OAK RIDGE NATIONAL LABORATORY

ORNL Facilities and Equipment for Use in High-Temperature Superconductivity Research and Development

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Facilities

Cable Test Development Laboratory (FED)

Description: Joint ORNL-Southwire facility is used for testing HTS cables and cable components, including power supplies, terminations, joints, and cryogenic systems. Cables up to 5–10 m long can be tested (straight and with bends up to 90 degrees). The laboratory has the following capabilities: 200-kV impulse testing; short-circuit testing to 25,000A; 100-kV ac withstand power supply; dc current to 3000 A; three-phase; ac current to 3000 A; and liquid nitrogen cable cooling system with subcooling to 70 K (1.5 kW) and pressurization to 10 atm.

HTS Application: Used for testing HTS cables and cable components, including power supplies, terminations, joints, and cryogenic systems.

Dielectrics Research Facility (FED)

Description: This high-bay laboratory contains unique equipment for research and development of electrical insulation for high-temperature superconducting devices. A special feature of this laboratory is the ability to characterize dielectric performance at cryogenic temperatures under a variety of pressure conditions ranging from high vacuum to 15 atm in liquid nitrogen. Equipment includes the following:

- High-voltage power supplies—500-kV Haefely-Trench impulse generator, with rise/fall time = 1.2/50 μs*;* 100-kV, 10-kVA American High Voltage manually controlled ac power supply; 150-kV, low noise (<3pC) Phoenix Technologies ac power supply with Allen-Bradley programmable logic control; and 100-kV Steelman dc power supplies
- High-voltage breakdown test facilities—High-pressure (up to 15 atm) cryogenic model cable test vessel; two chambers (one for short-term breakdown tests and one for long-term aging studies under cryogenic conditions); high-pressure (up to 15 atm) cryogenic sheet breakdown; chamber for gas and vacuum breakdown; and atmospheric pressure cryogenic tape tester
- Model cable wrapping facility
- Epoxy mold and fabrication capability for high-voltage testing
- Electrical shielding (two RF shielded room enclosures)
- Electrodes [range of electrode geometries, uniform field (profiled electrodes), non-uniform field (point-plane, sphere-plane, cylindrical, etc.)]
- Diagnostic equipment—Partial discharge analysis; tan delta bridge (power loss factor); ultra-high-speed streak/framing camera (to 20 million frames/s); gas-chromatography mass spectrometer (state-of-the-art bench analytical system, positive and negative ion capability offering unique selectivity, and chemical ionization source for high sensitivity); cryogenic enrichment gas chromatography with

electron capture detection (parts per billion sensitivity in complex backgrounds); gas chromatography with thermal conductivity detector; Extrel high-pressure mass spectrometer (direct sampling from high pressure up to 1 atm and detection of short-lived ionic and radical species); UV/VIS/IR spectrophotometer; and fast digital storage oscilloscopes (to 2G samples/s).

HTS Application: For research and development of electrical insulation for high-temperature superconducting devices. A special feature of this laboratory is the ability to characterize dielectric performance at cryogenic temperatures under a variety of pressure conditions ranging from high vacuum to 15 atm in liquid nitrogen.

High Flux Isotope Reactor (HFIR), a DOE National User Facility (CMSD)

Description: HFIR is a versatile, 85-MW isotope production and test reactor with the capability and facilities for performing a wide variety of irradiation experiments. HFIR is unique in the sense that it provides one of the highest steady-state neutron fluxes available in any of the world's reactors, and neutron currents from the four horizontal beam tubes are among the highest available.

The original primary purpose of the HFIR was the production of transuranium isotopes; however, many experimental irradiation facilities were provided for in the original design, and several others have been added. Experimental irradiation facilities available include (1) the hydraulic tube facility, located in the very-high-flux region of the flux trap, which allows for insertion and removal of irradiation samples while the reactor is operating; (2) thirty target positions in the flux trap, which normally contain transuranium production rods but which can be used for the irradiation of other experiments (two are instrumented target positions provided by a recent modification); (3) six peripheral target positions located at the outer edge of the flux trap; (4) numerous vertical irradiation facilities of various sizes located throughout the beryllium reflector; (5) two pneumatic tube facilities in the beryllium reflector, which allow for insertion and removal of irradiation samples while the reactor is operating for activation analysis; (6) four horizontal beam tubes, which originate in the beryllium reflector; and (7) four slant access facilities, called "engineering facilities," located adjacent to the outer edge of the beryllium reflector.

