HENRY ROYCE INSTITUTE

MATERIALS FOR ENERGY-EFFICIENT ICT
The Institute is focused on promoting translation from materials discovery to application and will play a major role in driving forward key elements of the Government’s industrial strategy, which lays a particular emphasis on enhancing the commercialisation of the UK’s world-leading basic research. The funding to the University of Cambridge has enabled the purchase of additional equipment. Particular support has been given to the research area of Materials for Energy-Efficient Communications Technology. This area focuses on improving energy storage technologies, reducing power consumption and developing new materials and devices able to harness energy from the environment.

The Royce equipment is principally based in, and run from, the Maxwell Centre, enabling both academic and industrial researchers to fabricate new energy-efficient devices, and to perform advanced characterisation of materials and machines. These characterisation techniques will, in turn, help to hasten the development of energy technologies that are safer and more efficient, including longer-life phone batteries and electric cars with extended ranges.

This booklet contains an overview of all the Royce equipment facilities based in Cambridge and their capabilities. Please contact the Academic Leads to discuss your research requirements.

For more information email us at royce@maxwell.cam.ac.uk or visit www.maxwell.cam.ac.uk/programmes/henry-royce-institute
**SPUTTER DEPOSITION AND NANOSCALE PATTERNING SUITE**

This suite consists of an AJA UHV sputter deposition system, and a Zeiss Cross-Beam 540 dual focused ion beam and scanning electron microscope (FIB-SEM) system.

The sputtering system has a load-lock and is configured with eleven targets, internal calibration, and automated processes for the growth of complex metallic heterostructures for devices.

The dual beam FIB-SEM system uses a range of ion sources including a Ga ion beam to enable direct patterning of micro- and nano-pillar devices, and offers an alternative to the standard subtractive etch process used in optical lithography. This system can produce smaller devices without breaking vacuum, and without forming ex-situ interfaces.

As a combined suite, these two systems offer the capability for 3D heterostructure device fabrication for magnetic and optical materials systems.

If your research interests involve physical vapour deposition, or patterning and growth of metallic structures for devices, please get in touch.

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The Ultra High Vacuum (UHV) deposition system comprises two interconnected UHV deposition chambers. The first chamber is a molecular beam epitaxy (MBE) system for the epitaxial growth of topological insulators and related materials, and the second is a thermal and electron beam evaporator for the deposition of a range of metals.

The flexible MBE system is capable of the well-controlled growth of high-purity epitaxial layers and heterostructures covering a wide range of different compounds with unusual properties, such as topological insulators and thermoelectrics. These materials include those which have already been identified as topological insulators such as Bi₂Te₃ and Sb₂Te₃, as well as other materials of interest. After growth in this chamber, samples can be transferred under contamination-free UHV conditions to the evaporator, described below, for deposition of capping layers.

The Prevac UHV evaporator chamber has several different sources using both thermal and electron beam evaporation techniques, to deposit a range of metals onto samples transferred from the MBE system. This UHV evaporator can also be used independently to provide high-purity materials suitable for spintronics and superconducting device research.

If your research interests require growth of materials using this UHV deposition system, please contact us.

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The Bruker Dimension Icon Pro is an atomic force microscopy system capable of accurately mapping material surface topography and device electrical properties at the nanoscale, across a full 8-inch (200 mm) wafer, without requiring any wafer rotation or manual position adjustment, thus allowing semi-automated morphology and performance sampling.

This system has the capability to measure a wide range of electrical properties on both unprocessed materials and processed operating devices, such as resistance, conductance, capacitance, local potential, piezoelectric response, magnetism and photoconductivity. It also has capability in the semi-quantitative local mapping of mechanical properties, using the Bruker “PeakForce QNM” mode.

Key functions include measuring the electrical conductivity of delicate samples (using PeakForce tunnelling atomic force microscopy) and the photoconductive properties of photovoltaic materials (using photoconductive atomic force microscopy).

If your research interests require characterisation of your materials or devices using wafer scale AFM, or if you are interested in the advanced materials property mapping capability of the system, please contact us.

