Highlights of the 1987 E-MRS Meeting

Symposia, Short Courses, Plenary Lectures, and a Special Session on High Temperature Superconductivity Featured

The 1987 Meeting of the European Materials Research Society (E-MRS) to be held June 2-5, 1987 at the Council of Europe in Strasbourg, France will feature three symposia, four invited Plenary Session lectures, a short course program offered for the first time in conjunction with an E-MRS meeting, and a special session on superconducting materials with high transition temperature.

Symposia

The symposia scheduled for the 1987 meeting are as follows:

- Symposium A: Photon, Beam and Plasma Enhanced Processing
- Symposium B: Growth, Characterization, and Processing of III-V Materials with Correlations to Device Performances
- Symposium C: Amorphous Hydrogenated Carbon Films

Proceedings of the symposia A, B, and C will be published by Les Editions de Physique, Avenue du Hoggar, B.P. 112, Zone Industrielle de Courtaboeuf, 91944 Les Ulis-Cedex, France; telephone 907 36 88. In the United States, contact Materials Research Society, Publications Department, 9800 McKnight Road, Suite 327, Pittsburgh, PA 15237; telephone (412) 367-3012.

Short Courses

The newly initiated short course program will offer two courses intended for physicists, students, and industry engineers. The course lectures are designed so they will not overlap with symposium sessions, allowing attendees to participate fully in the meeting. The courses are as follows:

- Near Surface Characterization of Materials Using MeV Ion Beam Techniques, chaired by G. Amsel (Groupe de Physique des Solides, Paris, France). The course outline includes an introductory lecture and lectures on Nuclear Reaction Analysis (NRA), Rutherford Backscattering and Recoil Analysis (RBS-ERD), channeling and surface scattering, and Proton Induced X-Ray Emission (PIXE).
- Characterization of Semiconductors by Scanning Electron Microscopy: Electron Beam Induced Current (EBIC), Cathodoluminescence (CL), and X-Ray Microanalysis, chaired by C. Donolato (CNR-LAMEL, Bologna, Italy). The program for this course includes introductory lectures, and discussions of physics principles, computer programs and modeling, specific applications, limitations, and recent developments.

Plenary Session Lectures

The Plenary Sessions will feature four invited lectures by Minko Balkanski, Jacques Derrien, Gottfried Landwehr, and Michael G. Somekh. The following extended abstracts summarize the Plenary lectures.

E-MRS
Discussion Meeting
Superconducting Materials with High Transition Temperatures

June 3, 1987
Strasbourg, France

E-MRS has added a one-day discussion meeting on superconducting materials to the symposia scheduled for the 1987 E-MRS Meeting held June 2-5 in Strasbourg. Invited talks will be given by B. Batlogg (AT&T Bell Laboratories, Murray Hill, New Jersey), J. Bednorz (IBM Research Laboratory, Zurich), and Paul C.W. Chu (University of Houston, Texas)

Contributed papers will feature theory and preparation methods, and will focus on structural, mechanical, and electrical properties of superconducting materials.

Structure of Fast Ion Conducting Glasses

M. Balkanski: Laboratoire de Physique des Solides, associé au Centre National de la Recherche Scientifique, Université Pierre et Marie Curie, 4, Place Jussieu, 75252 Paris Cedex 05, France.

For four thousand years mankind has used glasses in his everyday life and in the arts. Today many important technological developments call for more diversified and abundant uses of glasses. Tomorrow ultratransparent glass fibers will be transmitting more information than copper wires, and solid-state batteries will be using glass as an electrolyte. Yet glass is still one of the lesser known materials. The relationship between glass structure and its physical properties is still to be established.

Fast ion conducting glasses are used as solid electrolyte in solid-state integrable batteries (S_2IB). The optimization of this application depends essentially on two parameters: the glass should be an excellent electronic insulator and should, on the other hand, have good ionic conductivity. These two conditions should hold in the limits of very thin films.

