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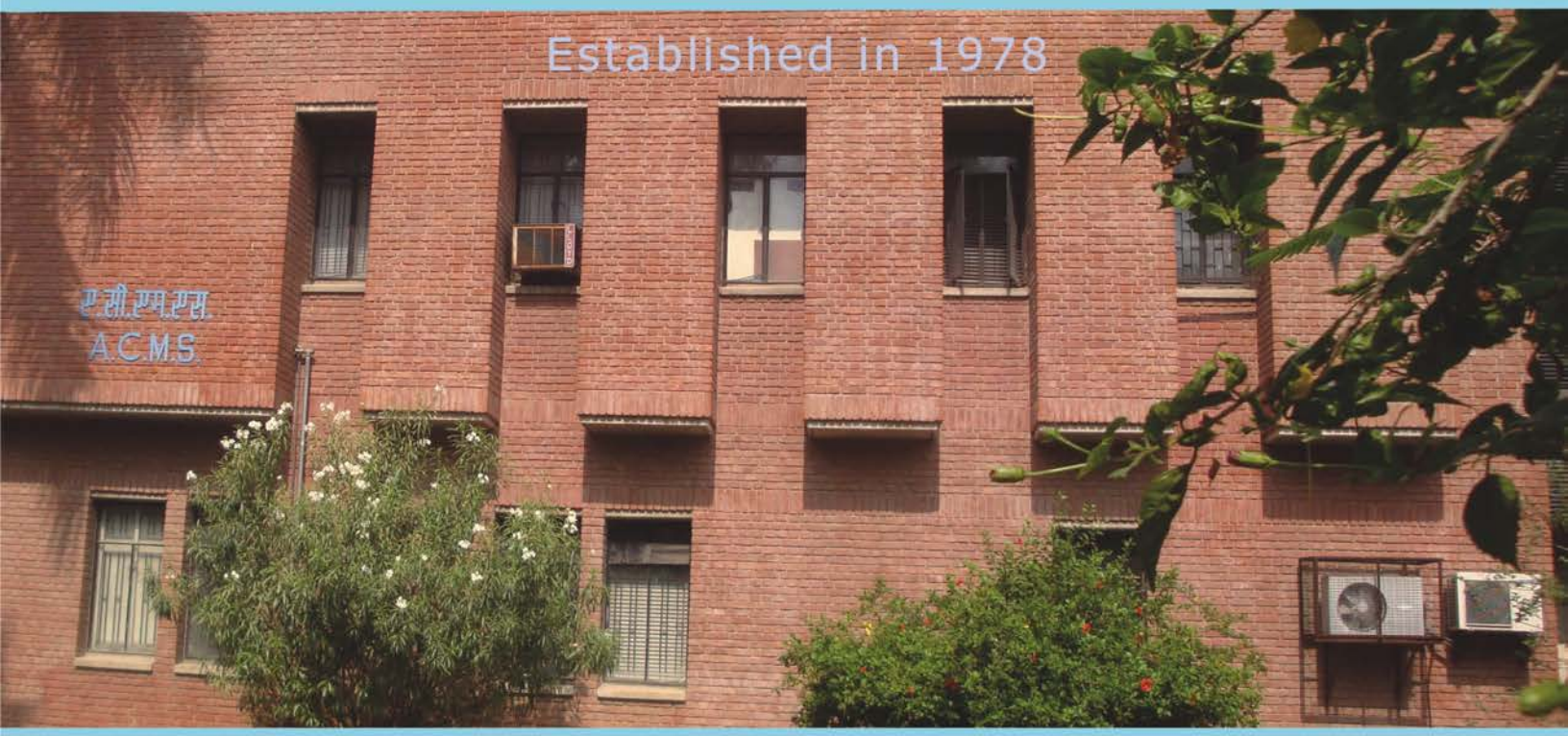
R&D Newsletter

Special Issue on
Advanced Center for Materials Science (ACMS)