HTS Application: Neutron-scattering experiments reveal the structure and dynamics of a very wide range of materials. The neutron-scattering instruments installed on the horizontal beam tubes are used in fundamental studies of materials of interest to solid-state physicists, chemists, biologists, polymer scientists, metallurgists, and colloid scientists. The instruments reveal the vortex crystal structure and magnetic and crystalline structures in HTS materials. These instruments are open to use by university and industrial researchers on the basis of scientific merit.

High Temperature Materials Laboratory (HTML), a National User Facility (M&C)

Description: The HTML is an acclaimed complex of sophisticated equipment designed to assist American industries and universities in developing advanced materials for automotive, diesel, and other energy-efficient applications. Physically, it is a 64,500-ft^2 building, in which reside six "user centers," which are clusters of specialized equipment revolving around a specific type of properties measurements. The user centers and their capabilities are

- Material Analysis User Center—electron microscopes, microprobe, AFM, SAM;
- Mechanical Characterization and Analysis User Center—tensile, flexural testing at elevated temperatures;
- Diffraction User Center—X-ray and neutron at ambient and elevated temperatures;
- Thermophysical Properties User Center—heat flow (conductivity, diffusivity), thermal expansion;

- Residual Stress User Center—X-ray and neutron diffraction for nondestructive analysis; and
- Machining, Inspection, and Tribology Research User Center—instrumented diamond grinding machines, dimensional measurement.

The HTML may be accessed by industry or universities at no charge if the results will be published. Full-cost recovery applies for use of this facility for proprietary research.

HTS Application: Scanning electron microscopes (SEMs) are used to examine surfaces and interfaces. The AFM is used to measure surface profiles and hence smoothness. The center has several high-resolution SEMs, a high-resolution Auger system used for surface compositional analysis with high spatial resolution, a focused ion beam microscope that can be used for making patterns on coated conductors to do localized thinning of samples (including samples for TEM), high-resolution TEM , and thermophysical property measurement equipment to measure thermal conductivity, perform tensile tests, and the like.

Wet Chemistry Laboratory (CSD)

Description: The Wet Chemistry Laboratory contains fume hoods, gloveboxes, and a rotor evaporator for handling organic and inorganic solutions as well as air-sensitive materials.

HTS Application: Synthesis of solution precursors for buffers and superconductors; chemical etching of HTS substrates.

Equipment

AC Susceptometer/DC Magnetometer (M&C)

Description: Lakeshore Model 7225

HTS Application: Used to measure ac susceptibility and dc magnetization for small samples (<3 mm)

Atomic Force Microscope (CSD, M&C)

Description: Digital instruments Nanoscope II, Nanoscope III

HTS Application: Used to characterize HTS and related material surfaces (measurement of surface smoothness)

BaF_2 Conversion Systems (three) (CSD)

Description: Two furnace, gas-handling systems with O_2 delivery and water vapor delivery, one for BaF_2, the other for TFA

HTS Application: YBCO film growth on XTAL and RABiTS

Controlled-Atmosphere Processing Furnaces (CMSD, M&C)

Description: (a) Reel-to-reel system for converting Y-Cu-BaF_2 precursor to YBCO on tapes of 1 to 3 m in length. This system consists of a 22-zone furnace, a metal reaction chamber, and controlled water vapor, oxygen pressure, and carrier gas supplies. (b) UHV induction furnace with controlled atmosphere capability; sample sizes to 3.5 cm.

HTS Application: HTS and related material heat treatments, including doping and annealing

Cryocooled 6-T Magnet with 20-cm Warm Bore (FED)

Description: A Nb-Ti magnet with a 20-cm-diam warm bore cooled by a Cryomech pulse-tube refrigerator. The bore length is approximately 0.5 m. An adjacent GM cryocooler can control sample tape or coil temperatures from 20 to 100 K, and three types of power supply are available: a 3000-A dc power supply, a 3000-A ac @ 60 Hz power supply, and a variable frequency (45–500 Hz), 100-A ac power supply.

