasions do arise, however, when it is important to have fairly sophisticated analytical instruments closely integrated with other types of experimental apparatus. For example, samples may be altered by being transferred to separate instrumentation, as in the characterization of thin films formed by molecular beam epitaxy by the use of integral low-energy electron diffraction, Auger spectrometry and residual gas analysis instrumentation. In a manufacturing environment a process may require rapid on-line monitoring to maintain quality control. Chemical reactors frequently have in-line gas chromatographs and infrared analyzers. Dedicated analytical equipment outside a centralized facility may also be required if it serves only the objectives of a single individual's research interest and both the degree of interaction needed and the duration of the project justify the associated cost. An example would be a laser Raman spectrometer uniquely set up to study combustion chemistry and dynamics.

3. Interaction with the Analyst

Given hundreds of analytical methods, it is obvious to ask how is an appropriate method selected for solving a given problem? Before the recent proliferation of so many new types of analytical instruments, a scientist or engineer would go to a designated analytical chemist in his organization and describe the information needed. The analytical chemist would either go to suitable references or know immediately from personal experience which technique to use. Today this dialogue could be considerably extended and would undoubtedly involve experts in several fields. The scientist or engineer initiating the study should be prepared to answer a number of questions which will aid in establishing the method or methods most suitable for solving the problem. The following is a list of representative questions frequently asked, separated into three general categories. In addition some comments are added which should help to understand the role of these categories in establishing an analytical strategy. If the scientist or engineer and the materials analyst are, in fact, the same person, the discussion is replaced by introspection but the questions remain the same.

- (a) Questions relating to the type and detail of information sought:
 - Is there a need to know what elements are present and/or how much of each?
 - Is there a need to know all the elements or only some of them?
 - Down to what level?
 - What degree of accuracy is required in the results?
 - Is crystal structure or microstructural information needed?

- (b) Questions relating to what is already known about the sample:
What is the physical state of the sample?
Is the sample a metal, ceramic, semiconductor, chemical compounds or polymer?
Does the sample consist of one or more than one phase?
Is the sample amorphous or crystalline?
Is sample handling of the sample required, either because of toxicity or because the sample might transform to something different before or during analysis?
Was the sample (or its source) exposed to any unusual physical or chemical conditions which might have altered its composition or microstructure?
Does the sample consist of layers and, if it does, how many are there and how thick are they?
How much of each sample is there and how many samples are to be run?
- (c) Questions relating to the working relationship between the analyst and the scientist or engineer:
How soon are results needed?
How much can be spent to obtain the results?
Is your presence needed during the analysis?
Is a detailed report on the results needed?
Are the samples proprietary?

These three categories of questions can be thought of as screens which help to identify one or more methods of approaching a given problem. They can easily be related to three general classes of analysis requests as follows.

- (i) In the simplest case, the analysis is merely to repeat a particular type of measurement made earlier on a similar sample. Therefore, the method has been preselected and the only issues to be resolved are associated with the questions of category (c). The answers are determined by laboratory policy, backlogs, urgency of the work and availability of people and equipment. An example of this class of problem would be a request for mercury analysis in water by atomic absorption as part of periodic sampling of a body of water.
- (ii) More frequently, a suitable method may be suggested based on earlier work, but the characteristics of the sample may be sufficiently different that the questions asked in category (b) also become important. The new sample may be of a different size or shape. It may also contain other elements or compounds that could complicate the analysis if the same analytical method is used. As an example, a researcher develops a new polymer containing a chlorinated flame retardant. He/she wants a chlorine analysis in the 50 ppm region but has only a 5 mg sample. Previously, successful results would be obtained by x-ray fluorescence.

- (iii) Samples that can be classified as totally unknown occur rarely, since materials are usually not picked at random for analysis. The situation does arise, however, when one material behaves differently from another and the reasons for the differences are sought. Such samples may be the result of laboratory experiments or may be associated with a device or process that has changed or even failed for some unknown reason. The individual initiating the work may not even be aware of the right questions to ask in category (a) and must therefore work with the analyst to define a set of unique material characteristics which can be used both to classify the specimen and also to contribute to an explanation of its behavior. Although problems in this class are sometimes amenable to quick solutions, they tend more generally to be the most challenging and expensive ones to deal with, since they frequently involve the use of multiple techniques and often the samples are not optimized for those techniques. An example of this class of analysis would be a request to analyze a catalytic material obtained from a new vendor which, although nominally of the same composition as the previously used material, dramatically changes the rate of a chemical reaction. Also included in this class are problems in failure analysis such as determining why a turbine blade has failed prematurely. In this situation questions in category (b) relating to the sample environment can be extremely important.

4. Choosing a Sample and a Method

The quality of an analysis can be no better than that determined by the sample on which the analysis is performed. In trace level determination, a sample can easily be contaminated by handling or by the use of unclean glassware or other laboratory supplies. Samples can also be affected by exposure to the atmosphere, as in the case of hygroscopic materials or hydrocarbon buildup on a sample to be studied by surface analysis techniques. Materials are also not always as homogeneous as expected. If a method is used with a precision of $\pm 1\%$ but the composition of a randomly picked sample varies by $\pm 10\%$, obviously the sampling error will dominate. This effect can take place at either the macroscale or the microscale. In other words, a 1 kg sample taken from a single location in a tank car can lead to the same magnitude of error in assessing the overall contents of the car as using a 1 ng microprobe measurement to determine the composition of a highly segregated alloy ingot.