Fast ion conduction is related to the glass structure. The effects of the glass matrix structure on the dynamical behavior in fast ion conduction is best seen when it is possible to modify the structure by an external parameter. Laser and temperature annealing create the conditions of structural changes. Laser annealing can be handled with sufficient precision in order to induce the glass-crystalline transition.[1] Depending on the incident laser energy density, three significant effects are observed by light scattering: (1) spectral band narrowing indicating cluster enlargement constitutes a *precursor effect*, (2) an *intensity increase effect* indicating a rapid rise of the density of clusters attending microcrystallite size, and (3) a *dynamical reversal effect* indicating glass crystalline instability. Cluster volume and crystallization appear as separate but related threshold phenomena.

Thermal annealing[2] is a more global process and the observations are less detailed. In comparing results from light scattering spectroscopy with those of complex impedence spectroscopy it is nevertheless possible to deduce, for example, the crystallization kinetics for Li doped fast ion conducting glasses. In this case it is useful to consider the glass matrix as a solvent and the doping salt as a solute. Depending on the Li concentration in the glass matrix or the doping additive, the solute may crystallize before the solvent, or inversely. The respective crystallization temperature of the solvent or the solute are in direct relation to the drop in ionic conductivity of the glass. This behavior also depends on the cation and anion sizes of the solute and their respective relation to the glass structure.

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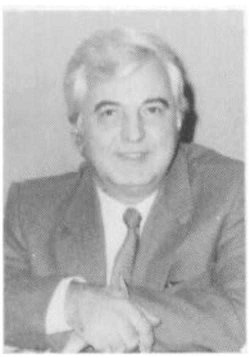


Minko Balkanski's studies began with the optical properties of semiconductors, phonon spectroscopy, and band structure. He later turned his attention to band structures of narrow-gap semiconductors, superlattices, ferroelectric semiconductors, and electron-phonon interactions. More recently he and his group have studied properties of amorphous silicon and gallium arsenide, time-resolved spectroscopic techniques for studies of nonlinear optical effects, and atomic motion in solids. Balkanski received the 1986 Von Hippel Award of the Materials Research Society.

Fine Structure in Electron Energy Loss and Auger Spectra: New Approaches to the Local Geometry Determination At Surfaces and Interfaces of Solids

J. Derrien: Centre National de la Recherche Scientifique, Laboratoire d'Etudes des Propriétés Electroniques des Solides, associated with Université Scientifique, Technologique et Médicale de Grenoble, B.P. 166, 38042 Grenoble Cedex, France.

The understanding of surface and interface phenomena is based on a fair correlation between the surface and interface physicochemical and crystallographic properties and their electronic properties. To look at the solid surfaces and interfaces on a microscopic scale, physicists and chemists are now using a great variety of sensitive techniques, which are very sophisticated and rather expensive.

Conventionally, Auger electron spectroscopy and electron diffraction techniques give information on the physicochemical and crystallographic behavior of the surfaces and interfaces while photoemission spectroscopy reflects the densities of occupied states of energy lower than that of the Fermi level ($E \leq E_F$).

One can easily see that at least two kinds of techniques are lacking: (1) For a surface or interface without a long-range order, where the diffraction techniques are unsuccessful, short-range order techniques need to be developed. (2) The band structure of a solid cannot be fully understood without information on the empty states extending above the Fermi level ($E \leq E_F$).

Recently, we have developed two new and simple techniques probing the local geometry of the sample, and a technique of empty states. All these techniques are used *in situ* and dedicated to surface and interface studies. We discuss in this lecture only the local geometry techniques based on fine structures observed in electron energy loss and Auger spectra.

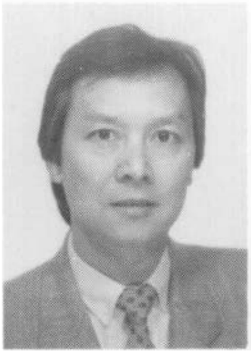
During the last decade, the EXAFS technique (Extended X-Ray Absorption Fine Structure) has made tremendous advances and is currently used for the local geometry determination of solids (nearest-neighbor distance and coordination numbers). This technique is based on the observation of fine structures oscillating in an extended energy range far from the core edge x-ray absorption of the sample. These oscillations come from an interference process between the wave function of the photoelectron excited by x-ray absorption from the core level, going out directly from the absorber atom and the wave function reflected by its nearest-neighbors.

