

## LABORATORY FACILITIES IN THE MILTON S. EISENHOWER RESEARCH CENTER

The Milton S. Eisenhower Research Center carries out investigations that contribute to contemporary science, and it also serves as a resource for other departments at APL. In addition, the Research Center has acquired a complete spectrum of modern instrumentation for analysis and research. It currently has over 30 laboratories, occupying approximately 10,000 square feet, equipped for modern physical research.

### ANALYSIS CAPABILITIES

#### Composition

Atomic composition is ascertained either by chemical means or by a variety of analytical spectroscopies. An *Auger electron spectrometer* can provide surface and depth composition information, as well as micrographs for topical information and spatial mapping of selected elements on the surface. A *scanning Auger PHI 545M microprobe* is available for measuring elemental composition to sensitivities of about 0.1 percent of a monolayer with a spatial resolution of 3 micrometers.

A *secondary ion mass spectrometer* (GCA IMS 101-B prototype) is also available that can measure composition with greater sensitivity than the Auger system. It has a unique energy window feature that provides good discrimination between atomic and polyatomic ions and has also been modified to permit ion-acoustic imaging studies of materials.

An *ETEC scanning electron microscope* has been modified to permit simultaneous thermoacoustic imaging studies, together with secondary electron and back-scattered electron imaging. It is also equipped with an energy dispersive X-ray detector for localized elemental analysis of specimens. In the conventional scanning electron microscope mode, the instrument is capable of 70-angstrom resolution with an ion pumping system to reduce sample contamination. An *atomic absorption spectrometer* is available for measuring elemental composition or impurities in solids.

Two medium-resolution *DuPont dual sector mass spectrometers* are in place to analyze gaseous, liquid, or solid samples over a 4 to 2400 mass range. Both electron impact ionization and chemical ionization can be used on these units, which are interfaced to a computerized data system that facilitates analysis and compares unknown spectra to a library of 35,000 compounds. An *HP-5970B mass spectrometer* and *HP-5890A gas chromatograph* combination is used for chemical species identification.

High-resolution nuclear magnetic resonance spectra can be observed for both proton and fluorine nuclei

in a liquid sample in a *Varian EM360L spectrometer*. Sample temperatures can be varied from  $-100$  to  $175^{\circ}\text{C}$ .

#### Structure

The detailed investigation of structure is conducted using X-ray scattering measurements. Both *wavelength-dispersive* (monochromatic X-ray source/multipositional film or counter collection of scattered beams) and *energy-dispersive* (polychromatic X-ray source/fixed angle processing of scattered beams by solid-state detector/multichannel analyzer electronics) instruments are used. The two procedures are complementary in that the wavelength-dispersive technique permits a wide-angle, high-resolution investigation of the scattering pattern, while the energy-dispersive technique allows a rapid, lower resolution investigation that is capable of yielding radial distribution functions and kinetic data, for example, on annealing transformations from an amorphous to a polycrystalline microstructure. The former is based on the use of a *Syntex P3M X-ray autodiffractometer* with a low-temperature chamber, while the latter is a *Seifert-based system*.

Surface structural information on single-crystal systems is obtained using current image diffraction and low-energy electron diffraction installed on the *PHI 545 scanning Auger microprobe*.

#### Transport

Extensive transport measurements to examine electronic properties are conducted to probe the behavior of new materials. These transport measurements include resistivity, Hall effects, and magnetoresistance with a wide range of temperatures (1.2 to 300 K), magnetic fields (0 to 60 kilogauss), and frequencies (0 to 100 kilohertz). Four probe electrical resistance measurements are performed employing two cryostats, one for zero-field measurements and the other for measurements in the presence of an external magnetic field. Instrumentation for noise measurements is also available. All operations are computer controlled, allowing precise measurements with high-temperature resolution ( $<0.1$  K).

A time domain spectrometer is available that can measure the complex dielectric constant (conductivity) of a solid from 0 to 20 gigahertz using fast Fourier transform techniques.