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The ambient processing cluster tool is a custom-built glovebox cluster tool that integrates different vacuum and liquid-based deposition technologies for a wide range of functional materials into a common inert glove box atmosphere. It comprises ten glove box modules that are interconnected by a semi-automated inert atmosphere transfer system. This facilitates combining different materials from both wet and vacuum processes into functional architectures.

The tool gives access to a wide range of functional materials, including transition metal oxides for battery and other applications, organic and hybrid semiconductors, two-dimensional materials, polymer composites etc. Its unique configuration allows integration of these different classes of materials into novel hetero-architectures, and enables fabrication of a wide range of devices, including solar cells, batteries, mechanical or thermoelectric energy harvesters, as well as integrated energy systems for energy-efficient ICT applications.

There are currently nine process modules that comprise the following:

**A. Battery Module**: PLD, evaporator, and DC/RF sputterer

This module supports deposition of sputtering of battery electrodes and solid electrolytes. With multi-source target arrays, metals and metal oxides can be deposited to form, for example, lithium ion batteries and supercapacitors. It can quickly achieve base pressure of $5 \times 10^{-7}$ mbar and enables deposition of relatively thick films of transition metal oxides for cathodes and solid-state electrolytes. The module is run under an argon atmosphere.

The PLD system has single and twin beam operation, thermal ramp of substrates up to 20 °C/min to 500 °C and 10 °C/min from 500 to 1000 °C, and has six locations for 1 inch diameter, 6mm-thick targets. The system can achieve up to 4 J/cm² laser fluence with 20 nm pulses, 28 MW power per pulse, maximum power of 700 mJ, and pulse repetition rates of up to 10 Hz. Process recipes can be programmed to allow automated running of processes.

The evaporator can accommodate a wide range of materials due to a PID-controlled source temperature up to 1200 °C, and has an integral link to the sputterer to enable sequential operations without breaking vacuum.

The sputterer is for deposition of metals and transition metal oxides for battery construction, with two DC and RF sputter target positions.

**B. Coating 1 Module**: high-resolution screen printer

This affords high resolution and high precision patterning of pin-hole free patterns to support the definition of device structures on a range of substrates, including glass and plastic film.

This includes multiple-print processes, stacking, wet-dry, dry-wet and limited micro gap printing. Many different base substrates can be printed on, including ceramics, silicon wafers, foils and paper.
C. Printing Modules: aerosol printer and slot-die coater

Aerosol printing allows a wide range of materials, including silver, to be deposited in fine and complex patterns down to 10 μm resolution using a suitable ink precursor. Device structures including small contact pads can be created and aligned to previous layers.

The slot-die coater allows pin-hole free films to be deposited, for example the deposition of encapsulating materials to seal solar cell devices from moisture and air.

D. Organic Modules: vacuum thermal evaporator and spin-coater

This module is designed to produce organic semiconducting devices such as organic LEDs, organic solar cells, OFETs and thermoelectrics. New device structures often require the combination of different preparation methods. The deposition of the required thin film can be realised by solution-based processes, or via physical vapour deposition under vacuum conditions. The system is split into two sections, one section is dedicated to solution processing, the second is for vacuum deposition of organic and metal layers.

The evaporator allows (co-)evaporation of organic molecular compounds, inorganic compounds (e.g. MoO₃, LiF) and/or metals (e.g. Au, Al, Ag). While metals and inorganic compounds can be simply evaporated using thermal sources (boat-type or effusion cells) under high vacuum conditions, organic compounds must be evaporated with special care. Precise temperature control over a wide temperature range is combined with a special source design to allow deposition of organics. The spin-coater may be programmed to modify recipes of multiple steps, and may operate over a wide parameter range (speed, acceleration, deceleration etc. may be carefully controlled). Complex devices where various solvents were used in deposition will benefit from a regenerative solvent vapour removal system integrated in the glove box purification system, to ensure a contamination-free environment.

