

Centre of Excellence for Advanced Materials, Manufacturing, Processing and Characterization (CoExAMMPC)

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(Deemed to be UNIVERSITY) -Estd. u/s 3 of UGC Act 1956



CENTRE OF EXCELLENCE FOR ADVANCED MATERIALS, MANUFACTURING, PROCESSING AND CHARACTERIZATION (CoExAMMPC)

The centre caters to the multidisciplinary needs combining research and development activities in different fields ranging from fundamental scientific concepts to applied engineering. It encompasses cutting edge areas in science, technology and engineering. The centre is actively involved in promoting active R&D activities through sponsored and consultancy projects. And also to achieve excellence in science & technology research and publish quality articles in SCI journals in order to enhance the h-index of the Centre/University on par with leading National / International institutions. The centre is committed to explore/ experiment all the aspects of new materials including design, development, synthesis, preparation, characterization, properties, testing, performance, sustainability etc. The center is well equipped with an integrated state of the art facilities with sophisticated analytical instrumentation.

The Centre of Excellence for Advanced Materials, Manufacturing, Processing and Characterization (CoExAMMPC), started in VFSTR University, Guntur as a major research centre in this part of the country to work in close collaboration with leading higher learning institutions and industries within India and abroad.

Objectives:

- 1. To create the state of art facilities for interdisciplinary research in Universities/ Institutions promoting professional activities.
- 2. To involve all UG / PG/ PhD research scholars from all the departments promoting interdisciplinary and multidisciplinary research mainly to encourage research in the emerging areas of Science & Technology.
- 3. To carryout industry sponsored projects
- 4. To support in material processing and to provide analytical characterization, material testing and consultancy services to Government institutions, educational institutions and industries.

- 5. To develop industry-ready, world class engineering graduates
- 6. To disseminate research and new technologies for the benefit of Unipart, its suppliers and wider industry
- 7. To carryout advanced research projects, consultancy activities and establish strategic partnerships with the industries and R&D organizations at the national and international levels

RESEARCH AREAS

Materials / Manufacturing / Processing / Characterization:

- Emerging New Materials
- Hybrid materials
- Smart materials
- Nanomaterials
- Biomaterials
- Polymeric materials
- Polymer nano composites
- Sensors (shape memory and biosensing)
- Materials for energy harvesting and storage
- Materials for surface protection
- Low Temperature cure benzoxazines
- High Refractive index monomers
- High performance polybenzoxazine alloys/ blends and copolymers
- Halogen free flame retardant laminates
- Optically transparent materials
- Fire retardant benzoxazines for aerospace interior applications
- Low k interlayer dielectrics
- High performance epoxy resins

- Space environment (LEO) stable hybrid composites (Radiation Resistance/EMI shielding)
- Development of high performance latent selfcuring epoxies
- High temperature resins for infusion and transfer moulding processes
- super hydrophobic materials
- Acoustic waves transparent/resistant adhesives and coatings
- Breakthrough new resin technology for advanced composites
- Acrylic resins for automotive coatings
- Spectroscopic techniques for the study of nanomaterials – IR and Raman Spectroscopy,
- Nano sensors, MOx composites, Nano polymers, Nano Hybrids and Carbon Materials.
- Optical waveguide technology, IR sensors, LIF, chip based Chemical and Bio-Chemical Sensors, Sensors for condition monitoring
- OTFs floating, targeted drug delivery systems for sustained and controlled release.
- Fatigue and fracture behaviour of materials
- Solvent-free modification of automotive polymers
- LASERS, Combustion & Propulsion Diagnostics, Homogeneous Charge Compression Ignition - HCCI, IC Engines, Laser diagnostics in turbulent reactive flows, F1 Engines - Ford, Subaru, Ferrari, highest efficiency and lowest pollution combustion of fuels, Quantitative Spectroscopy, Tomography
- Quantum cascade lasers (FP and DFB), etc., Devices and Circuits
- CHEMICAL METROLOGY Analytical gas standards
- COMPUTATIONAL STUDIES
- Environmental Analytical Chemistry, Advanced Instrumentation, Hyphenated Techniques.