Collecting samples from enough regions of the material to be analyzed to determine its homogeneity and/or average composition is an essential part of characterization measurements. If the method of

analysis to be used in a particular problem is known in advance, as in the case of a research experiment, samples should be prepared so that they are truly representative of the problem and also of the proper size and geometry to be compatible with the method to be used. For most methods there is some correlation between sample size, sensitivity, accuracy and precision. Situations where the amount of sample is limited are frequently encountered, however, and in these cases the quantity of sample may strongly influence the analytical method selected and minimum detectability limits. Consider the following:

- a geologist working in an inaccessible location gathering samples to map out its mineral content
- a forensic scientist working with only a small piece of evidence
- a metallurgical failure analysis expert working with a fragment of a broken device
- a research chemist or metallurgist developing a new material in which it is important to explore hundreds of variations of composition and/or phase distribution

There are also times when an abundant amount of sample is available but either the concentration levels sought by a particular instrumental method are too low to detect because of maximum acceptable sample size limitations, or interferences from other constituents can introduce serious errors in the analysis. In such situations, determining whether the component of interest (phase or element) is dispersed homogeneously throughout the sample or is nonuniformly distributed is important not only from the sampling point of view but also as a means of selecting a technique to give enhanced localized analysis for inhomogeneous samples. Being able to detect 1 ng of material in a 1 g sample is a real challenge for any bulk analysis technique but is very simple by electron microprobe analysis if the relevant location is known. In the case of solids, this can often be done by microscopic examination of the sample either as it is or specially prepared to enhance phase differences. When possible, conventional light microscopy should always be tried first. Low-magnification observation of an unmodified sample often reveals whether a sample is nonuniform. If the sample consists of particles observable under a low-power binocular microscope, they can often be separated for easier analysis by a variety of methods including sedimentation, magnetic separation or even hand selection with tweezers.

If magnifications above $\sim 100\times$ are needed and the sample consists of discrete particles, scanning electron microscopy is usually a better approach. However, since the attribute of color is not available, differences between particles tend to be based on morphology and, in some cases, on atomic-number-dependent electron backscattering. Furthermore, since particles smaller than a few tens of $1\ \mu\text{m}$ are difficult to handle, the analysis may have to be done *in situ* using energy-dispersive x-ray spectroscopy. This method primarily

provides qualitative elemental analysis and can be applied to particulates using magnifications up to about $5000\times$ if the particles are well separated. Light microscopy is used in the magnification range of $\sim 100\text{--}2000\times$ primarily to examine polished sections by reflection for opaque materials or transmission for transparent materials.

While some chemical information is obtainable by polarized light and refractive index measurements, the presence of multiple phases may indicate that the sample should be analyzed either by a high-spatial-resolution instrumental method, like electron microprobe analysis, or infrared microscopy. Alternatively, it may be necessary to dissolve the sample matrix so as to isolate second-phase particles for analysis by other techniques. The transmission electron microscope extends the ability to perform combined microstructural and microchemical analysis to magnifications well above $5000\times$, but the complexities of specimen preparation and the relatively high cost and inaccessibility of this method in most laboratories, compared with light and scanning electron microscopy, tend to make it a method which is chosen only when the other methods are unsuccessful.

If by microscopic examination the sample appears to be fairly homogeneous, the advantages associated with the spatial selectivity of microanalysis cannot be realized and various methods of bulk analysis are probably more suitable. In addition to traditional wet chemical analysis, these methods include spark-source mass spectrometry, optical emission and absorption spectrometry and x-ray fluorescence spectroscopy. Each has a sensitivity extending down to the parts per million level and, in some cases, well below. In addition, the optical and x-ray techniques can both be made quantitative with relative ease. These techniques can often be applied to inhomogeneous samples, provided that no breakdown by phases is required. Figures 2, 3 and 4 give an approximate summary of the performance of some of the methods described, with regard to their ability to determine phase morphology, crystal structure and elemental composition, respectively.

The preceding comments about determining sample homogeneity refer principally to solid inorganic samples, although all the techniques mentioned can be used on polymers as well, within certain limitations associated with vacuum and electron beam compatibility requirements of the electron microscopic methods. Furthermore, x-ray fluorescence measurements are primarily concerned with elemental analysis rather than compound identification. Since carbon, hydrogen, nitrogen and oxygen are ubiquitous in organic materials, qualitative elemental analysis by x-ray techniques (hydrogen is undetectable by these methods) does little to aid in the identification of the material other than to show that it is organic. Determining whether an organic sample consists of one or more phases or compounds is also an important issue,