E. Hybrid Module: vacuum thermal evaporator, spin-coater and hotplate

The hybrid module is designed to produce combinations of organic semiconductors, polymer nanocomposites and hybrid organic-inorganic semiconductors, such as metal halide perovskites. And can handle precursors such as MAI (Methylammonium iodide), PbCl₂ and PbI₂ for perovskite solar cells. The system is split into two sections. One section is dedicated to solution processing, the second is for vacuum deposition of layers in a special PVD system. The evaporator enables deposition of perovskite solar cells and organic/inorganic FETs. The spin-coater may produce films from a solvent based source, and the hotplate is used to drive off the solvent fraction from a coated film.
F. Coating Module: atomic layer deposition (ALD)

ALD affords exceptional conformal deposition of oxides in monolayer-by-monolayer precision. This allows very effective encapsulation of a device structure, and also allows fabrication of the highest quality dielectrics for capacitor and TFT structures.

G. Packaging Module: automated glue dispenser and UV curing press

This allows a range of UV-curable types of sealant beading to be precision-deposited for subsequent UV curing in the press. This module supports effective encapsulation, vital for many solar cell architectures.

H. Testing Module: Dektak profilometer

The profilometer can measure the height of features from around 50 nm to 150 μm. Surface roughness can be measured to sub-nm levels, supporting the characterisation of film morphologies. Whether measuring thicknesses to obtain critical values for device simulation or for process control, such measurements are vital for the range of deposition techniques used in battery, solar cell, TFT and OLED architectures.

I. Substrate Module: plasma ashing and vacuum oven

Plasma ashing of surfaces is an established technique for promoting adhesion of a subsequent coating, through enhancement of surface wetting characteristics by the removal of organic surface contaminants. The vacuum oven can be used with low heat to evaporate off the solvent component of a deposited layer, and may be operated without heat for temperature-sensitive systems. Such treatment can be important in the preparation of a surface for subsequent material depositions.

If your research interests require controlled deposition of any of the above materials or you would like to combine functional materials in novel ways, and have any questions on the tool’s capabilities or would like to discuss your experimental needs, please contact us.

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X-RAY PHOTOEMISSION SPECTROSCOPY SUITE

The Royce X-ray photoemission spectroscopy (XPS) facility comprises two state-of-the-art instruments with complementary capabilities that are able to perform analysis of chemical composition and electronic structure for a wide range of functional materials under inert, ultra-high vacuum conditions, as well as under realistic environmental operating conditions. The two instruments are operated as one facility by a trained and experienced technician, who can provide a full measurement and analysis service, but may also facilitate access to expert users.

INSTRUMENT 1 - ENVIRONMENTAL XPS

This near-ambient-pressure X-ray photoemission spectroscopy (XPS) system is for high-throughput chemical surface analysis under application-relevant environmental conditions. The system overcomes barriers of traditional XPS systems by enabling analyses at a wide range of atmospheres, ranging from 10⁻⁷ mbar up to 100 mbar. Via three separate mass-flow controlled gas inlets, the system allows the user to create a wide range of reactive environments, and hence to directly probe energy materials and devices under application relevant conditions. Via a sample holder with built-in laser heating, the tool allows direct probing of samples at temperatures up to 1000 °C in these atmospheres, enabling instant new insights into material growth and discovery, in particular for nanomaterials relevant to energy applications.

There is a built-in plasma cleaner, specific holders designed to minimise cross-contamination, and horizontal sample loading. Sample loading is also possible without high vacuum, enabling a wide range of materials to be probed, including liquids, biological materials, ceramics, polymers, and materials with high vapour pressures.

The system uses a SPECS XR 50 MF X-ray Source, a μ-FOCUS 600 X-ray monochromator, and differentially-pumped PHOIBOS 150 1D-DLD NAP analyser. The system also has a scannable focused extractor-type ion source for depth profiling.

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**INSTRUMENT 2-UHV PHOTOEMISSION XPS**

The second instrument is a state-of-the-art Escalab 250Xi photoemission system, which is capable of performing a wide range of surface and bulk spectroscopies under inert, ultra-high vacuum (UHV < 10^{-8} mbar) conditions. Under such conditions it is possible to obtain high sensitivity information about chemical composition and electronic structure on clean surfaces without the measurement being compromised by surface reactions or atmospheric species being adsorbed on the surface.