OTHER CONTRIBUTORS FROM VIGNAN'S UNIVERSITY

Dr. Shaik. Anwar

Ph.D (HCU) Asymmetric reactions

Dr. N. Satya Vijaya Kumar

Ph. D (S.R.M. Univ.) Small Molecular Assemblies for Water Splitting

Dr. G. Srinivasa Rao

Ph.D (JNTU-K) Hybrid Vehicles

Dr. K. V. Madhuri

Ph. D (S. V. Univ.) Thin Film Technology

Dr. D. Vijaya Ramu

Ph. D (South Korea)

Dr. N. S. Sampath Kumar

Ph. D (SRM Univ.) Proteomics, Aquatic Biotechnology

Dr. Avinash A. Parsalkar

Ph. D (University of Ulm, German) Disease Pathogenesis, Biomarker

Dr. K. Prakash Narayana Reddy

Ph. D (DFRL) Diagnostic Microbiology, Vaccine development, Chicken Antibodies

Prof. Krupanidhi.S.

Ph. D (S. V. Univ.) Genetics, Molecular Biology, Immunology

Dr. V. Madhusudhan Rao

Ph. D (BHU) Lithium Ion Batteries

Dr. K. Prabhakara Rao

Ph.D (IITM) New Materials for Energy, DSSC, Porous coordination polymers

Dr. K. Ravi Kumar

M. Sc., Ph. D (IIT-H) Organometallic Chemistry

Dr. Anandarup Goswami

Ph. D (Cornell) Electro-catalysts, Core / Shell nanoparticles, Mesoporous materials, nanocomposites

Dr. Chandra Sekhar Kuppan

Ph. D (CLRI) Polymers, Surfactants **Dr. D. Vinay Kumar** Ph. D (NIT-W) IC Engines, Alternative Fuels

Dr. Sanjit Kumar Ph. D (IIT-Kgp) Hydrology, Climate Change assessment

Dr. A. Sirisha Ph. D (IIT-Kgp) Irrigation, Soil and Water Management

Prof. G. Durga Sukumar Ph.D (IITKgp) Power Electronics

Prof. M. Rama Krishna Ph.D (IIT-R) Nanocomposites, Epoxy Resins

Dr. B. Nageswara Rao Ph.D (JNTU-H) IC Engines, Bio Fuels, Heat Transfer, Refrigeration

COLLABORATORS - Academic

Prof. Wu Ren-Jang Providence University, TAIWAN.

Dr. Prathik Roy University of Canterbury, NEW ZEALAND.

Prof. K. Mukkanti J.N.T. University Hyderabad, INDIA.

Prof. Chi-Te Liang National Taiwan University, TAIWAN.

Prof. Shih-Lin Hung National Chiao Tung University, TAIWAN.

Dr. T. K. Lin Sinotech Engineering Consultants, Inc., TAIWAN.

Dr. Ferdi Karadas Bilkent University, Ankara, Turkey

Dr. Katzu Fukuzato Daihatsu Engg., JAPAN. Dr. R. N. Chauhan National Chiao Tung University, TAIWAN.

Soongsil University; KoreaTech University Hannam University; Daegu University SOUTH KOREA.

Ecolé Centrale des Nantes FRANCE.

FACILITIES AVAILABLE

- Atomic Force Microscope
- Electro Chemical Workstation
- Fourier Transform Infra-Red Spectrometer
- Raman Spectrometer
- X-ray Diffraction
- Particle Size Analyzer
- Thermogravimetry and Differential Thermal Analysis (TG/DTA)
- Differential Scanning Calorimeter
- Scanning Electron Microscope
- Energy Dispersive Spectroscopy (EDS)
- Optical Microscope
- Tubular Furnace
- Muffle Furnace
- Water Purification System
- Elemental (C,H,N,S) analyzer
- UV/Vis Spectrophotometer
- Fluorescence Spectrophotometer
- UV Illuminator
- HPLC/ GC
- DC Magnetron Sputtering
- Thermal Deposition
- E-Beam Evaporation
- Pellet Maker
- Thickness monitor
- Chiller
- Probe and Bath Sonicator
- Digital Micro-hardnessTester
- Sieve Shaker Gyratory (motorized)
- Metallurgical Microscope DQS I4 with antifungal and plan achromatic lens with image analysis software.