HTS Application: Used for characterizing electrical performance of HTS tapes (tens of centimeters in length) and coils in the presence of a background magnetic field in any orientation from 20 to 100 K

Electron Beam Evaporation System (CSD, M&C)

Description: Airco Temescal 14-kV, three-gun, single-pocket-thickness quartz crystal monitors, RGA mass spectrometer; controlled gas delivery (Ar, H2, and H2O)

HTS Application: Used for stationary growth of e-beam seed layers on textured metallic substrates and thicker precursor films for YBCO

Ellipsometer (CSD)

Description: Gaertner Scientific Model LI 17

HTS Application: Optical spectroscopy enables thickness determination

High-Pressure, High-Temperature Furnace (M&C)

Description: Furnace can produce up to 180 atm at 850EC

HTS Application: Used for high-pressure BSCCO experiments

Lithography Facility (CMSD)

Description: Standard mask photolithographic and wet-etching facility

HTS Application: Patterning of HTS coatings and buffer layers

Low-Temperature Scanning Probe Microscope (STM) (M&C)

Description: Low-temperature scanning probe microscope

HTS Application: Microscopic characterization of HTS electronic properties and magnetic profiles

Magnetron Sputtering Systems (DC or RF) (CMSD, M&C)

Description: One system contains two targets, with 10-cm target diameters, snf substrate temperatures to 850EC. A second system is used for reel-to-reel dc sputtering of silver for overcoating HTS tapes. A third system is a 2-rf-source, reel-to-reel unit for deposition of oxide buffer layers on metal tapes. A fourth unit is a 2-rf-source system for studies of buffer layer deposition.

HTS Application: Deposition of coatings, including buffer layers of oxides and other materials

Profilometer

Description: Alpha Step 500

HTS Application: Measurement of thickness of buffer layers and superconductors

Pulsed Laser Deposition Facilities (CMSD and CSD)

Description: The CMSD facility has a LPX 325 160-W laser with 1-J/pulse energy and 250-Hz maximum rep rate. Four oxide PLD chambers are serviced by this laser. One chamber is dedicated exclusively to HTS-coated conductor research; the others are multipurpose and are used for oxide thin film research. A UHV PLD with RHEED capability. One system has the capability to deposit full RABiTS architecture, including the HTS layer, on samples in lengths ranging from 1 cm to 1 m. All chambers have multitarget carousels and multiple gas flow capabilities.

The CSD facility has a Lambda Physik XeCl 193-nm excimer laser (Model LPX 305); up to 50-Hz and 500 mJ/pulse; Neocera PLD chamber with six targets.

HTS Application: The CMSD systems are used for understanding oxide nucleation and epitaxial on biaxially textured metals and single crystals. The CSD facility is used for deposition of oxides and HTS films on short samples (<5 cm) for HTS coated conductor research.

Reel-to-Reel Continuous Processing Systems for YBCO Tape (M&C)

Description: A total of 11 reel-to-reel systems are used to produce 1- to 3-m lengths of buffered, textured metallic substrate (RABiTS) with high critical-current-density YBCO coatings. The systems include

- RF annealer to texture nickel or nickel-alloy foil;
- Reel-to-reel bonification and cleaning of nickel tapes;
- CeO_2 electron beam evaporation system or sol-gel dip coating system;
- YSZ and CeO_2 RF Sputtering system;
- System for precursor to YBCO deposition by electron beam evaporation or by sol-gel TFA;

- Post-annealing system to convert precursor to YBCO;
- Silver passivation layer deposition system;
- (T_c, I_c) characterization system over entire length;
- Reel-to-reel X-ray diffraction system.

HTS Application: Used for researching all aspects of coated conductor development in a moving, continuous reel-to-reel manner

Reel-to-Reel Dip Coating Unit (CSD)

Description: Capable of coating several meters of metal tape with oxide buffer layers; tape passes through furnace with 10-cm uniform hot zone up to 1200 C. Speeds up to 100-m per hour.