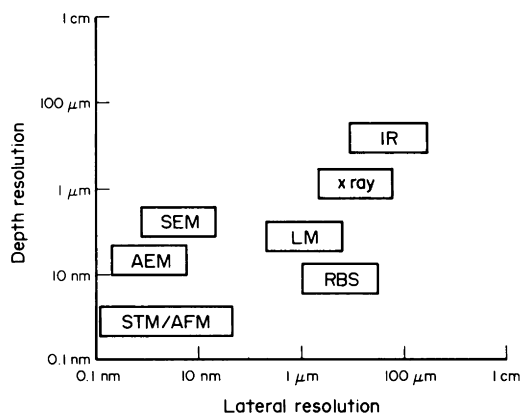


Figure 2
Lateral and depth resolution comparison for the methods indicated in Fig. 1 used for studying morphology

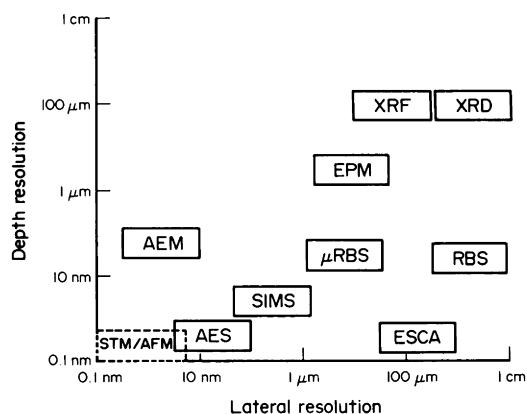


Figure 4
Lateral and depth resolution comparison for the methods indicated in Fig. 1 used for elemental analysis

however, but the types of samples, the separation techniques used and the kinds of information required can differ considerably from those associated with the examination of inorganic samples. First of all, most samples are either submitted as solutions or are taken up into solution before analysis. Separations are usually achieved by solution chemistry, solvent extraction or chromatography (gas or liquid). Separated fractions are either analyzed by direct introduction into a physically coupled instrument like a mass or infrared spectrometer or are collected and transferred to another location for analysis. If the sample is a polymer of only one type, the information sought may be molecular weight distribution and chromatography (gel permeation) may be all that is required. In

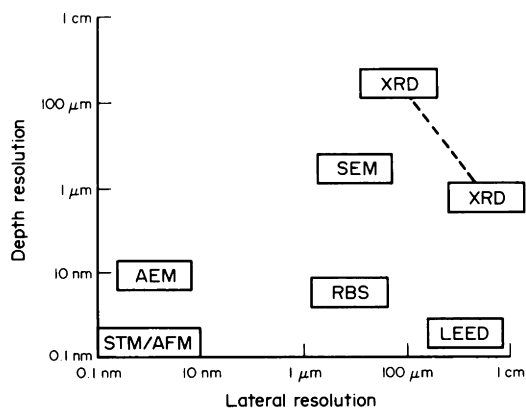


Figure 3
Lateral and depth resolution comparison for the methods indicated in Fig. 1 used for obtaining crystal structure information

other cases the information needed is the structural formula. This could be determined from information obtained by a variety of methods including mass spectrometry, infrared spectroscopy and nuclear magnetic resonance, as well as by traditional carbon, hydrogen and oxygen analysis based on combustion and gas absorption. The sample size and concentration required again become an issue, because the characteristics of the various techniques differ broadly. While nuclear magnetic resonance is excellent for structure determination, it requires larger samples than the other methods mentioned and can rarely detect compounds or functional groups at a concentration level less than 0.1%. Organic mass spectrometry, on the other hand, can work with much smaller samples and detect compounds even below the parts per billion level in a well-separated sample. Structural information is harder to obtain, however, and compounds with high molecular weights (> 2000) are difficult to analyze by conventional means. With systems of sufficiently high resolution and soft ionization sources, it is possible to obtain molecular ions and determine stoichiometry based on direct packing fraction measurements. In some cases, structure can also be determined using conventional electron impact ionization and comparing fragmentation pattern spectra with those of known compounds. Infrared spectroscopy can also have relatively high sensitivity to specific functional groups and can be used to examine relatively small samples, although larger ones are preferred for higher sensitivity. It is used more effectively in relating the spectrum of an unknown sample to a reference spectrum rather than as a means of determining the structure of a complete unknown. Of particular value is the fact that it can be routinely used to examine solid samples such as crosslinked insoluble polymers.

5. Cost and Complexity

There can be an enormous range in the costs associated with materials characterization, depending on the complexity of the problem, the amount of information sought, the number of samples to be run, how quickly the analysis has to be done and the investment in equipment and manpower. Multielement analysis for large numbers of routine samples can be effected for less than US\$25 per sample, while complex surface analysis can exceed US\$1000 per sample. Access to a particular type of instrumentation of the types described so far does not provide all the information needed about whether a given technique will have the sensitivity, resolution or sample handling capability to do a given job. It is possible to purchase complete mass spectrometer systems for under US\$5000 or over US\$500 000. While the former may be perfectly adequate as a residual gas analyzer where unit mass resolution is sufficient, it would be totally unsatisfactory for the analysis of high-molecular-weight organic compounds. Even if the larger sum of money were available, a high-resolution mass spectrometer for isotopic analysis would be decidedly different from one used for combined gas chromatography mass spectrometry.