Industrial

Dr. Mohammad Reza Saboktakin MD

Nano Biomaterials Nano BMat Company GmbH (HRB-132698), Beim Strohhause 27, c/o Firmenpool, 20097 Hamburg, GERMANY

Dr. Mohammad Reza Saboktakin MD

Nano Biomaterials Arian Chemie Gostar Research Company, Tabriz, IRAN.

Dr. TijanaBojiæ, MD

TijanaBojiæ KOMINIKACIJE Respiratory Machine Artificial SARS Nanotechnology "Tijana Bojiæ KOMINIKACIJE" and Assistant Res. Prof., Institute of nuclear scineces Vinèa, Univ. of Belgrade SERBIA

Mr. V. Pradeep

Nano Sensors Vasti Industries, INDIA.

Dr. A. B. Sudhakara Sastry

Nanomaterials, Aarshadhaatu-Green Nano Technologies India Private Limited, INDIA.

Mr. R. Chandra Mohan

Polymer Composites, Resins and Allied Products, INDIA.

Dr. A. B. Sudhakara Sastry

Nanomaterials Sri Maharshi Research Institute of VedicTechnology INDIA.

Publications

Publications (Journal papers)	=	42	2
Conference Proceedings	=	48	
Poster Presentations	=	24	

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RESEARCH PROJECTS

Prof. S. Krupanidhi

- Dr. Dirisala VijayaRamu
- Dr. Shivakiran Sathyanarayan Makam
- Dr. K. Prabhakara Rao
- Dr. Shaik. Anwar
- Dr. Nannapaneni Satyasree
- Dr. K. Venkata Madhuri
- Mr. P. Ashokkumar
- Mr. P. Bangaraiah
- Dr. P. Srinivas
- Mr. A. Raghunath & Prof. K.V.K. Kishore
- Prof. N. Usha Rani
- Prof. G. DurgaSukumar
- Prof. K. Venkata Rao
- Dr. D. Jagadish
- Dr. Koneru Kalpana, Mrs. Ch.Hymavathi,
- Mrs. K. Santhisri & Mrs. P.R.S.M. Lakshmi

Together contributed over the past three years, sponsored projects to the tune of Rs. 672.31 Lakhs

SCANNING ELECTRON MICROSCOPE (SEM)

Introduction: SEM Invented some 50 years ago, SEM is now a mature technique and is applied widely in many scientific applications. SEM is a powerful technique in the examination of materials, metallurgy, geology, biology and medicine etc.

Principle: A normal scanning electron microscope operates at a high vacuum. The basic principle is that a beam of electrons is generated by a suitable source, typically a tungsten filament or a field emission gun. The electron beam is accelerated through a high voltage (e.g.: 10-30 kV) and pass through a system of apertures and electromagnetic lenses to produce a thin beam of electrons., then the beam scans the surface of the specimen by means of scan coils (like the spot in a cathode-ray tube "old-style" television)

Electrons are emitted from the specimen by the action of the scanning beam and collected by a suitably-positioned detector.

The microscope operator is watching the image on a screen. Imagine a spot on the screen scanning across the screen from left to right. At the end of the screen, it drops down a line and scans across again, the process being repeated down to the bottom of the screen.

Applications/User purpose: SEM is having applications for user's purpose in various scientific/engineering fields such as....

1. Materials Science: Nanomaterials, polymers, composites, building materials, steel & metal alloys, wood, textile & paper, cement industry, ceramic & glasses etc.



2. Life Sciences: Cell & tissue morphology, plant & animal biology, sub-cellular analysis, pharmaceutical, biomedical engineering, drugs, microbiology, spore observation etc.

