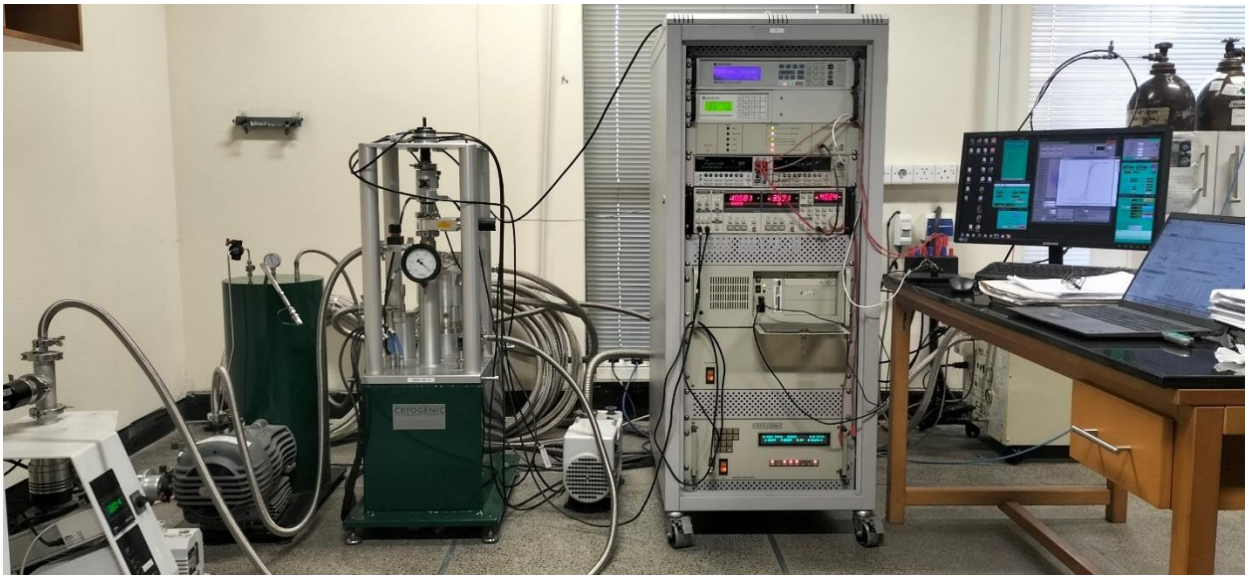
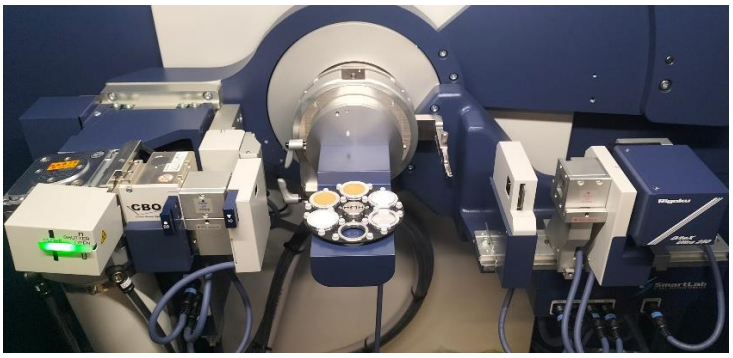
Advanced Research Instruments Training: Physical Properties Measurement Lab

Updated on: 10-06-2026 **Training Material** https://physlab.org/equipment-howtos/





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Workshop Venue: Physical Properties Measurement Lab, SBASSE, LUMS

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Training Material https://physlab.org/equipment-howtos/

All training manuals, operational guides, worksheets, and supporting study materials are available through the "Training Material" link above. Participants are encouraged to review these resources before and after the workshop for better understanding, hands-on practice, and future reference.