The trade-off between cost and performance features is characteristic of all major pieces of analytical instrumentation. To deal effectively with this issue when selecting an instrument to purchase or use for a given application requires the development of a specification for instrument performance relative to intended use. Again taking mass spectrometry as an example, if the need is for high-sensitivity, high-resolution analysis of gas chromatograph effluents, an instrument might be specified with a highly efficient electron impact source coupled to a fast-switching, double-focusing analyzer with high-sensitivity electrical detection. Furthermore, it should also be possible to specify a generally accepted performance standard. For example, the instrument should be able to detect 10^{-9} C of ion per μg of methyl stearate at a mass resolution of 1 part in 10 000 and a source voltage of 8 kV. Generally, when selecting an instrument of any type, cost rises with performance. It is therefore incumbent on the buyer to be realistic about how many special features and what level of performance are needed for a given range of applications. It makes little sense to pay the added cost for a scanning electron microscope capable of $100\ 000\times$ magnification relative to a lower resolution model if the details of interest are clearly observable at $5000\times$ or if the nature of the sample is such that pictures at higher magnifications are not possible.

6. Trends

The major impact of computers and microelectronics on materials characterization techniques, already

alluded to, will undoubtedly continue for a number of years. In addition to instrument control, data collection and the processing of results, computers will play a more important role in method selection and the management of laboratory information. It can be expected that more general spectral databases and efficient means for searching them will lead both to faster and to more complete results. Since specimen preparation is often a rate-limiting step in many forms of analysis, laboratory robots are now being used more extensively to also improve quality and productivity.

In very general terms, most analytical instruments can be thought of as consisting of a source, an analyzer and a detector. As was shown in Fig. 1, the source can be used on the interaction of the sample with probing radiation of any wavelength or particles of different types and energies. It may even be the specimen itself, as in the case of the analysis of radioactive samples. The analyzer serves as a means of separating the signals emitted or scattered by the sample, based on differences in their physical or chemical properties, and finally the detector converts the separated signals into a comprehensible form. A major trend, which emerged during the 1980s, is to try all manner of combinations of sources, analyzers and detectors and thereby create instruments which are optimized for particular applications. In organic mass spectrometry, for example, ion sources are based on electron impact, field ionization, field desorption, laser ionization and both ion and neutral particle beams. In gas chromatography, detectors include flame ionization, electron capture, thermal conductivity and infrared spectrometry.

If the sample is only partly consumed or not consumed at all by a given detection process, multiple detectors can be used in series to provide complementary information. This has been done in both gas and liquid chromatography. Sometimes the detector is itself a spectrometer and thus allows separated species to be characterized more fully. Examples include gas and liquid chromatography-mass spectrometry and both gas and liquid chromatography-infrared spectrometry. An important characteristic of spectrometric detectors used in combination methods is that they must either be very fast or function in a parallel detection mode. Thus, in the examples just mentioned, a mass spectrometer capable of scanning its complete mass range in a few seconds is used in one case and Fourier transform infrared spectrometry in the other. If separation techniques are to be used with atomic emission techniques, some of the newly developed types of parallel detection optical spectrometers will prove very valuable. In other analytical situations, several detectors capable of monitoring a variety of signals are used at the same time. For example, simultaneous x-ray and electron detection in scanning electron microscopes and analytical transmission electron microscopes makes it possible to

relate chemical to microstructural information easily.

It is always difficult to predict where any field will go in the future, but clearly defined needs coupled with active technologies can provide some hints. All analytical methods share the common goals of higher speed, lower cost, greater sensitivity, more flexibility and increased accuracy. Since the early 1970s, remarkable progress has been made in advancing localized elemental analysis capability to the limit of single-atom detection by atom-microprobe analysis and ultrahigh-resolution scanning transmission electron microscopy, although only limited kinds of sample are suitable for these methods. Atomic resolution characterization of surfaces is now possible as a result of recent development of the scanning tunnelling and scanning force microscopes (STM and AFM). These instruments provide a relatively low-cost way of imaging many surfaces directly with atomic resolution. Unlike electron microscopes they utilize very fine physical probes and do not need to operate in a vacuum environment. In addition to surface morphology characterization, variations of the technique offer promise in the measurement of localized physical and chemical properties. The position of STM/AFM techniques in the lower left of Figs. 2–4 is indicative of their high potential to expand the frontiers of materials characterization. There is still a need, however, for higher spatial resolution techniques that can identify compounds, particularly organics, since a knowledge of elemental constituents is usually of little value in distinguishing one material from another. Selected area diffraction and, more recently, micro-diffraction are routinely used to identify inorganic compounds in transmission electron microscopes. The newer method has even given results from areas less than 5 nm in diameter. However, these approaches are of very limited value in the examination of organic materials, because of electron beam damage and the fact that many organics of interest are in a noncrystalline form.