The instrument can be used in the following modes:

- **XPS** for chemical analysis of surfaces under inert, UHV conditions
- **Ultra-violet photoemission spectroscopy** (UPS) for measurements of valence bands and work functions with a 21.2 eV excitation source
- **Angle resolved XPS** (AR-XPS) by varying the angle of the sample to vary the analysis depth down to a few nm. This is a non-destructive technique
- **Depth profiling XPS** (DP-XPS) which combines a sequence of argon ion gun etch cycles with XPS analysis

If your research interests require access to either of the two systems, and you have any questions about the capabilities of either of the two tools, please contact us.

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ELECTRON BEAM LITHOGRAPHY

The Raith EPG 5200 electron beam tool has a thermal field emission gun for operation at 100 kV, and a high KV for high aspect ratio nanostructures, including high speed direct write with full automation. It has the fastest Gaussian beam system on the market, with a fast arbitrary shape pattern generator of 100 MHz. It can process 200 mm (8 inch) wafers and 7 inch masks with a minimum feature size below 8 nm. The precise overlay of features is less than 10 nm; this is currently the fastest tool for high density patterns.

The equipment can be used for flexible configuration to fit application requirements and is an essential manufacturing tool for fabrication of deep nanoscale devices. The system contributes to developing novel nanoelectronic devices, on-chip integrated optoelectronic circuits, quantum devices, layered material related devices, photonic and plasmonic systems. It allows both the cutting edge fabrication of small scale (lab level) devices, and for expanding these capabilities towards large scale production. It forms a bridge between basic research activities and wafer-scale manufacturing.

If your research interests require you to pattern silicon wafers with feature sizes ranging from hundreds of micrometres down to 8 nm, please contact us.

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This UV lithography tool allows the user to pattern photoresists on silicon wafers. This is a key step in any lithographic process, and may be used to fabricate, for instance, MEMS energy harvesters, microelectronic devices, and patterned electrodes for batteries or solar cells.

The UV lithography tool is the latest fourth generation Karl Suss MA6 Mask Aligner, which allows patterning of features with resolution down to approximately 700 nm, and with an alignment accuracy of 250 nm.

This tool represents a substantial evolution compared to previous generations, since all operations are fully automatic, with software-controlled optics and wafer chuck, which provides an automatic wafer edge compensation upon loading a substrate, as well as a video-assisted self-alignment function based on automatic pattern recognition of alignment marks.

The tool can process substrates ranging from millimetre-sized samples up to 6-inch wafers, and allows exposure in several contact modes: proximity, soft, hard, and vacuum. Furthermore, the long-focal optics are suitable for processing either thin (high-res) or thick (e.g. SU8) photoresists.

If your research interests require you to pattern silicon wafers with feature sizes ranging from hundreds of micrometres down to 700 nm, please contact us.

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IN SITU ELECTRON MICROSCOPY

The in situ electron microscopy package comprises an FEI Tecnai F20 high-resolution TEM/STEM microscope with two dedicated TEM holders, a fast camera designed for in situ experiments, and a holder vacuum storage unit.

1. Protochips Poseidon continuous flow liquid cell in situ holder with additional electrochemical cell. The sample can be examined as part of a continuous flow experiment or by using a static cell with electrical contacts one can image structural changes during electrochemical reactions.

2. Gatan double tilt vacuum transfer holder Model 648 that enables air-sensitive samples to be transferred to the electron microscope without the sample being exposed to the atmosphere. In order to study air-sensitive samples (e.g. Li-ion battery material) in the electron microscope, in terms of their structures and morphologies, we need a simple method to transfer samples from the batteries, typically disassembled in an Ar glove box, to the TEM sample holder.

3. Gatan OneView Camera Model that enables TEM images to be acquired at high frame rates (up to 300 fps) making it ideal for in situ studies of reactions, phase transformations, domain wall movement, etc. High quality video can be recorded over a range of resolution and speed combinations, from 4096 x 4096 pixels at 25 fps, to 512 x 512 pixels at 300 fps. The camera is supplied with a dedicated PC incorporating a ‘LookBack’ streaming video function to post-event trigger video capture; it is possible to keep a rolling capture up to 20s in length to avoid missed reaction events.

4. Gatan turbo pumping station Model 655 designed for storing the TEM holders under vacuum.

If your research interests require in situ electron microscopy, you would like to use the vacuum transfer holder or liquid cell holder, and/or use the OneView Camera, have any questions regarding in situ TEM capabilities or would like to discuss your experimental needs, please contact us.