Physical Properties Measurement Lab, Central Lab offers hands-on technical training on advanced research instruments to facilitate high-quality research and industrial problem-solving. This training is designed to help researchers, scientists, and industrial professionals understand instrument capabilities, select suitable measurement methods, optimize measurement conditions, and confidently connect their research objectives with reliable materials characterization. The workshop will help participants strengthen their experimental planning, improve data quality, and accelerate meaningful research outcomes.

Day-1 Rigaku SmartLab SE (Link)

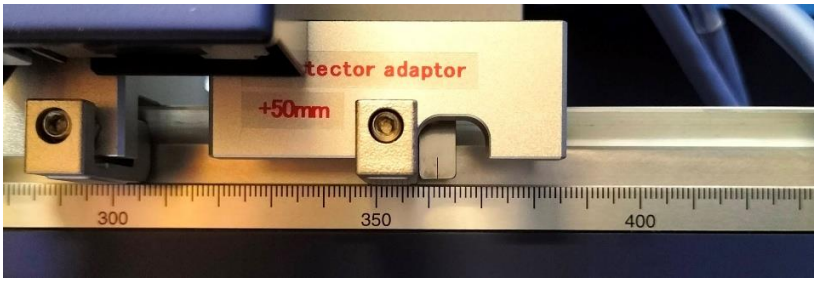
Rigaku SmartLab SE is a versatile research-grade X-ray diffractometer that allows measurement conditions to be optimized according to the crystalline quality of the sample, whether poorly crystalline, nanostructured, textured, or highly crystalline materials. By selecting suitable combinations of slit optics, Kβ filters, monochromators, and detector settings, researchers can precisely control diffraction intensity, angular resolution, and background suppression to obtain high-quality, application-specific measurements. The SmartLab SE supports advanced techniques including HRXRD, reciprocal space mapping, X-ray reflectivity, rocking curves, pole figure analysis, and grazing-incidence XRD, together with automated alignment, data acquisition, and advanced analysis capabilities through the SmartLab SE platform. In this training, participants will learn how to configure and optimize the SmartLab SE for different research applications, enabling them to acquire reliable high-quality data and effectively connect instrument capabilities with advanced materials, semiconductor, and nanotechnology research.

Step-by-Step SmartLab SE Operation and Basic XRD Concepts (Link)

Step-by-Step SmartLab SE Operation introduces participants to the safe startup, measurement, data export, and shutdown workflow of the Rigaku SmartLab SE, along with essential XRD concepts needed for routine operation. In this training, participants will learn how to turn on the instrument, activate the X-ray generator, prepare powder or thin-film samples, set basic measurement conditions, save/export data, perform peak search, and generate reports. The session also connects practical operation with core XRD principles, including Bragg's law, BB/PB geometry, CBO optics, slits, detector selection, Kβ filtering, monochromator use, and sample requirements, helping new users operate the SmartLab SE confidently and obtain reliable diffraction data.

Optimization of XRD Measurement Conditions (Link)

Optimization of XRD measurement conditions is essential for obtaining accurate and reliable diffraction data. Important parameters such as sample holders, Kβ radiation, scan speed, step size, receiving slits, slit optics, and BB/PB alignment strongly affect peak intensity, resolution, background, and overall data quality. Improper configuration may introduce unwanted peaks, broaden or shift peaks, reduce precision, or affect quantitative analysis. In this training, participants will learn how to optimize the SmartLab SE by selecting suitable sample holders, managing Kβ radiation, adjusting scan speed and step size, tuning Receiving Slit #1 and #2, and performing proper Bragg-Brentano alignment for general measurements and Parallel Beam alignment for thin films, enabling clean, high-quality XRD data for reliable research interpretation.



GiXRD (Link)

Grazing Incidence X-ray Diffraction (GiXRD) is a powerful, surface-sensitive technique for studying thin films, coatings, and nanostructured materials. By using a very low incident angle, it enhances diffraction from the surface layer while suppressing signals from the substrate, making it ideal for detecting weak thin-film features. In this training, participants will learn how to configure and optimize the Rigaku SmartLab SE for GiXRD measurements, enabling them to acquire high-quality data and confidently analyze thin-film structure, crystallinity, and related properties for advanced research applications.