Analysis of these oscillations permits the determination of the nearest-neighbor interatomic distance, their nature, and the coordination number. Development of the EXAFS technique has been greatly eased by high flux synchrotron radiation sources. Nevertheless, when dealing with surface and interface phenomena, the signal/background ratio is very small, and a study with surface EXAFS may last several hours, even with synchrotron radiation sources. Moreover, *in situ* surface EXAFS experiments with synchrotron are not easily accessible. In order to eliminate these drawbacks, we propose a simple alternative. If one analyses the energy distribution N(E) of electrons backscattered from a solid surface irradiated with an electron primary beam, one can observe oscillating fine structures in the energy range corresponding to losses due to core edges-ionization. These fine structures arise from the same interference process as previously described for EXAFS. Therefore, the local geometry can be determined by analyzing these oscillating fine structures found in loss spectra (Surface Extended Energy Loss Fine Structure, SEELFS).

In principle, the SEELFS technique offers good surface sensitivity, and applicability to both ordered and disordered surfaces. Moreover, it is a relatively simple, laboratory-based apparatus and therefore more accessible than surface EXAFS studies by synchrotron radiation.

In the first part of this lecture, the physical principles of the SEELFS technique, its capabilities and limits will be discussed. Several examples will be given, with particular attention to the local order of clean surface, adsorbate monolayer on clean surface, initial stages of metal-semiconductor interface formation, and metallic cluster growth on solid substrate.

In the second part of this lecture, we show evidence of extended fine structures observed above core-valence-valence Auger transitions in Auger spectra of several materials. We tentatively suggest their underlying physical origins and analyze these oscillating structures following the same standard EXAFS formalism. The lattice parameters deduced from these extended fine Auger structures (EXFAS) are in good agreement with those deduced from synchrotron radiation EXAFS data, conferring therefore to the conventional Auger technique new capabilities as a surface sensitive local order probe.



Jacques Derrien has been a professor of microelectronics at the University of Grenoble since 1983. He is the leader of the Surface and Interface Group in a CNRS Laboratory (LEPES). Since 1982, Derrien has served as chairman of the Surface and Interface Committee of the Groupe Francais de Croissance Cristalline, and since 1986 he has been a National Committee member of the Société Francaise de Physique. His research involves fundamental studies on metal-semiconductor and dielectric-semiconductor interfaces heterojunction and multilayer properties, using various surface techniques and molecular beam epitaxy.

Electronic Structure of Semiconductor Superlattices and Heterostructures

G. Landwehr: Physikalisches Institut, Universität Würzburg, West Germany.

The investigation of heterostructures, superlattices and quantum wells is at present probably the most active area of semiconductor physics. The interest in the subject has two roots: the new manmade structures are a hunting ground for physicists interested in fundamental physics, and there is a considerable potential for the realization and development of novel devices for both micro- and optoelectronics.

The activities in this new field of research are very substantial. Usually more than one conference on the subject is being organized per year, not to speak of workshops and summer schools. Consequently, it is impossible to give a comprehensive review in a single talk. Therefore, this talk will outline historical developments and concentrate on a few highlights.

The first published work on artificial semiconductor superlattices can be traced to 1970, when Esaki and Tsu[1] proposed a quasi-one-dimensional periodic structure consisting of very thin alternating semiconductor layers. These consisted either of different material or of subsequent n- and p-type layers of the same semiconductor. The idea was to achieve resonant tunneling in multiple quantum well structures. Obviously, the requirements for an abrupt transition from one layer to the other imposed

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very stringent conditions on the film growth techniques. Therefore, it took some time before structures with square well potential profiles could be grown. The technique which became the most useful for this purpose was molecular beam epitaxy (MBE), which allows producing films a few atomic layers thick in ultrahigh vacuum by controlled slow evaporation. It turned out that GaAs and AlAs have almost the same lattice constant and that it was possible to grow $Ga_{1-x}Al_xAs$ mixed crystals of high perfection on GaAs substrates. Because the thickness of the layers can be made small in comparison with the de Broglie wavelength, electrons trapped in GaAs potential wells are subject to quantum mechanical boundary quantization. As a consequence, superlattices and quantum well structures have a quasi-two-dimensional character because the electrons are free to move parallel to the interface, giving rise to the so-called electric sub-bands.