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The 3D CT microscope is used for in situ characterisation of the composition, deformation and damage development of materials for ICT at length scales on the order of 1 micron. It is useful for determining the relationship between processing and microstructure, for observing fracture mechanisms, for investigating properties at multiple length scales, and for quantifying and characterising microstructural evolution.

It can perform in situ (time dependent) studies to understand the impact of heating, cooling, oxidation, wetting, tension, tensile compression, imbibition, drainage and other simulated environmental studies.

It can perform non-destructive views into deeply buried microstructures that may be unobservable with 2D surface imaging; compositional contrast for studying low Z or “near Z” elements and other difficult-to-discern materials.

A particular emphasis will be the development of specialised loading stages that will allow for accurate monitoring of 3D deformation processes (such as the swelling of a battery) during operation.

If your research interests require specialised 3D observation of microstructure evolution and you have any questions on the tool’s capabilities or would like to discuss your experimental needs, please contact us.

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The thermoelectric property measurement system is a Linseis Thin Film Analyser, that allows simultaneous measurements of the electrical conductivity $\sigma$, the Seebeck coefficient $\alpha$, and the thermal conductivity $\kappa$ of thin films. From the measurements of these quantities the figure of merit $ZT = \sigma \alpha^2 T / \kappa$ and the efficiency of a thermoelectric converter, in converting a flow of waste heat associated with a temperature gradient into useful electrical power, can be estimated. The measurement is based on depositing the functional material as a thin film onto a micro-fabricated silicon chip equipped with the electrodes and temperature sensors that are needed to perform the thermoelectric measurements. The system allows measurements as a function of temperature $T$ from about $-170 \, ^\circ C$ to $200 \, ^\circ C$. The system is also equipped with an electromagnet for Hall measurements on metallic samples.

If your research interests require measurements of electrical conductivity, Seebeck coefficient or thermal conductivity of thin film functional materials and you have any questions on the tool’s capabilities please contact us.

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The electrochemical quartz crystal microbalance (EQCM) with Low Current Potentiostat tool comprises a Biolin Scientific Q-Sense Explorer system with QE 401 electronics unit, a Q-Sense chamber platform, a Q-Sense flow module and a Q-sense electrochemistry module. This is combined with a Bio-Logic SP-200 potentiostat/galvanostat with Electrochemical Impedance Spectroscopy (EIS) and Ultra Low Current mode.

This system provides the capability to characterise the changes in mechanical properties and mass of thin film materials during electrochemical cycling, chemical reactions or phase changes. Film thicknesses can be from <1 nm to several microns depending on the material. The EIS capability provides characterisation of diffusion processes within electrode materials or other electrochemical systems. Overall, it provides capability for mechanical characterisation of a wide range of material systems including battery materials, supercapacitor materials, thermoelectric materials and thin film materials for ICT applications.

If your research interests require measurement of the mechanical changes of thin film materials during electrochemical cycling, reactions, phase changes or you would like to discuss your experimental needs, please contact us.

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The suite contains state-of-the-art equipment for high voltage measurements, and includes a Cascade Tesla, 200 mm, high voltage, high current semiautomatic probe station, a Keysight B1505A Semiconductor Parametric Analyser/Curve Tracer, a number of stand-alone, high precision Source Measure Units (SMUs), and a high voltage capable Keysight 2 GHz Oscilloscope.

This set of equipment allows testing and characterisation of devices and materials in wafer, die or packaged forms, very accurately from -55 °C to +300 °C. Ratings of the equipment are up to 200 A and 3 kV for wafer-level measurements using the probe station, and 0.01 fA to 1500 A and 10 kV for packaged samples. The Parametric Analyser/Curve Tracer also has C-V capability from 1kHz to 5 MHz, with a combined DC voltage rating of 3 kV. The oscilloscope with the high voltage probe can capture switching transients up to 4kV.
HIGH FREQUENCY ANTENNA MEASUREMENT SYSTEM

The suite includes high frequency test equipment, in particular a MGV Star Lab Antenna Measurement System (650 MHz to 18 GHz) and a Keysight N222A PNA Network Analyser (10 MHz to 26.5 GHz). The antenna testing system, in combination with the network analyser, allows accurate measurement of antenna radiation patterns. The vector analyser can also be used independently to measure scattering parameters of devices and systems.