χ–ϕ Scanning Technique (Link)

χ–ϕ Scanning Technique is an advanced XRD approach used to suppress strong diffraction peaks from single-crystal substrates and reveal weak signals from thin films. By carefully tilting the sample (χ) and rotating it (ϕ), the substrate can be moved away from its optimal diffraction condition, significantly reducing its intensity while preserving the thin-film response. In this training, participants will learn how to configure and optimize χ–ϕ scans on the Rigaku SmartLab SE, enabling them to minimize substrate interference and accurately analyze thin-film structure, texture, and crystallinity for reliable research outcomes.

ω Scanning at Fixed 2θ for Overcoming χ Tilt Limitations (Link)

ω scanning at fixed 2θ is a practical XRD method used when the required χ-tilt range is limited by the SmartLab SE geometry. In orientation-dependent thin-film measurements, χ and ϕ scans are commonly used to study texture and intensity variation at a selected Bragg reflection; however, important diffraction features may be missed if the χ scan is truncated. By performing a full-range ω scan at the same fixed 2θ position, participants will learn how to recover the missing tilt-dependent intensity profile and obtain a more complete understanding of thin-film orientation behavior. This training will help researchers configure the SmartLab SE safely and effectively for fixed-Bragg ω scans, especially when χ scanning cannot fully capture the required diffraction information.

SmartLab Studio II: Basic XRD Phase Identification (Link)

SmartLab Studio II is an advanced XRD analysis platform used to convert measured diffraction data into meaningful material information through peak evaluation, background correction, Kβ peak checking, and qualitative phase identification. In this training, participants will learn how to import XRD data, evaluate and refine peak positions, remove false or Kβ-related peaks, apply Search/Match using the PDF-2 database, and select chemically reasonable candidate phases. This session will help industrial professionals and researchers confidently interpret XRD patterns, identify crystalline phases, recognize common analysis errors, and prepare reliable phase-identification reports for research and industrial applications.

Day-2 Rigaku SmartLab SE

ASC Measurements with PB or BB Alignment (Link)

Automatic Sample Changer (ASC) measurements on the Rigaku SmartLab SE enable automated, sequential XRD analysis of up to six samples, improving measurement throughput, repeatability, and laboratory efficiency. In this training, participants will learn how to prepare samples correctly, perform required BB or PB optic and sample alignments, configure ASC measurement flows, and run multiple samples under controlled measurement conditions. The session will also highlight key practical factors affecting data reliability, including powder particle size, surface flatness, sample holder volume, minimum powder thickness, and holder-depth-related 2θ offsets, enabling researchers and industrial professionals to obtain reproducible and high-quality XRD results from automated measurements.

X-ray Reflectivity (XRR) Alignment, Measurement and Analysis (Link)

X-ray Reflectivity (XRR) is an advanced thin-film characterization technique used to determine film thickness, density, and surface/interface roughness from low-angle reflected X-ray intensity and Kiessig fringe analysis. In this training, participants will learn how to configure the Rigaku SmartLab SE for XRR, perform optic and sample alignment, select suitable low-angle measurement ranges, and analyze reflectivity curves using model-based fitting methods. This session will help researchers and industrial professionals understand thin-film quality beyond phase identification, enabling reliable evaluation of coatings, multilayers, semiconductor films, and nanoscale surface layers.

Day-3 Rigaku SmartLab SE

Rocking Curve (RC) Measurement and Analysis (Link)

Rocking Curve (RC) is a high-resolution XRD technique used to evaluate the crystalline quality of thin films, epitaxial layers, and highly oriented materials by scanning ω around a selected Bragg reflection while keeping 2θ fixed. In this training, participants will learn how to configure PB optics with the χ–ϕ attachment and optional Ge(220) ×2 monochromator, perform optic/sample alignment and pre-measurement optimization, and acquire rocking curve data for assessing peak width, mosaic spread, strain, and defect-related broadening. This session will help researchers and industrial professionals understand how the FWHM and shape of the rocking curve reflect crystal perfection, enabling more reliable evaluation of epitaxial films, semiconductor layers, and advanced thin-film materials.