HTS Application: Buffer layers are applied via solution routes to textured metallic substrates. HTS precursors are also applied with the system at speeds of 20 to 30 m per hour.

Scanning Electron Microscope with Electron Backscatter Patterns (EBSP) Attachment (M&C)

Description: Phillips XL-30 high-resolution, hot-field-emission gun microscope with electron backscatter diffraction camera as well as software for automated indexing. Microscope also has capability to perform high-spatial-resolution wavelength dispersive spectroscopy.

HTS Application: Microscopic grain orientations (texture determinations) in materials can be obtained over large areas with this automated system.

Source Electron Beam Evaporation Systems (CMSD and M&C)

Description: (a) MDC-manufactured multipocket 3-electron-gun (5 kW power) independently operated sources with quartz crystal monitors, high vacuum, all-metal chambers with load-locking. (b) Reel-to-reel system with single electron beam, multipocket evaporator, and RF annealer for preparation of 1- to 3-m lengths of buffered substrate. (c) Reel- to-reel, 3-electron-beam evaporator for co-evaporation of Y, Cu, and BaF_2 precursors on lengths up to 3 m. (d) Basic 3-electron-beam evaporation system (single pockets) with feedback using quartz crystal monitors, all-metal system with load locking; 3-kW power supply.

HTS Application: Systems a–c are used for growth of oxide buffer layers and HTS precursor films on textured substrates. System d is used development of the ex situ BaF_2 process for coated conductors.

SQUID Magnetometer (CMSD)

Description: Quantum Design MPMS, 7-T field; in situ sample rotation; temperatures from 3 to 400 K.

HTS Application: Magnetic properties of all superconducting materials and magnetic materials versus T, B, and orientations.

T_c, J_c Measurement Apparatus (FED)

Description: J_c of samples up to 1 m can be measured quickly in a liquid nitrogen bath. Available power supplies provide up to 3000 A dc and 3000 A ac. Short samples can be cryocooled to 15–20 K and exposed to up to 6 T background field in any orientation.

HTS Application: Electrical property characterization of high-temperature superconductors

Transport Critical Current Density Measurement Systems (CMSD, M&C)

Description: (a) 8-T horizontal field; transport currents to 100 A; variable temperature; sample may be oriented at any angle with respect to the applied magnetic field; sample lengths <2 cm. (b) 15-T vertical field; transport currents to 100 A; variable temperature; sample lengths <2 cm. (c) 8-T dc magnet for ac loss characterization in short samples; variable temperature; sample lengths up to 10 cm. (d) Cryocooled system with pulsed currents up to 100 A; short samples (<2 cm) may be placed in magnetic fields up to 1.5 T; sample temperature, field level, and field orientation may be varied.

HTS Application: Transport ac energy losses of HTS coatings and associated substrates. J_c measurements may be made as a function of temperature, magnetic field, and orientation in the field.

X-Ray Diffraction (M&C, CMSD)

Description: M&C equipment: (a) Picker4-circle goniometer, providing phi and omega scans, pole figures, and texture information. (b) Rigaku 4-circle goniometer plus with reel to-reel; extremely large chi circle, enabling reel-to-reel sample evaluation; tapes can be scanned at a rate of 5 cm/s (one X-ray peak). (c) Two diffractometers that perform theta-2-theta scans for phase formation and texture information.

CMSD equipment: A 4-circle high-resolution system for obtaining phi and omega scans and pole figures

HTS Application: Crystalline structural characterization of HTS materials and buffered metallic substrates

Z-Contrast Scanning Transmission Electron Microscope (CMSD)

Description: VG Microscopes HB603U scanning transmission electron microscope: 300 kV, 1.26% spot size, information limit 0.62%, Z-contrast imaging

HTS Application: The techniques of Z-contrast imaging and atomic-resolution electron energy loss spectroscopy (EELS), both developed at ORNL, allow the atomic and electronic structure of materials to be probed with unprecedented spatial resolution. Applications include imaging grain boundaries in HTS and substrate materials, including characterization of nanoscale modifications of the electronic structure due to defects.