Focus of ACMS

- Support R&D activities by ensuring easy accessibility and maximum utilization of sophisticated analytical equipments with minimal downtime
- Support mega projects of IIT-K faculty and researchers/industry
- Establish facilities that cover all possible aspects of materials characterization
- Contribute towards development of well-trained manpower for industry, academia and research through a series of short-term workshops/courses/seminars

soft copy of the newsletter is available at
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INDIAN INSTITUTE OF TECHNOLOGY KANPUR

ACMS Facility Rejuvenation

A major initiative has been undertaken recently to rejuvenate and upgrade the facilities at ACMS. The main focus has been to establish state-of-the art characterization facilities that cater to multiple disciplines across the institute. A number of equipments have been procured under this initiative. It is expected that most of the equipments shall be installed by March 2014.

Equipments Procured

BET Surface Area Analyzer

Model/Supplier: Autosorb I; Quantachrome Corp.

Physisorption techniques are used to determine the surface area and pore structure. The system that is due for installation at ACMS characterizes samples with nitrogen gas as the standard adsorptive gas and has the versatility for analyses of materials with low surface areas using krypton and other adsorptive gases, micropore analyses (pore radii between 5 and 15 Angstroms). The system is capable to perform automatic single-point and multi-point BET surface area, Langmuir surface area, full adsorption and desorption isotherms, and pore size and pore volume distributions. It is also equipped with chemisorption analysis, and also, thermal programming oxidation, reduction, and desorption.

Background Information

Surface area and pore size distribution are important attributes of porous materials. Typical values of the BET area for porous materials vary between 50 to 2000 m^2/g. Average pore-width of the activated carbon fibers is usually in the range between 2 to 50 nm, whereas that of nanofibers is less than 2 nm. The BET area analyzer is equipped to provide the data on the pore volume. The average pore diameter is determined from the surface area and total pore volume. The pore size distribution (PSD) is determined based on BJH (Barrett, Joyner, and Halenda) method, which involves the area of the pore walls, and uses the Kelvin equation to correlate the relative pressure of N_2 in equilibrium with the porous solid, to the size of the pores where capillary condensation takes place.

Application

This equipment caters to the needs of the various research groups engaged in the synthesis and characterization of the porous materials, including activated carbon fibers and nano-carbon fibers, metal supported catalysts. The shape of the typical isotherm obtained for carbon fibers will provide various useful information, for example, on the large uptake of nitrogen at low P/Po indicating filling of the micropores. The linear portion of the curve represents multilayer adsorption of nitrogen on the surface, and the concave upward portion of the curve represents filling of meso- and macropores. An entire isotherm is needed for one to calculate the pore size distribution of the material.

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High Temperature Simultaneous Thermo-Gravimetric Analyzer (TGA) and Differential Scanning Calorimetry (DSC)

Model/Supplier: STA 8000 & 8500 Perkin Elmer Ltd.

Thermal analysis is regularly done on a range of crystalline and non-crystalline materials to understand the kinetics and thermodynamics of various phase transformations. Advantage of thermal analysis is that it can be carried out even on non-crystalline materials, such as polymers as well as inorganic materials. Differential scanning calorimetry (DSC) measures change of heat in sample with reference to a standard reference and hence we get a plot of differential heat as a function of temperature. Any transformation is recorded as a peak in the plot. Various attributes, such as the amount of heat, start temperature for transformation, activation energy, etc. can be determined from such plots.

Features

- Calorimetric sensitivity of 0.2μW allows capturing faintest of transformation where stored energy is as small as 1J/gm
- High accuracy of heat flow can be obtained (0.2%), High precision of heat flow measurement (0.03%)
- Dynamic range of calorimeter (upto 1300mW)
- Very large range of temperature can be explored (-180^{0}C to 750^{0}C), high accuracy of temperature measurement (0.05^{0}C), and high precision of temperature measurement (0.008^{0}C)
- Fine resolution of the weight balance (0.2 μ-g)
- Large temperature range from 15^{0}C to 1600^{0}C
- High heating rate (upto 1000^{0}C/min) and cooling rates

Application

- Thermal stability of severely deformed materials
- Thermal degradation of high temperature materials
- Glass transition and melting point of various bio-polymers

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Microscopy Facility

Scanning Electron Microscope (SEM)

Model/Supplier: JSM-6010LA; JEOL

This is a compact SEM for the quick image analysis with high resolution and compositional information. The integral EDS detector can be used for micro-compositional analysis.