Pole Figure XRD for Texture and Orientation Analysis

Pole Figure XRD is an advanced texture-analysis technique used to map the crystallographic orientation distribution of grains in thin films, coatings, single crystals, and bulk polycrystalline materials. In this training, participants will learn how to configure the Rigaku SmartLab SE for pole figure measurements using χ–ϕ rotation, fixed **2θ** reflection geometry, Schulz slit setup, optics/sample alignment, background correction, and suitable α/χ and β/ϕ scan ranges. This session will help researchers and industrial professionals distinguish random orientation, fiber texture, and single-crystal symmetry, enabling reliable evaluation of epitaxial films, textured coatings, anisotropic materials, and crystallographic orientation relationships.

Day-4 Cryogen-Free Vibrating Sample Magnetometer

Cryogen-Free Vibrating Sample Magnetometer (VSM) at SBASSE, LUMS is the High Field Measurement System combines the latest cryogen-free technology with sophisticated measurement techniques, providing a versatile, powerful investigative device achieving low temperatures (5K) and high magnetic fields (up to 7 Tesla). A number of probes may be used with the basic cryogen-free High Field Measurement System and to extend its capabilities. This training offers a unique chance to delve into the world of material characterization through this state-of-the-art instrumental understanding.

Magnetic Material Characterization by Cryogen-Free VSM (Link)

Cryogen-Free Vibrating Sample Magnetometer (VSM) is used to measure magnetic moment as a function of magnetic field and temperature, enabling identification of diamagnetic, paramagnetic, ferromagnetic, ferrimagnetic, antiferromagnetic, and superconducting behavior. In this training, participants will learn the basics of M–H hysteresis, ZFC/FC temperature scans, magnetic transition temperatures, coercivity, remanence, saturation magnetization, and low-temperature magnetic response using fields up to ±7 T and temperatures down to about 4–5 K. This session will help researchers connect VSM data with magnetic phase transitions, domain behavior, spin-glass effects, superconductivity, and magnetocaloric properties for advanced materials research.

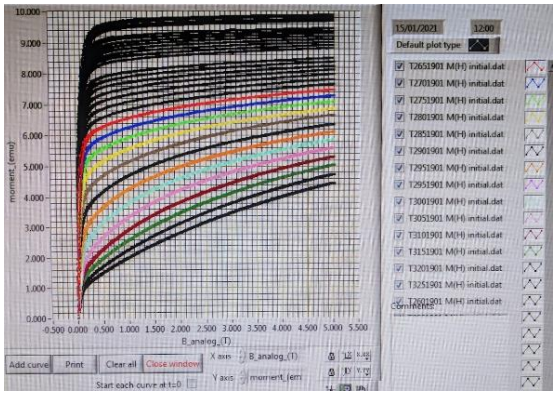
AC Susceptibility (ACS) Probe Measurements (Link)

AC Susceptibility (ACS) is used to study the dynamic magnetic response of materials under a small oscillating magnetic field, giving the in-phase χ' and out-of-phase χ'' components of susceptibility. The χ' component represents the reversible magnetic response, while χ'' indicates phase lag, energy loss, dissipation, or irreversible magnetic processes. In this training, participants will learn how to configure the ACS probe, set AC field amplitude and frequency, mount and detect samples, and perform χ vs temperature, χ vs DC field, χ vs AC field, and χ vs frequency measurements. This session will help researchers analyze superconducting transitions, spin-glass behavior, magnetic losses, irreversibility, and frequency-dependent magnetic dynamics in advanced materials.