It is interesting to note that boundary quantization and electric sub-bands were first observed in single heterostructures, in silicon field-effect transistors (MOSFETs). Already in 1966, Fowler, Fang, Howard and Stiles[2] measured Shubnikov-de Haas oscillations in devices of this kind, clearly demonstrating that one was dealing with a two-dimensional system. Subsequent theoretical work by F. Stern on n-channel devices[3] (in which the electrical sub-bands and the relevant bandstructure parameters were obtained by solving self-consistently both Schrödinger's and Poisson's equations) allowed analyzing the experimental data in considerable detail. Not only transport measurements at low temperatures in high magnetic fields yielded information, but also optical and magneto-optical experiments.[4] Subsequent experimental and theoretical work on p-channel MOSFETs indicated that the high electric field present at the Si-SiO_2 interface profoundly influences the bandstructure, especially the effective masses.[5]

The shape of the potential well in a MOSFET can, in the first approximation, be considered triangular. A similar situation holds for a modulation doped GaAs-(GaAl)As heterostructure. In this type of device (realized by Dingle et al. in 1978)[6] a (GaAl)As film doped with Si is covered by a GaAs layer. The donor electrons spill over into the GaAs, forming a two-dimensional electron gas of exceptionally high mobility. If the doped layer is separated by a thin spacer of the same material and if the interface is of high perfection, mobilities above $2 \cdot 10^6$ cm²/Vs can be achieved. Using the same principle, p-type GaAs-(GaAl)As heterostructures with a hole mobility exceeding 10^5 cm²/Vs were produced.

Several unusual features were found when these structures were studied in detail: The Kramers degeneracy is lifted at finite wave vectors and the heavy hole band splits into two branches, one associated with a small effective mass, resulting in high mobilities.[7] When analyzing magneto-optical and magneto-transport data one has to take into account a change of the electronic band structure by high magnetic fields. The technical potential of modulation doped GaAs-(GaAl)As is obvious; the high electron mobility transistor (HEMT) is based on this concept. The existence of high mobility p-channel heterostructures has made complementary circuits feasible.

The first tunneling experiments by Esaki and co-workers in devices with several rectangular quantum wells clearly demonstrated the validity of the concept.[8] Compared with the MOSFETs and the modulation doped GaAs-(GaAl)As heterostructures, analysis of the experimental data based on the simple "particle in a box" concept is straightforward because only elementary textbook quantum mechanics is involved. However, complications do arise if one tries to calculate the electrical sub-bands derived from the degenerate valence bands. This holds especially if one tries to incorporate a strong magnetic field in the calculations.

The GaAs-(GaAl)As superlattice has been called type I. In a lattice of this kind the conduction band edge of the (GaAl)As is above that of the GaAs and the valence band edge is below the corresponding one in GaAs. The relative position of the band edges is hard to determine beforehand. From the first optical experiments performed on single quantum well structures by Dingle and co-workers in 1974,[9] it was deduced that 85% of the band offset could be attributed to the conduction band and 15% to the valence band. Several recent experiments have required revising these numbers.

In the last decade, a wealth of information has been obtained on GaAs-(GaAl)As superlattices. Modern MBE technology has allowed varying the width of the potential wells and their distance in a systematic way. In this way the coupling between the potential wells could be changed at will and studied by various techniques. Unfortunately it is not possible to discuss the many relevant experiments made so far.