Features

- BSE Resolution: 5nm at 20kV
- Magnification: 5x to 300000x
- Accelerating Voltage: 0.5 to 20kV
- Automatic SEM condition set-up based on sample type

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Advanced High Sensitive Spectral Confocal and Multiphoton Microscope System for Live Cell Imaging

Model/Supplier: LSM780 NLO, Carl Zeiss GmbH

This facility includes: (i) a high-end inverted fluorescence microscope with onstage incubator for cell culture; (ii) programmable/motorized control features (X, Y and Z scans) for capturing bright field and fluorescence imaging (for DAPI, rhodamine, TRITC, FITC, GFP, YFP, and CFP) using a high resolution peltier cooled monochrome camera. The system is supported with tunable femtosecond laser source (tuning range: 690-1040 nm) with PMT and GaAsP modules for signal detection. The system is capable of simultaneous acquisition and separation of 10 fluorophores and includes control software modules for controlling all motorized components of microscope, laser scan head, laser module, stage incubation system and the digital camera. The facility in addition has incubator and bio-safety cabinet for handling of mammalian cell and tissue cultures.

Application

- Imaging dynamic morphometric changes in cells and organ cultures using fluorescence markers to understand properties such as cell growth, differentiation and migration.
- Photo activation or photo-conversion experiments to mark a single cell, organelles in a cell or molecules in a cell to track dynamic changes over time in response to changes in the environment.
- Intracellular trafficking such as cargo delivery and their fate using fluorescence probes.
- FRET analysis to quantify the proximity and interaction of molecules in a living cell.
- Imaging of dynamic changes in the cellular physiology such as changes in the levels calcium, pH, etc.
- Checking efficiency of contrast reagents in cell and organ cultures.

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Field Emission (FE)-SEM with in situ Tensile Testing and EBSD

Model/Supplier: JSM-7100F; JEOL

The high resolution secondary as well as backscattered electron images shall now be possible with the new addition of the FESEM. The FESEM is equipped with SE, BSE and EDS detectors in addition to EBSD, tensile stage module and a heating stage. With EBSD, the equipment has an unmatched capability of micro-texture analysis. The tensile stage makes it possible to carry out in-situ tensile testing and the heating stage can be used to take the images at up to 500°C.

Features

- SE Resolution: 3nm at 15kV/5nA
- Magnification: 10x to 1000000x
- Accelerating Voltage: 0.5 to 30kV
- EDS point, line and area scan possible
- Elemental mapping by EDS
- Micro-texture analysis with smallest step size possible
- Tensile stage with SPECS

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Optical Microscope with Differential Interference Contrast (DIC) Imaging Capability

Model/Supplier: Carl Zeiss GmbH

For microstructure visualization and quantitative stereology.

Mechanical Testing Facility Augmentation

100kN Universal Testing Machine (UTM)

Model/Supplier: BiSS Ltd.

It is a ***tension-compression-torsion*** equipment that will be dedicated to study monotonic deformation of variety of materials from polymers to superalloys. The equipment has additional load cells, extensometers, strain gages, and different fixtures to handle different classes of materials in different geometries (flat and round).

UTM for Bending, Fracture and Fatigue Test

Model/Supplier: BiSS Ltd.

This UTM will offer a dedicated ***fatigue-fracture test facility*** that can be used for carrying out low and high cycle fatigue experiments and fracture studies. Specimens of different sizes in flat and circular cross section can be studied over wide stress and strain amplitudes. Both the UTMs have a facility for Digital Image Co-relation that can allow full field measurement of strain in the specimen. It is to be mentioned that tests can be carried out over a wide range of temperature (RT to 1600°C) and strain rate in different loading conditions in these equipments on hard as well as soft materials.