3. Restoration: SEM is useful in restoration of historical monuments.

4. Earth Sciences: Palaeontology, petrology, mineralogy, oil & gas, ore processing etc.

ENERGY DISPERSIVE SPECTROSCOPY



Introduction: EDS makes use of the X-ray spectrum emitted by a solid sample bombarded with a focused beam of electrons to obtain a localized chemical analysis. All elements from atomic number 4 (Be) to 92 (U) can be detected in principle, though not all instruments are equipped for 'light' elements (Z < 10). Qualitative analysis involves the identification of the lines in the spectrum. Quantitative analysis (determination of the concentrations of the elements present) entails measuring line intensities for each element in the sample and for the same elements in calibration Standards of known composition. By scanning the beam in a television-like raster and displaying the intensity of a selected X-ray line, element distribution images or 'maps' can be produced. Also, images produced by electrons collected from the sample reveal surface topography or mean atomic number differences according to the mode selected. The scanning electron microscope (SEM), which is closely related to the electron probe, is designed primarily for producing electron images, but can also be used for element mapping, and even point analysis, if an X-ray spectrometer is added. There is thus a considerable overlap in the functions of these instruments.

Principle: EDS is an analytical technique used for the elemental analysis or chemical characterization. It relies on an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing a unique set of peaks on its electromagnetic emission spectrum.

To stimulate the emission of characteristic X-rays from a specimen, a high-energy beam of charged particles such as electrons or protons, or a beam of X-rays, is focused into the sample being studied. At rest, an atom within the sample contains ground state (or unexcited) electrons in discrete energy levels or electron shells bound to the nucleus. The incident beam may excite an electron in an inner shell, ejecting it from the shell while creating an electron hole where the electron was. An electron from an outer, higherenergy shell then fills the hole, and the difference in energy between the higher-energy shell and the lower energy shell may be released in the form of an X-ray. The number and energy of the X-rays emitted from a specimen can be measured by an energy-dispersive spectrometer. As the energies of the X-rays are characteristic of the difference in energy between the two shells and of the atomic structure of the emitting element, EDS allows the elemental composition of the specimen to be measured.

Application/User's purpose:

1. **Materials Science:** Nanomaterials, polymers, composites, building materials, steel & metal alloys, wood, textile & paper, cement industry, ceramic & glasses etc.

2. Life Sciences: Plant & animal biology, sub-cellular analysis, pharmaceutical, biomedical engineering, drugs, microbiology etc.

3. Restoration: EDS useful in restoration of historical monuments.

4. Earth Sciences: Palaeontology, petrology, mineralogy, oil & gas, ore processing etc.

THERMOGRAVIMETRY AND DIFFERENTIAL THERMAL ANALYZER (TG/DTA)

Introduction/Principle: Thermogravimetric analysis (TGA) is conducted on an instrument referred to as a thermogravimetric analyzer. A thermogravimetric analyzer continuously measures mass while the temperature of a sample is changed over time.

A typical thermogravimetric analyzer consists of a precision balance with a sample pan located inside a furnace with a programmable control temperature. The temperature is generally increased at constant rate (or for some applications the temperature is controlled for a constant mass loss) to incur a thermal reaction. The thermal reaction may occur under a variety of atmospheres including: ambient air, vacuum, inert gas, oxidizing/reducing gases, corrosive gases, carburizing gases, vapors of liquids or "self-generated atmosphere"; as well as a variety of pressure including: a high vacuum, high pressure, constant pressure, or a controlled pressure.

The thermogravimetric data collected from a thermal reaction is compiled into a plot of mass or percentage of initial mass on the y axis versus either temperature or time on the x-axis. This plot, which is often smoothed, is referred to as a TGA curve. The first derivative of the TGA curve (the DTG curve) may plotted to determine inflection points useful for in-depth interpretations as well as differential thermal analysis.

A TGA can be used for materials characterization through analysis of characteristic decomposition patterns. It is an especially useful technique for the study of polymeric materials, including thermoplastics, thermosets, elastomers, composites, plastic films, fibers, coating, paints, and fuels.

Application/User's purpose:

1. Thermal stability: TGA can be used to evaluate the thermal stability of a material. In a desired temperature range, if a species is thermally stable, there will be no observed mass change. TGA is used in the analysis of ceramics and thermally stable polymers.



2. Oxidation and\or Combustion: The simplest materials characterization is the residue remaining after a reaction. The thermogravimetric analyzer would combustion the sample by heating it beyond the ignition temperature of a sample. The resultant TGA curve plotted with the y axis as percentage of initial mass would show the residue at the final point of the curve. Oxidative mass losses are the most common observable losses in TGA.