The full characterization of thin organic films even when lateral spatial resolution is not an issue is complicated for other reasons. X-ray photoelectron

spectroscopy has already proved itself to be a powerful tool, but the chemical shift information contained in a spectrum is often difficult to extract because of the inherent energy resolution limits of the technique. Perhaps one of the most interesting and challenging problems is the atomic level characterization of interfaces within materials, where any attempt to reach the interfaces by material removal, as is done in secondary-ion mass spectrometry and Auger electron analysis, so alters them as to make the information obtained useless. For these kinds of problems, non-destructive techniques like solid-state nuclear magnetic resonance and some forms of laser spectroscopy may provide valuable solutions.

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A

Acoustic Emission

Acoustic emission (AE) is the transient mechanical vibration accompanying a sudden release of stored elastic energy or chemical free energy. It occurs as a consequence of the imposition of environmental stress on materials. The AE response of a large number of materials has been studied as a function of mechanically, thermally or chemically induced stresses. Detection, analysis and interpretation of AE from materials, parts and structures forms the basis of this nondestructive evaluation (NDE) method.

AE originates from a variety of sources in materials. These include localized, rapid movements of material, such as initiation and propagation of cracks, twinning, slip, martensitic phase transformations and void coalescence. Any similar dynamic and localized movement of material will also act as an AE source. Processes that occur via atom-by-atom diffusion are not usable AE sources. Dislocation motions, except for cooperative motion of many dislocations, are not usable AE sources. Matrix crazing, fiber-matrix disbonding, fiber fracture and delamination are sources of AE in fiber composites.

AE sources tend to be localized events in materials. AE precedes gross failure of the material when it is used in a part, component or structure and thus AE can be an effective NDE tool when properly detected and analyzed.

1. General Features of AE Signals

AE signals are stimulated in materials by mechanical, thermal or chemically induced stresses. Each material exhibits its own unique AE response characteristics. However, two broad types of AE response can be described which serve as useful qualitative descriptions: continuous AE and burst AE. Continuous AE has waveforms similar to Gaussian noise and is generally associated with the more ductile materials. It changes in average amplitude, but not in general appearance, as the stimulation parameters of the workpiece being studied are changed. It tends to show a large increase in average amplitude when a material yields, either locally or in a gross fashion. Continuous AE is believed to be made up of a large number of very small vibratory transients, randomly distributed in time. Burst AE has larger amplitudes and the burst events are sufficiently separated in time that they appear to be discrete events. Burst AE is usually observed when brittle materials or materials with brittle components are being studied. Stable crack growth in high-strength steel and fiber fracture in

composites generate burst AE. Burst AE is the more useful type for NDE applications because it is more closely associated with processes that can lead to local or catastrophic failure.

Materials in the mid-range of ductility frequently show predominantly continuous AE at low values of stimulation, then burst AE appears as the stimulation increases and failure of the workpiece approaches. Matrix crazing in composites frequently has the appearance of continuous AE, whereas fiber fracture produces burst AE.

2. Unique Features of Acoustic-Emission NDE

Most NDE methods depend on injecting a directed beam of energy into a material. This is the case, for example, in the x-ray, ultrasonic, eddy-current and microwave methods. An analysis of the scattering, attenuation or absorption of the injected beam as it interacts with a discontinuity in the material is then required in order to describe the discontinuity, the output beam being received by an appropriate sensor. The material of the workpiece being studied plays an entirely passive role in this process, although the material and its characteristic defect distribution significantly influence the choice of NDE method. The same form of energy is received in each of these cases as is injected.

AE is stimulated by an entirely different form of energy than is received at the sensor for analysis. The workpiece must have mechanical stresses induced into it either by mechanical, thermal or chemical means. AE, an acoustic (usually ultrasonic) transient vibration, is generated when a discontinuity suddenly releases all or a portion of the stress induced into it. The material, its discontinuity and the workpiece geometry determine what the output signal will be. The AE generated by a particular discontinuity under a given set of stimulation conditions contains unique information concerning the changes that occurred at the discontinuity. Note that it is only changes that can be discerned with AE, thus it is a dynamic NDE method, as well as being a method that is intensively related to the specific material being tested.

AE has serious disadvantages as well as distinct advantages when compared with other NDE methods. It can only detect discontinuities that cause abrupt changes in their surrounding stress fields as a result of stimulation. These discontinuities are very likely to be the most serious ones in a particular workpiece, but large discontinuities that are not revealed by an AE test need to be considered seriously, especially in the case of fatigue service. The

stimulation required must be greater than that expected in service, to ensure that existing discontinuities are not serious, even for static service. If mechanical loading is the stimulation, for example, equipment foreign to, and sometimes unavailable to NDE groups will be required. Furthermore, AE is inferior to other NDE methods in its ability to describe the size and shape of a discontinuity; x-ray and ultrasonic methods are superior. However, AE has the advantage that a discontinuity can be located by using multiple sensors (as described in Sect. 4) without a long, detailed and expensive search, if the discontinuity is an AE source. AE can also detect discontinuities that are below the detection limit of other NDE methods by at least an order of magnitude; an example is the formation of a crack embryo due to a breakaway of a dislocation pileup.