High Temperature Creep Tester

Model/Supplier: BiSS Ltd.

The ***creep station*** can test two samples at the same time at different stress and temperature levels with the capability to carry out constant load and constant stress tests in tension and compression. It comes with high temperature furnace and high temperature extensometers for accurate strain measurement.

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Instrumented Charpy Tester

Model/Supplier: Zwick GmbH

For notched and unnotched sample impact testing at temperatures ranging from -196°C to 350°C.

Instrumented Micro-Indentation

Model/Supplier: CSM International

For Vickers and Rockwell micro-hardness testing from ambient temperature to 450°C.

Universal Hardness Testing Machine

Model/Supplier: FH-10; Tinius-Olsen Ltd.

For bulk hardness measurement using Rockwell, Brinnel, Vickers indentors.

Nano-Indentor with *in situ* Scanning Probe Microscopy (SPM)

Model/Supplier: TI 750; Hysitron Ltd.

(i) Nano-Mechanical Testing System with modulus mapping capability;
(ii) Nanotribology studies (e.g. scratching at different loads, wear volume, coefficient of friction)
(iii) Assay of coating lifetime
(iv) Loss and storage modulus and damping measurement
(v) Indent and wear track imaging

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X-ray Photoelectron Spectroscopy (XPS) with Auger Electron Spectroscopy (AES) Module

Model/Supplier: PHI 5000 Versa Probe II, FEI Inc.

XPS is a spectroscopic technique to quantitatively measure the elemental compositions and chemical and electronic states of the elements of a material, including their bond energy. To obtain the XPS spectra, the sample is irradiated with a beam of X-rays. The kinetic energy and number of electrons escaping from the top 1 to 10 nm of the material are measured and analyzed. The VersaProbe can be used much like an SEM when characterizing non-homogeneous materials. It provides high sensitivity for micro-area spectroscopy with x-ray spot sizes ranging from <10 μm to 200 μm. This is a direct result of using a focused x-ray beam and a spectrometer that is optimized for high sensitivity. This design allows complete XPS experiments to be performed at all spot sizes including: survey spectra, multi-region high resolution spectra, sputter depth profiling, angle dependent depth profiling, line scans, and maps.

Application

XPS is routinely used to analyze a wide variety of materials (both conducting and non-conducting) including inorganic compounds in powder form, metal alloys, semiconductors, polymers, glasses, ceramics, paints, viscous oils, glues, papers, inks, woods, bones, medical implants and several other biomaterials. The surface-characterization of range of materials include metals, polymers, and coatings can be performed through this technique. The unique features of XPS is that it can detect all elements with an atomic number (Z) of 3 (lithium) and above, within 0.01 monolayers of the element on a surface. The ultimate spatial resolution for spectroscopy and mapping is the same, less than 10 μm. The high micro-area sensitivity of the VersaProbe makes depth profiling with small diameter x-ray beams very practical. Thus, the instrument is likely to be used by several research and development groups around Kanpur in the state. XPS that will be installed at ACMS is equipped with an ESCA microprobe and a 20 kV C60 cluster ion gun, equipped with a comprehensive database. The C60 sputter ion gun provides the ability to sputter clean polymer surfaces without causing significant chemical damage. In addition, the optional electron gun provides a raster scanned electron beam for AES spectroscopy, depth profiling, and mapping.

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Stable Isotope Ratio Mass Spectrometer (IRMS)

Model/Supplier: Thermo Scientific Delta V advantage IRMS;
Flash EA 2000; Conflo IV Universal Interface; Gas Bench II system

The mass spectrometry facility is equipped with a Delta V advantage Stable Isotope ratio mass spectrometer (IRMS) coupled with an Elemental Analyzer Flash EA 2000 and Gas Bench II sample preparation device to perform continuous-flow high-precision stable hydrogen (δ^2H), oxygen ($\delta^{18}O$), carbon ($\delta^{13}C$), and nitrogen ($\delta^{15}N$) isotope ratio analyses on a variety of inorganic and organic samples including carbonate sediments, soil, water, plants, organics, aerosols, atmospheric gases, archaeological materials, animal tissue, bone collagen, etc.