3. Thermo-gravimetric Kinetics: Thermogravimetric kinetics may be explored for insight into the reaction mechanisms of thermal decomposition. Activation energies of the decomposition process can be calculated using Kissinger method.

4. Operation in Combinations with Instruments: TGA continuously weighs a sample as it is heated to temperatures of up to 2000 °C for coupling with FTIR and Mass spectrometer gas analysis. As the temperature increases, various components of the sample are decomposed and the weight percentage of each resulting mass change can be measured.

DIFFERENTIAL SCANNING CALORIMETER (DSC)



Introduction/Principle: DSC, is a thermo-analytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference is measured as a function of temperature. Both the sample and reference are maintained at nearly the same temperature throughout the experiment. Generally, the temperature program for a DSC analysis is designed such that the sample holder temperature increases linearly as a function of time. The reference sample should have a well-defined heat capacity over the range of temperatures to be scanned. DSC measures energy directly and allows precise measurements of heat capacity.

The basic principle is that when the sample undergoes a physical transformation such as phase transition, more or less heat will need to flow to it than the reference to maintain both at the same temperature.

Application/User's purpose: DSC is widely used across a range of applications, both as a routine quality test and as a research tool.

1. Polymers: DSC is used widely for examining polymeric materials to determine their thermal transitions. The observed thermal transitions can be utilized to compare materials.

2. Liquid crystal: DSC is used in the study of liquid crystal. As some forms of matter go from solid to liquid they go through a third state, which displays properties of both phases. This anisotropic liquid is known as a liquid crystalline or mesomorphous state. Using DSC, it is possible to observe the small energy changes that occur as matter transitions from a solid to a liquid crystal and from a liquid crystal to an isotropic liquid.

3. Oxidative stability: DSC for the study of oxidative stability requires an airtight sample chamber. Usually, such tests are done isothermally (at constant temperature) by changing the atmosphere of the sample. Such analysis can be used to determine the stability and optimum storage conditions for a material or compound.

4. Drug analysis: DSC is widely used in the pharmaceutical and polymer industries. DSC is a handy tool for studying curing processes, which allows the fine tuning of polymer properties. The cross-linking of polymer molecules that occurs in the curing process is exothermic, resulting in a positive peak in the DSC curve that usually appears soon after the glass transition.

5. Chemical analysis: Freezing point depression can be used as a purity analysis tool when analysed by DSC. This is possible because the temperature range over which a mixture of compounds melts is dependent on their relative amounts.

ELEMENTAL (C, H, N, S) ANALYZER

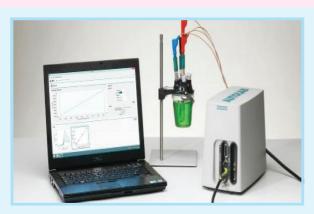
Introduction: CHNS elemental analysers provide a means for the rapid determination of carbon, hydrogen, nitrogen and sulphur in organic matrices and other types of materials. They are capable of handling a wide variety of sample types, including solids, liquids, volatileand viscous samples, in the fields of pharmaceuticals, polymers, chemicals, environment, food and energy. Simultaneous CHNS analysis requires high temperature combustion in an oxygenrich environment and is based on the classical Pregl-Dumas method. This combustion can be carried out under both static conditions i.e. introduction of a set volume of oxygen or dynamic conditions i.e. a constant flow of oxygen for a set period of time. Often, catalysts are also added to the combustion tube in order to aid conversion.

Principle: In the combustion process (furnace 1000°C), carbon is converted to carbon dioxide; hydrogen to water; nitrogen to nitrogen gas and sulphur to sulphur dioxide. If other elements such as chlorine are present, they will also be converted to combustion products, such as hydrogen chloride. A variety of absorbents are used to remove these additional combustion products as well as some of the principal elements, sulphur for example, if no determination of these additional elements is required. The combustion products are swept out of the combustion chamber by inert carrier gas such as helium and passed over heated (about 600°C) high purity copper. This copper can be situated at the base of the combustion chamber or in a separate furnace. The function of this copper is to remove any oxygen not consumed in the initial combustion and to convert any oxides of nitrogen to nitrogen gas. The gases are then passed through the absorbent traps in order to leave only carbon dioxide, water, nitrogen and sulphur dioxide.