Although InAs and GaSb are not perfectly lattice matched, it is nevertheless possible to grow superlattices of reasonable crystallographic quality on GaSb substrates. In this kind of superlattice, the conduction band edge of the InAs is located below the valence band maximum of the GaSb. This leads to a different kind of superlattice (type II) in which electrons and holes reside in the different materials close to the interface. As in a single heterostructure, the bands are curved in this region due to the presence of space charge. Type II superlattices have interesting properties.[10] It is obvious, however, that the theoretical treatment is difficult because the matching of the wave functions at the interface is by no means trivial, and because the band offset is not known *a priori*.

Still another kind (type III) of superlattice is possible if a zero-gap material like HgTe is combined with a semiconductor like CdTe. Structures of this kind have been produced by Faurie and co-workers.[11] Experimental studies have shown that such devices have a significant band edge absorption and that they have potential as infrared detectors. The existence of quasi-interface states has been postulated for these superlattices.

Another interesting recent development is connected with strained superlattices, for example, the Si-(SiGe) system. The difference in the lattice constant of these two elemental semiconductors is about 4%. If Ge films are grown on an Si surface by MBE, misfit dislocations do occur at the interface. If, however, a GeSi alloy is deposited, a dislocation-free layer can be grown, which is lattice-matched to the Si. The thickness of the dislocation-free film increases with decreasing Ge content. In this way superlattices with novel properties can be produced.[12]

Finally, the recent progress in semiconductor superlattices and heterostructures has been possible only because of significant advances in crystal growth techniques. Not only has MBE made rapid progress, alternative techniques like metalorganic chemical vapor deposition (MOCVD) have been developed further. The remarkable success in the field has been possible only because of the close cooperation between materials scientists and fundamental researchers.

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Gottfried Landwehr holds a chair for experimental physics at the University of Würzburg. From 1953 to 1968 he was a research scientist at the Physikalisch-Technische Bundesanstalt in Braunschweig. From 1959 to 1961 he was research assistant professor of electrical engineering at the Semiconductor Laboratory of the University of Illinois at Urbana-Champaign. Between 1978 and 1983 he was the director of the High Magnetic Field Facility of the Max-Planck-Institut für Festkörperforschung in Grenoble, France. Landwehr's main research activity is semiconductor research, especially in conjunction with high magnetic fields and two-dimensional systems.

Acoustical Imaging: Past, Present, And Future

M.G. Somekh: Department of Electrical and Electronic Engineering, University College London, Torrington Place, London WC1E 7JE, United Kingdom.

This paper will present an overview of the field of acoustic imaging with particular reference to applications of the scanning acoustic microscope to materials science and the semiconductor industry.

In order to understand the possible applications of high resolution ultrasonic imaging, the paper will be introduced with a brief survey of the physical interactions of ultrasound with materials. The direct interaction of ultrasound with the mechanical properties of materials, together with the realization that the wavelength of ultrasound in most materials is of the order of a micron at microwave frequencies, will explain the motivation of several generations of research workers who attempted to obtain acoustic imaging with resolution comparable to that of the optical microscope. We will summarize some early developments, showing how the present configuration developed by Quate and his student Lemons is an extraordinarily simple and elegant solution to the problem of obtaining diffraction limited resolution using acoustic waves.

The general configuration of a modern high resolution acoustic microscope is described, and the factors limiting the resolution and picture acquisition time are presented. We will explain how present instruments can obtain submicron resolution (operating frequency $\simeq$2 GHz) using water to couple the acoustic energy between the sample and the specimen. Although better resolution is possible, the attenuation in the coupling fluid means that quantitative information is much harder to extract at higher frequencies. Resolution rivaling that of the scanning electron microscope is possible using exotic coupling fluids such as liquid helium where the fluid attenuation is very small. However, despite the excellent resolution, the interaction of the sound with the sample surface is so small that most of the contrast may be attributed to topography rather than mechanical properties.

The precise form of the microscope depends on the application and the resolution. For operating frequencies above 500 MHz the instrument may truly be called a microscope, acquiring images with optical resolution in approximately 10 seconds. Operation at lower frequencies requires scanning through a considerably greater distance, thus placing mechanical constraints on the maximum speed of scanning. The low frequency acoustic microscope may be better thought of as a high resolution nondestructive testing imaging instrument, as the spatial resolution is generally several tens of microns.