Application

The Elemental Analyzer, and Gas Bench devices are used for on-line extraction of gas-phases from samples and introduction of the purified gases directly into the ion source of mass spectrometer for stable isotope ratio measurements. The Elemental Analyzer with an automated solid sampler is used to combust solid inorganic/organic samples in continuous-flow for $\delta^{13}C$ and $\delta^{15}N$ analyses. The Gas Bench II system is used for temperature dependent carbonate-acid reaction and extraction of CO_2 gas, which is subsequently measured for $\delta^{13}C$ and $\delta^{18}O$ values. This system can also be used for carbonate samples, gaseous species, dissolved inorganic carbon, and for water isotopes (δD and $\delta^{18}O$).

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Spectroscopy

Electron Probe Micro-Analyzer (EPMA)

Model/Supplier: JXA-8230; JEOL

This is a W/LaB_6 based EPMA with four channels for WDS analysis along with an EDS detector. EPMA is the most accurate technique for micro-composition analysis. Both standard and standardless analyses are possible in point, line or area scan modes. Elemental mapping with high resolution can be done with both WDS and EDS techniques. Quantitative analysis of all the elements from B to U is possible.

- LaB_6 gun provides the best combination of beam stability and resolution
- Range of Elements: B to U
- Accelerating voltage: 0.2 to 30kV
- Probe Current: 10-12 to 10-5 nA
- Probe current stability: +/-0.05%/h
- SE Image Resolution: 5nm
- WDS Wavelength detection: 0.087 to 9.3nm
- Four channels ensure fast analysis for multicomponent alloys and minerals

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XRD Facility Augmentation

Panalytical XRD

Model/Supplier: Panalytical

The Panalytical XRD will offer in-situ high temperature stage with simultaneous X-ray diffraction facility. This equipment can be used to monitor the evolution of phases in a variety of materials as a function of temperature. This facility will enable the investigation of phase transformation in a variety of materials in both bulk as well as thin film form.

Two Circle Diffractometer

Model/Supplier: Rigaku

This will be used for routine X-ray analysis in applications, such as phase determination, crystallite size and strain determination. It is envisaged as a workhorse diffractometer for advanced investigation.

Four Circle Diffractometer

Model/Supplier: Rigaku

The state of the art four circle diffractometer offers a unique tool high resolution X-ray diffraction tool for routine phase analysis, crystallite and micro-strain measurement as well as for niche experiments that include texture and residual stress measurement, grazing incidence diffraction, small angle scattering and reciprocal space maps determination.

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Sample Preparation Facility

Fusion Bead Machine for X-Ray Fluorescence Spectrometer (XRF)

Model/Supplier: Rigaku

The existing Rigaku WD-XRF system (X-ray generator 4kW , 60kV-150mA) can detect elements from Na to U in a variety of materials in solid, powder, filtered form. The concentration range goes from ppm levels to 100 wt%. All analyses are done on pressed pellets (30 mm diameter) made from fine powder (particle size ~50 micro meter, required quantity of about 5). Recently the facility has acquired a Claisse M4 Fluxer machine for sample preparation by fusion and preparing homogenized glass disks, which eliminate particle size and mineralogical and matrix effects, resulting in high analytical accuracy.

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Gold Sputter Coating Unit

Model/Supplier: JEC 3000; JEOL

For coating non-conductive sample for electron microscopy.

Vibratory Polisher

Model/Supplier: Vibromet 2; Buehler

For sample preparation for orientation imaging microscopy

High Precision Diamond Wire Saw

Model/Supplier: STX-202, MTI Corporation

For machining thin-slices for electron-microscopy.

Wire Electric-Discharge Machine (EDM)

Model/Supplier: JEC 3000; JEOL

Flat sample machining and to prepare CT samples for fracture toughness testing

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