Application/User's purpose: CHNS elemental analysers have been used in analytical laboratories for over thirty years. The method is



used extensively across a wider range of applications, including pharmaceuticals, chemicals, oil-related products, catalysts and food. In the oil industry, an important application is the regular monitoring of coke build-up on refinery catalysts to ensure that regeneration procedures (involving controlled burning of the carbon) are executed at optimal intervals. Since many of these catalyst systems involve large quantities of noble metals such as platinum, palladium and rhenium, mismanagement of this testing would entail serious financial losses. In food analysis, the determination of nitrogen (as a surrogate for protein) is very important for pricing grain and evaluating meat products, and is increasingly undertaken by combustion analysis.



Introduction: A potentiostat is the electronic hardware required to control a three electrode cell and run most electro-analytical experiments. The system functions by maintaining the potential of the working electrode at a constant level with respect to the reference electrode by adjusting the current at an auxiliary electrode. This equipment is fundamental to modern electrochemical studies for investigations of reaction mechanism related to redox chemistry and other chemical phenomena. Modern potentiostat are designed to interface with a personal computer and operate through a dedicated software package. The automated software allows the user rapidly to shift between experiments and experimental conditions.

ELECTROCHEMICAL WORKSTATION

Principle: A potentiostat measures the potential difference between the working and reference electrode, applies the current through the counter electrode and measures the current as an iR voltage drop over a series resistor (R_m). The control amplifier (CA) is responsible for maintaining the voltage between the reference and the working electrode as closely as possible to the voltage of the input source E. It adjusts its output to automatically control the cell current so that a condition of equilibrium is satisfied. from an electrical point of view, the electrochemical cell and the current measurement resistor (R_m) may be regarded as two impedances. Z, includes R in series with the interfacial impedance of the counter electrode and the solution resistance between the counter and the reference. Z₂ represents the interfacial impedance of the working electrode in series with the solution resistance between the working and the reference electrodes. The role of the control amplifier is to amplify the potential difference between the positive (or noninverting) input and the negative (or inverting) input.

Application/User's purpose: Electrochemical workstation (potentiostat) has many applications for user's purpose. Few are listed as below...

- 1. Amperometry
- 2. Cyclic Voltammetry
- 3. Coulometry
- 4. Polarography
- 5. Square Wave Voltammetry

FOURIER TRANSFER INFRA-RED SPECTROSCOPY (FTIR)

Introduction: FTIR is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas. An FTIR spectrometer simultaneously collects high-spectral-resolution data over a wide spectral range. This confers a significant advantage over a dispersive spectrometer, which measures intensity over a narrow range of wavelengths at a time. The term Fourier-transform infrared spectroscopy originates from the fact that a Fourier transform (a mathematical process) is required to convert the raw data into the actual spectrum.

Principle: Fourier-transform spectroscopy is a less intuitive way to obtain the same information. Rather than shining a monochromatic beam of light at the sample, this technique shines a beam containing many frequencies of light at once and measures how much of that beam is absorbed by the sample. Next, the beam is modified to contain a different combination of frequencies, giving a second data point. This process is repeated many times. Afterward, a computer takes all this data and works backward to infer what the absorption is at each wavelength.

The interferogram has to be measured from zero path difference to a maximum length that depends on the resolution required. The interferogram is converted to a spectrum by Fourier transformation. This requires it to be stored in digital form as a series of values at equal intervals of the path difference between the two beams. The result of Fourier transformation is a spectrum of the signal at a series of discrete wavelengths.



Application/User's purpose:

1. Microscopy and imaging: An infrared microscope allows samples to be observed and spectra measured from regions as small as 5 microns across. Images can be generated by combining a microscope with linear or 2-D array detectors.

2. Nanoscale and spectroscopy below the diffraction limit: The spatial resolution of FTIR can be further improved below the micrometer scale by integrating it into scanning near-field optical microscopy platform. The corresponding technique is called nano-FTIR





Introduction: Raman spectroscopy is used to observe vibrational, rotational, and other low-frequency modes in a system. Raman spectroscopy is commonly used in chemistry to provide a structural fingerprint by which molecules can be identified.