Resistivity, Hall Effect and Magneto-Transport Measurements (Link)

Resistivity and Hall Effect measurements using the Low-T DCR probe are used to study electrical transport properties of thin films and bulk samples under controlled temperature and magnetic field. In this training, participants will learn sample mounting, electrical contact preparation, probe wiring, breakout-box connections, and measurement methods such as four-probe resistivity, Van der Pauw resistivity, Hall effect, and magnetoresistance. This session will help researchers determine sheet resistance, bulk resistivity, carrier type, carrier density, mobility, superconducting transition behavior, and field-dependent transport response for advanced electronic, magnetic, and semiconductor materials.

SOP for CircuitWorks Conductive Epoxy.docx



Day-5 Cryogen-Free Vibrating Sample Magnetometer

AC Heat Capacity Probe by AC Calorimetry (Link)

AC Heat Capacity Probe is used to measure the heat capacity and thermal phase transitions of very small samples using the AC calorimetry method. In this training, participants will learn how oscillating heat is applied to a sample, how temperature oscillations are detected by thermocouples and lock-in amplifier, and how amplitude and phase shift are used to evaluate heat capacity. The session will also cover sample mounting on the silicon-nitride membrane, vertical/horizontal sensor configurations, cabling, probe loading, frequency selection, and the importance of choosing a stable phase window of about **30–60°** for reliable data. This training will help researchers measure subtle thermal changes, phase transitions, and heat-capacity behavior of micro-scale materials from low temperature to room temperature and under magnetic field-dependent conditions.

Troubleshooting, Optimization and Issue Resolution in Cryogen-Free VSM

Troubleshooting and optimization of the Cryogen-Free VSM are essential for obtaining stable, reproducible, and noise-free magnetic measurements, especially during low-temperature and weak-moment experiments. In this training, participants will learn how to identify and address common operational issues such as helium-flow instability, temperature loss near 5 K, vacuum degradation, scroll-pump/VTI circulation problems, Ni-standard centering and calibration errors, weak-signal M–H noise near zero field, and MT-curve oscillations caused by environmental or flow-related effects. The session will also cover practical optimization steps including needle-valve adjustment, maintaining suitable VTI pressure, monitoring He-pot level, using TMP support for low-temperature stability, cleaning sample space, controlling AC airflow around the probe, selecting proper temperature ramp rates, and configuring Lock-In Amplifier auto-range settings for weak magnetic samples. This training will help researchers operate the VSM more confidently, diagnose measurement problems, improve data quality, and reduce downtime during advanced magnetic characterization.

Centering of Ni-Standard Sample.docx

Evacuating and Recharging the System -(Version_ VSM-2020-I).docx

He-circulation and the temperature control.docx

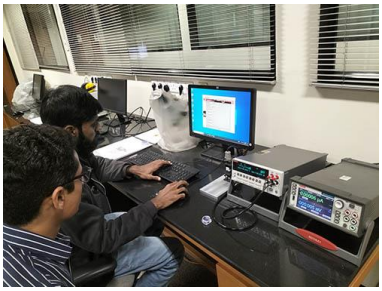
Noise Free M-H hysteresis Loop for samples with weak magnetic response.docx

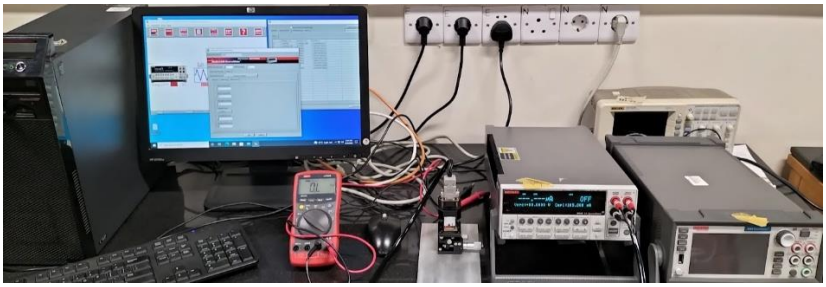
Temperature loss of all stages and the loss recovery.docx