3. Analog-Instrumentation Methods

AE is most frequently detected using a piezoelectric sensor (transducer) which is attached to the workpiece via a good ultrasonic couplant. Its purpose is to convert the AE, a transient mechanical vibration, into a corresponding electrical signal. Amplification of the sensor output is normally performed in two stages. The output first goes to a preamplifier of high quality (low noise input), where the signal is amplified by a factor of 10–100. The preamplifier output should be capable of driving a long cable and should be matched to a second amplifier where further amplification is performed. The amplified signal may then be processed in a variety of ways to produce a record upon which a quality evaluation of the workpiece under study can be made.

A classical way of producing an AE record is to simply generate a record of the total AE counts as a function of the stimulation parameter. To do this, the amplified signal is fed to an electronic counter with an adjustable trigger. The trigger is set at a value slightly above, usually twice, the ambient electronic noise level of the amplification system. A plot may then be produced that shows AE count summation as a function of the stimulation parameter. A variation of this method is to reset the counter after a predetermined short period of time. It is then possible to generate a record of the AE count rate as a function of the stimulation parameter.

A simple way of producing an AE record is to feed the amplified signal to a root-mean-square (RMS) meter. The average RMS level is indicative of the amount of continuous AE; spikes on the RMS output are indicative of burst AE. Again, the AE record is correlated with the stimulation parameter to produce a test record.

The dynamic range of signals from an AE test can be very high, and sometimes exceeds 60 dB. For this reason, logarithmic amplifiers, rather than

linear amplifiers, are sometimes chosen. The data-acquisition procedures described may all be used in either the linear or logarithmic mode.

4. Digital-Instrumentation Methods

The increased availability and versatility of micro-computer, minicomputer and computer components have led to a significantly enhanced capability for extracting information from AE signals. However, since a single AE test may involve millions of signals, innovative methods of extracting the most significant information from individual AE signals are still necessary.

Commercially available systems can record signal features such as time of occurrence, rise time, duration, peak amplitude, peak dwell time, fall time and stimulation-parameter information. This information can be stored digitally and the information can then be used for both in-process and post-process analysis of AE data.

Amplitude-distribution analysis has proved to be effective in a number of circumstances. In this technique, the number of signals whose amplitude exceeds a prescribed value is plotted against amplitude. Alternatively, the absolute value of the derivative of this curve can be plotted. For example, amplitude distributions have been used to reveal multiple AE sources in materials and to detect the operation of a new AE source, perhaps the one that may lead to material failure, as the stimulation parameter is increased.

Both rise time and peak dwell time can be used to separate real AE from extraneous signals of similar appearance. Electrical transients tend to have very fast rise times and short dwell times, whereas frictional rubbing phenomena tend to have slow rise times and long dwell times. Thus, ability to manipulate data with respect to these two parameters is a distinct advantage.

Flaw location, if the flaw is a source of AE, can be accomplished by a computer-aided multiple-sensor array. Assumptions implicit in a straightforward application of location theory are that the AE signal travels from the source to each sensor in the detection array without change in waveform or propagation speed. These conditions are seldom met exactly, but are approximated closely enough in many applications to make AE flaw location a powerful NDE tool. AE flaw-location techniques have become more sophisticated rapidly, advancing in step with advances in computer technology.

Ability to turn an AE data-acquisition system on and off is a distinct advantage in some tests. In fatigue tests, gating the system on during only the most significant portion of each loading cycle prevents mixing AE crack-growth signals with extraneous noise. Time gating is useful in monitoring percussion-welding or electric-resistance-welding (spot-welding)

processes, since it permits the isolation of the most important AE processes during the welding sequence.

Many attempts have been made to perform fast Fourier transforms of AE signals, and to analyze the signals in the frequency domain. While some attempts have been successful, most have not. This technique is complicated by the fact that the AE signal is severely modified as it passes from source to sensor. Work-piece geometry seriously modifies the signal, as do attenuation characteristics of the material.

5. Unique Capabilities of the Acoustic-Emission Method

AE inspection methods can be used in a number of situations where other NDE methods would be prohibitively expensive or totally impossible. In these inspection applications, the information which can be acquired is not likely to fulfill requirements of existing codes. In some cases, no such guidance exists for any NDE inspection method. Thus, new goals need to be set for the inspection and new procedures need to be established for interpreting the data in many cases.

AE can be used to monitor structures and machines continuously while they are in operation—a significant advantage for NDE. Examples are steam turbines, electric generators, circulating pumps, pipelines, valves, chemical plants, aircraft and offshore platforms. Studies are in progress to define procedures for comprehensive monitoring of nuclear reactors.

In-process monitoring of welds has also been perfected as an inspection method. Electric-resistance, percussion and tubing-pinch welds can be quality assured as they are made. Laser welds can be successfully inspected as they are made, and the technique is highly developed for welding cables to connectors. Successful methods have been developed for in-process inspection of welds in heavy-section steel, where multiple passes of weld material are laid down. There is a large cost advantage in detecting a bad weld under these conditions, since the cost of removing large amounts of material to repair the bad weld pass is saved.

In-process inspection of other manufacturing processes, such as punching, metal forming and casting, is under development.