A Keysight 65 GSa/s Arbitrary Waveform Generator and a Keysight Infiniium 20 GHz Oscilloscope are also available in the suite and can be used to investigate programming kinetics of resistive switches/phase change memories.

If you wish to use this equipment for material or device characterisation, please contact us.

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MAGNETIC PROPERTY MEASUREMENT SYSTEM

The Quantum Design cryogen-free Magnetic Property Measurement System (MPMS) has an operational temperature range of 1.8 to 400 K, a 7 Tesla magnet, and modules for the application of multiple external fields, including: magnetic, electric, mechanical, thermal, and optical.

The system has a magnetic moment sensitivity of better than $10^{-8}$ emu and enables detailed, long-duration, measurement and testing of magnetic phenomena in materials and devices that exhibit a strong coupling between their magnetic and electronic/thermal/optical properties.

The MPMS consists of the following modules:

1. Electronic transport probe with a 5-axis sample holder for magnetoelectric measurements;

2. High pressure cell for pressure-magnetism coupled measurements of bulk crystals, thin-films and devices;

3. Magneto-optical module with a monochromatic light source that uses a Xenon bulb and filter for illuminating samples with varying wavelengths in the 360 nm to 845 nm range for measuring photo-induced magnetisation phenomena across a wide range of temperatures and applied magnetic fields;

4. Oven for measuring high temperature properties up to 1000 K, which is particularly important for studying phase transitions in multicaloric materials that operate above room temperature.

If your research interests require detailed measurements of advanced functional magnetic materials, devices, and circuits, in which magnetic, electronic, optical and thermal properties are strongly coupled, and have any questions, please contact us.

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The Nanoscale Quantum Sensing and Imaging Suite uses atomic defects in nanoscale diamond crystals to probe the local temperature and magnetic field of a sample with a spatial resolution of ~10 nm. The Cambridge nanoscale quantum sensing suite comprises two systems which operate in different temperature regimes:

1. A room-temperature imaging system that is custom-designed in collaboration with Asylum Research, and integrates a scanning probe with optical detection of the diamond defect.

2. A low-temperature system that operates from 4 K to 300 K, and has a two-axis vector magnet of 2 T/8 T.

The room-temperature imaging system is housed in a temperature-controlled and vibrationally isolated housing, and both systems provide <0.5 K temperature stability and <5 nm drifts over the course of several hours. The dynamic range of the system spans DC to MHz with sub-mT and mK resolution for the magnetic and temperature sensing modes, respectively.

The technique is non-invasive and thus enables the characterisation of surface and interface effects in highly sensitive samples with nanoscale magnetic features. The suite is also ideal for probing caloric effects in thin films and devices: mK sensitivity enables the mapping of transient heat in high bandwidth devices and circuits with nm spatial resolution. In particular, this capability enables measurements and characterisation of devices during testing operations under ambient conditions.

If your research could benefit from this nanoscale imaging tool or if you would like more information on its capabilities, please contact us.

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The wide bore magnet is a 12 T solenoid fitted with a VTI with a 100 mm usable bore supplied by Oxford Innovative Cryogenic Engineering. Temperature control is possible from 325 K to < 2 K. The system will have a field homogeneity of 0.05% over a 1 cm DSV and 0.5% over a 4 cm DSV. The system is entirely cryo-cooler operated with a He gas filled cooling loop. The VTI will operate in static, dynamic and one-shot modes.

The system is intended to facilitate materials characterisation and process development across a wide range of topics within the theme of **Materials for Energy Efficient ICT**. While the system is provided with a generic fixed sample probe and a 100 A transport probe technical support will be available to exploit the large internal bore of this magnet by designing custom measurement probes. A wide range of standard laboratory equipment is available in the host laboratory to use in conjunction with the system.

This system is ideal for studying mesoscopically ordered materials, superconductors and low loss high permeability materials.

If your research interests might benefit from the use of this equipment, or if you have any questions on the system’s capabilities or would like to discuss your experimental needs, please contact us.

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