Working on the shape of MT curves after the scroll pump repairing.docx

Four-Probe Measurement with LabTracer (Link)

Four-probe measurement with LabTracer is used to measure sheet resistance and resistivity of thin films and bulk samples with improved accuracy by separating current-carrying and voltage-sensing terminals. In this training, participants will learn how to configure LabTracer, operate the Keithley 2440 SourceMeter in 4-wire mode, perform current-sweep measurements, save data, and calculate sheet resistance or resistivity using appropriate thin-film, bulk, and correction-factor methods. This session will help researchers obtain reliable low-resistance measurements while minimizing lead and contact resistance errors for electronic, semiconductor, and functional material studies.



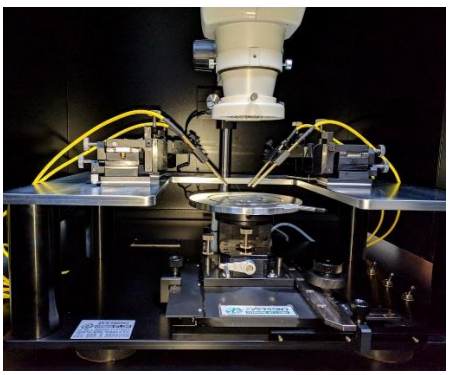
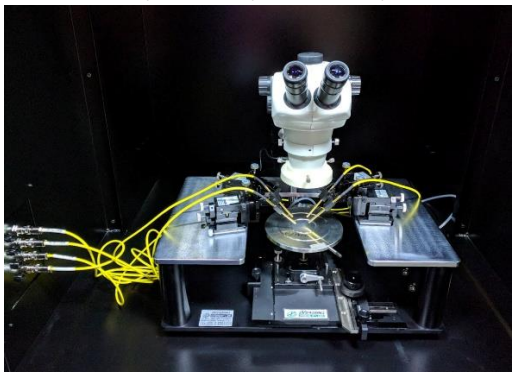
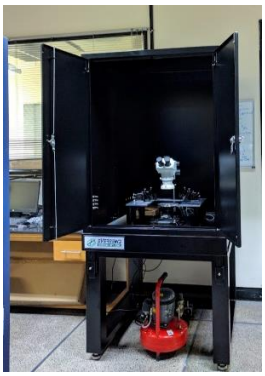


PE-50 Plasma Etcher: Plasma Cleaning and Surface Modification (Link)

PE-50 Plasma Etcher is used for dry plasma cleaning, etching, and surface modification under low-pressure plasma conditions. The type of active gases and their ratio are selected according to the material being processed, while optimum and uniform results are achieved by maintaining the plasma chamber pressure in the range of 0.15–0.30 Torr. The system has an RF power capability of 100 W, selected to support effective plasma processing while avoiding excessive sample heating. During operation, active ion species interact with the sample surface through adsorption and desorption reactions, and volatile reaction products are removed by the vacuum pump. In this training, participants will learn key process parameters such as vacuum set point, gas stabilization time, plasma duration, RF power, and gas-flow optimization, enabling them to safely process samples and improve surface properties for thin-film deposition, microfabrication, bonding, and materials research.

Everbeing C-6 Micro Probe Station (Link)

Everbeing C-6 Micro Probe Station is used for precise electrical characterization of microelectronic devices, MEMS, wafers, glass substrates, and other small devices where input/output contacts may not be visible to the naked eye. The system combines a microscope, vibration-free table, shielding box, vacuum chuck, tungsten probe tips, and four EB-050 micropositioners with 0.8 µm positioning resolution and 12 mm travel range, enabling accurate probing of device contacts. In this training, participants will learn how to position samples on the 6-inch vacuum chuck, align microprobes, use triaxial tip holders for DC measurements, and establish reliable electrical contact using fine tungsten tips, enabling accurate device-level measurements for semiconductor, MEMS, thin-film, and microfabrication research.

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