## Magnetic

Static magnetization measurements are being performed on an *S.H.E. Corp. variable-temperature superconducting magnetometer* (at The Johns Hopkins University). The instrument, capable of measurements between 2 and 400 K in the field range of 0 to 50 kilogauss, has an ultimate sensitivity equivalent to a change in mass susceptibility of  $10^{-11}$  electromagnetic unit per gram in fields as small as 1 gauss. The stability, range, and sensitivity of the instrument allow precise static magnetization measurements on all materials.

Dynamic magnetic measurements are obtained using spin resonance techniques in broad temperature (4 to 300 K) and frequency (2 to 35 gigahertz) ranges on a modern *computer-controlled Varian spectrometer*. Frequency-dependent effects in small fields are studied via alternating-current susceptibility measurements using a mutual inductance bridge. Easy and rapid temperature control is available at frequencies ranging from a few hertz to several megahertz.

A broadband high-sensitivity *nuclear magnetic resonance spectrometer* is used to observe the nuclear magnetic resonance signals from a variety of nonzero spin nuclei. This spectrometer is applied to nonhigh-resolution types of spectroscopy on solids, liquids, and gases such as chemical shift determination ( $> 50$  milligauss), isotope ratio determination, relaxation time ( $T_1$  and  $T_2$ ) measurements, gyromagnetic ratio measurements, and nuclear quadrupole effects.

The laboratory also contains a *Mössbauer system* for examining magnetic materials from 4 to approximately 1000 K. The system includes a superconducting magnet that provides fields up to 75 kilogauss. The technique makes use of the nucleus, via its nuclear energy levels, as a sensitive probe of the microscopic atomic environment.

## Thermal and Mechanical

Information from the thermal methods [*differential scanning calorimetry* (DSC) and *differential thermal analysis* (DTA)] coupled with *thermomechanical analysis* (TMA) and *thermogravimetric analysis* (TGA) provides quantitative and qualitative estimations of solid-state reactions. A complete *Perkin Elmer thermal analysis system* is available for determining mechanical, thermodynamic, and kinetic properties of various materials. The TMA system provides measurements of penetration, expansion, contraction, and extension of materials as a function of temperature from  $-170$  to  $325^\circ\text{C}$ , while the TGA system measures weight changes as a function of time or temperature from ambient to  $1000^\circ\text{C}$ . In the DTA and DSC systems, a sample and a reference are subject to carefully programmed temperature profiles, and the change in energy observed (DTA) or energy required for energy

balance (DSC) is used to measure properties associated with phase transitions over wide ranges.

Laboratory capabilities also include the measurement of localized thermal properties of small specimens using scanned imaging techniques, including the location of near-subsurface structures via thermal and elastic contrast mechanisms. Related capabilities include short-pulse (20 nanoseconds) acoustic propagation and attenuation measurements.

Specimen thickness in the range of 100 angstroms to 131 micrometers is obtained with a *Dektak 3030 stylus profilometer*.

## Optical

Instrumentation for a variety of optical measurements is available. Spectroscopic equipment includes *Spex visible and ultraviolet spectrometers*, a *Perkin Elmer 330 ultraviolet/visible/infrared spectrometer*, a *Mattson Sirius 100 Fourier transform infrared spectrometer* (10 to 20,000 wavenumbers), and a *Perkin Elmer 621 grating spectrometer* (250 to 4000 wavenumbers) with cryogenic attachments. Apparatus is also available to obtain Raman and fluorescence spectra based on a *Spex 1400 double monochromator* with holographic gratings, helium-neon or argon lasers, and photon counting. A multichannel Raman system is being added to allow simultaneous measurement of a complete spectrum.

Pulsed neodymium-yttrium-aluminum-garnet and continuous-wave argon ion and helium-neon laser sources are available for laser imaging studies, laser ultrasound generation, and laser interferometric detection. The instrumentation allows the investigation of the localized thermal and mechanical properties of materials. The complex dielectric function and thickness of solid films can be examined using a *Rudolph ellipsometer*.

A high-quality *Vickers M-41 trinocular microscope* with complete photographic capabilities is available for optical microscopy of samples.

Several light-scattering methods are available for a variety of problems. An *intensity correlation spectrometer* for characterization of the structure of materials by measuring the overall size and polydispersity of scattering samples is available. Photon counting methods are used to detect the light scattered using a 50-megawatt helium-neon laser. Another light-scattering apparatus is used to measure, as a function of wavelength, either total or angular light scattering.