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See also: Wood: Acoustic Emission and Acousto-Ultrasonic Characteristics

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Acoustic Microscopy

In acoustic microscopy, magnified acoustic images of the elastic structure of the surface or interior of a solid are produced by passing high-frequency focused acoustic pulses through the material and displaying the received signals in image form as shades of gray. Usually, much higher frequencies are used than in other kinds of acoustic imaging. The acoustic pulses are focused directly, or the energy used to produce the pulses is focused into a very small region. When a small area of a specimen is scanned with a series of focused pulses and the transmitted or reflected signals are viewed on an imaging system (a television picture tube, a gray-scale recorder or some other scanned display), the magnification of the resulting image is equal to the ratio of the diameter of the display to the diameter of the area scanned. This procedure is also used in electron microscopy and scanning electron microscopy to produce magnified images by scanning a finely focused beam of electrons.

Acoustic microscopy has been used successfully in materials characterization and development as well as in biomedical applications (Lemons and Quate 1979). Because the displacement amplitude associated with propagation of high-frequency sound waves is at most a fraction of an angstrom, a poor chemical bond between two materials is readily detected by monitoring the reflectivity or transmission of signals at the interface. The cleanliness of homogeneous materials, the integrity of large-scale integrated circuits, the attachment of very thin films to supporting substrates, and the integrity of semiconductor-heat-sink bonds are also being evaluated with both reflection and transmission acoustic micrographs. Acoustic surface wave interference images are used to examine surface deformation—caused, for example, by abrasion, wear and machining—as well as the grain size of materials that are difficult to examine optically. The capability of acoustic microscopy to image small regions in which near-surface residual stresses have changed the elastic properties of materials has led to work on visualizing microstress fields. Because the acoustic contrast in biological specimens is often far greater than the optical contrast, a considerable body of work has been directed toward the imaging of cellular structure, collagen and other biological materials.

At present, acoustic microscopy includes several methods for the formation of magnified acoustic images. The four discussed here are scanning acoustic microscopy, photoacoustic microscopy, electron acoustic microscopy and scanning laser acoustic microscopy.

1. Background

In 1936, the Russian physicist Sokolov introduced an acoustic imaging device (the Sokolov tube) which

allows acoustic signals transmitted through an object to be displayed in real time on a television screen. The Sokolov tube is an image converter. The shadowgraph image is projected on a piezoelectric crosscut quartz plate one-half an acoustic wavelength in thickness. The back of the plate develops an electric charge image proportional to the acoustic amplitude incident on its front surface. As in all standard video-converter systems, the charged surface is scanned by a synchronized electron beam to provide the television display signals. Supporting the quartz plate between the water coupling medium and a high vacuum, however, is very difficult. The stress imposed by the atmosphere requires thick quartz plates with small surface areas. The thickness requirement limits the operating frequencies to less than 5 MHz. Unlike later micrographs, these unmagnified shadowgraph images were formed by near-field unfocused beams and showed very poor resolution. Because of the poor sensitivity of the original Sokolov tubes both the acoustic source and the object being imaged had to be placed very close to the imaging screen. In his original proposition, however, Sokolov had noted that the wavelength of sound in water at frequencies in the vicinity of 3 GHz would be comparable to the wavelength of light in the visible spectrum. Since the resolution of an imaging system is directly proportional to the wavelength of the illumination (or, in this case, insonification), he concluded that acoustic images could be formed equivalent in resolution to optical images. In fact, however, several decades of technology development were necessary before acoustic images could be produced with resolved detail equal to images formed with visible light.

The early 1970s were of critical importance to the development of acoustic microscopy. Of particular note was the formation of a research group at Stanford University under the direction of Quate. By 1974, the Stanford group had made Sokolov's speculation a reality (Lemons and Quate 1974). The images produced by the Stanford devices, later called scanning acoustic microscopes, were clearly competitive in resolution with optical microscopy images.

2. Scanning Acoustic Microscopy

Systems based on the Stanford design were being offered commercially by the early 1980s. In these devices, a highly focused sound source is scanned along a series of closely spaced lines and synchronized with the line-by-line scanning rate of an imaging system, usually a cathode-ray tube or a gray-scale recorder. Operating in either the pulse-echo or pulse transmission mode, the Stanford systems (see Fig. 1) use sputtered zinc oxide transducers to generate acoustic signals that are focused by sapphire lenses to focal spots 1 μm or less in diameter. Magnification is achieved by making the lines scanned by the focused