Just as the structure of the instrument depends on the frequency of operation so in general does the application. We discuss materials applications of both the high and low frequency instruments. The low frequency acoustic microscope generally operating at about 50–100 MHz has found considerable application for subsurface imaging of optical opaque materials. In this implementation, acoustic lenses with small aperture angles are generally used so that good penetration into the material is achieved. Subsurface imaging applications of voids, diffusion bonds, and delaminations in integrated circuit packages are presented. We compare these results with those obtained using thermal imaging and x-ray radiography.

Wide angle acoustic lenses usually operating at much higher frequencies (although some wide angle low frequency results are also presented) are usually used for high resolution surface and near-subsurface imaging. Wide angle acoustic lenses not only give better spatial resolution because of higher numerical aperture, but can show features not visible optically because a unique contrast mechanism operates involving excitation and re-radiation of Rayleigh or surface waves. Provided the aperture angle of the lens is greater than the critical angle for the excitation of these waves (typically about 30 degrees), they will be excited on the sample prior to propagating along the surface re-radiating their energy continuously. The re-radiated energy from the sample surface is detected by the lens which is sensitive to the phase of the returning radiation. This mechanism means that the acoustic lens acts as an interferometer between the rays incident on the sample at near-normal incidence and those exciting surface waves. Contrast on a sample is thus very sensitive to the elastic properties of the material, allowing very accurate determination of the surface wave velocity.

The presence of surface waves on the sample also means that defocusing the microscope does not blur the image in the same sense as in optical microscopy but primarily changes the relative contrast between different features. The optimum contrast in an image can therefore be obtained merely by taking a series of images with varying lens sample separation.

In addition, the presence of the surface wave on the sample surface means that any feature on the sample, (such as a crack, grain boundary, or inclusion) strongly affects the propagation of the surface wave, thus modulating the image intensity in that region. The acoustic microscope is therefore particularly sensitive to the presence of surface breaking discontinuities, being capable of detecting features many orders of magnitude smaller than the acoustic wavelength. A series of images showing how the microscope indicates the boundary conditions at the discontinuity is presented. Images showing the sensitivity to surface breaking discontinuities are presented on several materials.

The propagation of surface waves is also severely disturbed by the presence of delamination and impaired bond in layers within a Rayleigh wavelength of so of the sample surface. Results are presented which demonstrate the sensitivity to near-surface features. In particular we (1) show that failures in integrated circuits caused by electrical overstress, which are difficult to observe by other means, can be imaged in the acoustic microscope; and (2) discuss an experimental and theoretical approach to measuring bond strength.

Several more recent developments in acoustic imaging at University College and elsewhere are presented. These include quantitative determination of spatial variations of mechanical properties and the use of frequency coding techniques to separate the effects of surface topography from specimen reflectivity. We also show the results of some recent attempts to improve the spatial resolution of the microscope by frequency modulation, and demonstrate how such techniques can greatly relax some of the technological limitations on the design of the instrument.

In the final part of the paper we return to our discussion of the development of ideas in the field, but at this stage we look not at developments in the past but to those we anticipate in the future. Among these is the

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tendency toward quantitative imaging, which is perhaps ultimately where acoustic microscopy has advantages over other new forms of microscopy in which exact theoretical modeling is much more difficult. Another interesting trend is the application of many of the ideas developed for acoustic microscopy to nondestructive evaluation. The SAM, which in many ways arose from more conventional NDE techniques, has developed where it is now in a position to return ideas to the original field. Finally, we review the contribution that acoustic microscopy may be expected to make to materials research in the next few years.

Michael G. Somekh was appointed lecturer in the Department of Electrical and Electronic Engineering, University College London in 1985, and in 1986 he became director of the Wolfson Unit for micro-NDE, a small consultancy group applying new techniques developed within the department to the problems of industrial clients. His research activities in scanning acoustic microscopy particularly involve developing frequency coding techniques to improve the quantitative information obtained from the microscope. In addition, his interests include scanning optical microscopy and thermal imaging, especially for semiconductor characterization.