A *laser Doppler velocimeter* provides velocity measurements of fluids in steady and pulsatile flow. Operated in the differential scattering mode, it has a maximum Doppler frequency of 1 megahertz. A *two-beam velocimeter* is available to measure the size and velocity of laser-produced bubbles in liquids.

## MATERIALS PREPARATION

Well-equipped materials preparation and processing laboratories are available for the synthesis and growth of a variety of solids.

## Crystal Growth

Single-crystal growth is carried out by flux melt and vapor phase methods in high-temperature furnaces. The *flux melt furnace* can reach temperatures in excess of 1500 K and can be programmed to raise or lower temperatures at rates of less than 0.5 K per hour. Many solids with high melting temperatures not attainable by other means can be prepared using this method. The *vapor phase growth furnace* has a temperature capability greater than 1400 K, a temperature zone with uniformity of  $\pm 1$  K over greater than 10 centimeters, and a gas flow or vacuum operation, and it can be operated as a pulling furnace. Chemical vapor deposition using a number of different carrier gases can be used to produce crystals or epitaxial layers in the system.

Slow cooling and diffusion apparatus is used to achieve growth of crystals that can be prepared by solution growth or gel techniques. A number of organic and inorganic compounds have been prepared by this method.

## Thin Films

Thin film vacuum deposition is carried out in several systems. Semiconductor oxide metal compounds and alloy films are sputtered in two 18-inch vacuum systems capable of both radio-frequency and direct-current ion sputtering at power levels of up to 1.5 kilowatts. Two separate, smaller vacuum systems are used for vacuum deposition of metals. One uses thermal evaporation and the other, direct-current sputtering. The latter is used to fabricate multielement sputtering targets for reactively sputtering metal oxide alloys. An additional system has been specially constructed for pyrolytic decomposition or organic compounds to deposit metal oxide films.

A very-high-vacuum *electron-beam evaporation system* is available for special thin film preparation. A *dual-source thermal evaporation system* is in operation that allows processing of compounds. Many of these systems are equipped with substrate temperature controls that permit deposition at either cryogenic or elevated temperatures.

Laser chemical vapor deposition and other photochemical processing techniques can be done on a variety of substrate materials. Sample preparations requiring high-vacuum techniques such as freeze-pumping, sublimation, and deposition from gas-phase reactions can be carried out. High-resolution optical luminescence, absorption, and excitation spectroscopy can be done while sample temperatures are controlled in the

range of 10 to 300 K. Short-wavelength, high-power excimer and nitrogen lasers are available. High-resolution electron spin resonance spectra of a wide variety of samples, including metals and semiconductors condensed from gas phase reactions, can be observed at temperatures ranging from 4 to 300 K.

The extensive *organic chemistry laboratory* facilities provide the Research Center with the ability to prepare a wide range of new and commercially unavailable chemicals for a variety of materials-related programs. The laboratory is now being used to synthesize systematically new compounds and alloy systems such as charge-transfer complexes, processible polymers, and metal oxides.

## SPECIAL-PURPOSE LABORATORIES

A variety of special-purpose laboratories not explicitly described here are available (Table 1).

**Table 1**—Research Center Laboratories (partial listing).

Analytical  
 Artificial Intelligence Research  
 Atmospheric Reactions and Flame Structures  
 Auger Electron Spectroscopy  
 Biodynamics  
 Corneal Light Scattering and Infrared Absorption  
 Correlation Spectroscopy  
 Glass Blowing  
 Laser Chemistry  
 Laser Measurements  
 Laser Spectroscopy  
 Magnetodynamics  
 Mass Spectrometry  
 Materials Preparation  
 Matrix Isolation and Magnetic Resonance  
 Microphysics  
 Mössbauer Spectroscopy  
 Neutral Beams  
 Nuclear Magnetic Resonance  
 Optical Materials Characterization  
 Organic Chemistry  
 Scanning Electron Microscopy  
 Secondary Ion Mass Spectrometry  
 Solid State Research  
 Thermal Imaging Spectroscopy  
 Vacuum Deposition  
 X-Ray Scattering

## THE AUTHOR

THEODORE O. POEHLER's biography and photograph can be found on p. 141.