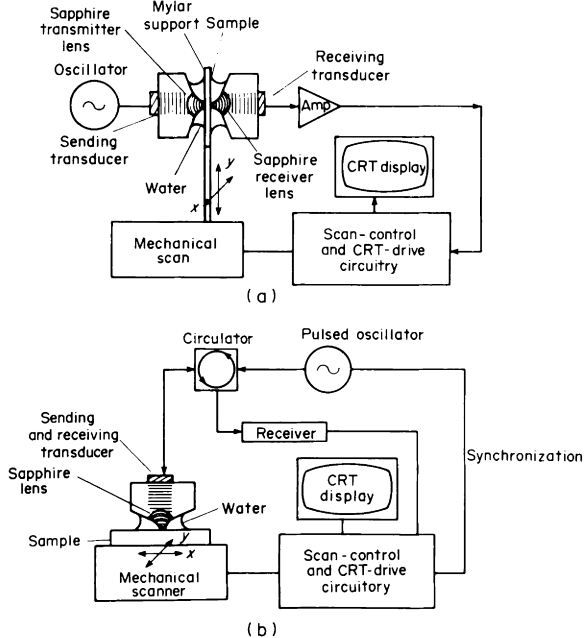


Figure 1
Scanning acoustic microscopes: (a) pulse transmission and (b) pulse-echo modes (courtesy of C. Quate and L. Lam, Stanford University)

transducer shorter and closer together than the lines that make up the image. Consider, for example, a $1\text{ mm} \times 1\text{ mm}$ area that is scanned by moving a combined acoustic source and receiver, each focused to $2\text{ }\mu\text{m}$ in diameter over 500 lines that are 1 mm long and spaced $2\text{ }\mu\text{m}$ apart. When the received sonic signal is displayed in 500 equally spaced lines on a $100\text{ cm} \times 100\text{ cm}$ imaging screen, the result is an acoustic micrograph with a magnification of $1000\times$. A $5\text{ cm} \times 5\text{ cm}$ scanned area displayed on the same $100\text{ cm} \times 100\text{ cm}$ screen generates an acoustic micrograph with a magnification of $20\times$.

The primary strength of the scanning acoustic microscope design is its remarkable versatility. Microscopes can be constructed for use at a wide range of frequencies for scanning objects over a wide range of sizes. Acoustic images generated by focused pulses from 1 GHz to 3 GHz typically scan a specimen area of $0.25\text{ mm} \times 0.25\text{ mm}$. They penetrate no deeper than $150\text{ }\mu\text{m}$ and the typical magnification is approximately $1000\times$. For industrial acoustic images, it is not unusual to scan an area $15\text{ cm} \times 15\text{ cm}$ with 10 MHz focused pulses, penetrating to a depth of 5 cm or more and displaying the received signals at magnifications of $2\text{--}3\times$.

Since the mid-1970s, most of the Stanford work has been directed at frequencies between 1 GHz and

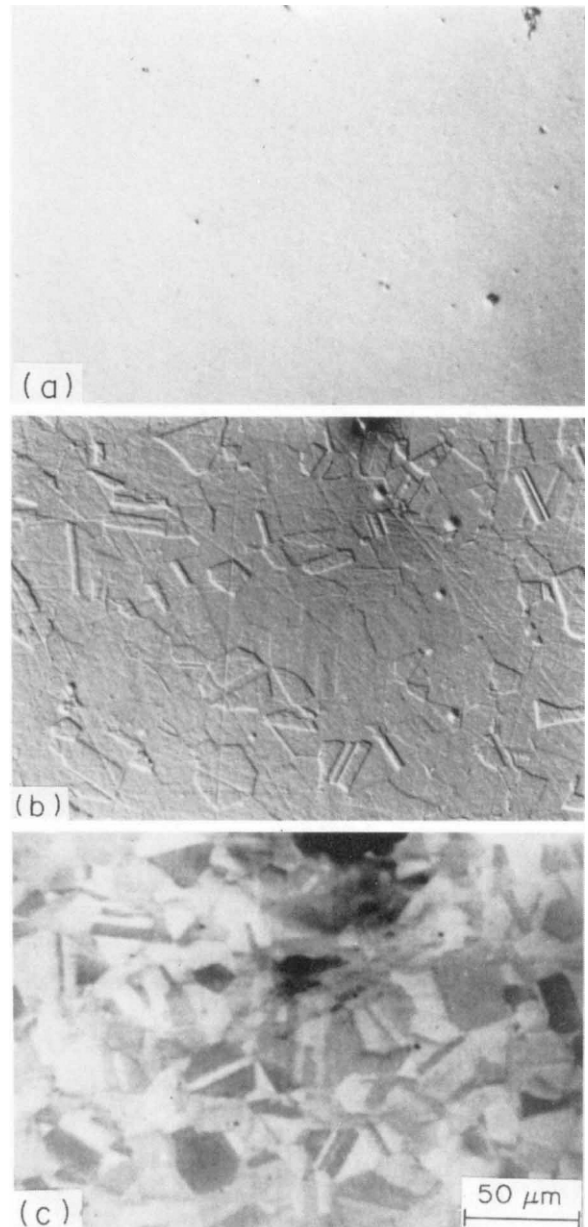


Figure 2
Images of grain structure in Inconel: (a) optical image, (b) optical image etched, and (c) 2.7 GHz acoustic image of the same region (courtesy of L. Lam, Stanford University)

5 GHz . At University College London work has been done at 150 MHz – 1 GHz (Ash 1980), and industrially oriented work at Hughes Aircraft Company (Weglin 1979), the General Electric Company and elsewhere has been done at $1\text{--}450\text{ MHz}$.