Principle: Raman spectroscopy relies on inelastic scattering, or Raman scattering, of monochromatic light, usually from a laser in the visible, near infrared, or near ultraviolet range. The laser light interacts with molecular vibrations, phonons or other excitations in the system, resulting in the energy of the laser photons being shifted up or down. The shift in energy gives information about the vibrational modes in the system. Infrared spectroscopy yields similar, but complementary, information.

Typically, a sample is illuminated with a laser beam. Electromagnetic radiation from the illuminated spot is collected with a lens and sent through a monochromator. Elastic scattered radiation at the wavelength corresponding to the laser line (Rayleigh scattering) is filtered out by either a notch filter, edge pass filter, or a band pass filter, while the rest of the collected light is dispersed onto a detector.

Application/User's purpose: Raman spectroscopy is used in chemistry to identify molecules and study chemical bonding. Raman provides a fingerprint to identify molecules. In solid-state physics, Raman spectroscopy is used to characterize materials, measure temperature, and find the crystallographic orientation of a sample. As with single molecules, a solid material can be identified by characteristic phonon modes. Information on the population of a phonon mode is given by the ratio of the Stokes and anti-Stokes intensity of the spontaneous Raman signal. Raman spectroscopy can also be used to observe other low frequency excitations of a solid, such as plasmons, magnons, and superconducting gap excitations. Distributed temperature sensing (DTS) uses the Ramanshifted backscatter from laser pulses to determine the temperature along optical fibers. The orientation of an anisotropic crystal can be found from the polarization of Raman-scattered light with respect to the crystal and the polarization of the laser light, if the crystal structure's point group is known.

In nanotechnology, a Raman microscope can be used to analyze nanowires to better understand their structures, and the radial breathing mode of carbon nanotubes is commonly used to evaluate their diameter.

X-RAY DIFFRACTION (XRD)

Introduction/Principle: XRD is a scientific technique using X-ray on powder or microcrystalline samples for structural characterization of materials. A diffractometer produces waves at a known frequency, which is determined by their source. The source is often x-rays, because they are the only kind of light with the correct frequency for inter-atomic-scale diffraction. When these waves reach the sample, the atoms of the sample act just like a diffraction grating, producing bright spots at particular angles. By measuring the angle where these bright spots occur, the spacing of the diffraction grating can be determined by Bragg's law. Because the sample itself is the diffraction grating, this spacing is the atomic spacing.

Application/User's purpose: Relative to other methods of analysis, powder diffraction allows for rapid, non-destructive analysis of multicomponent mixtures without the need for extensive sample preparation.

1. Phase identification: The most widespread use of powder diffraction is in the identification and characterization of crystalline solids, each of which produces a distinctive diffraction pattern. Both the positions (corresponding to lattice spacings) and the relative intensity of the lines in a diffraction pattern are indicative of a particular phase and material, providing a "fingerprint" for comparison.

2. Crystallinity: Powder XRD can be used to determine the crystallinity by comparing the integrated intensity of the background pattern to that of the sharp peaks.

3. Lattice parameter: The position of a diffraction peak is 'independent' of the atomic positions within the cell and entirely determined by the size and shape of the unit cell of the crystalline phase. Each peak represents a certain lattice plane and can therefore be characterized by a Miller index.

4. Phase transitions: At some critical set of conditions, for example 0 °C for water at 1 atm, a new arrangement of atoms or molecules



may become stable, leading to a phase transition. At this point new diffraction peaks will appear or old ones disappear according to the symmetry of the new phase.

5. Crystal structure refinement and determination: Crystal structure determination from powder diffraction data is extremely challenging due to the overlap of reflections in a powder experiment. A number of different methods exist for structural determination, such as simulated annealing and charge flipping. The crystal structures of known materials can be refined, i.e. as a function of temperature or pressure, using the Rietveld method.



Introduction: AFM is a type of scanning probe microscopy (SPM), with demonstrated resolution on the order of fractions of a nanometer, more than 1000 times better than the optical diffraction limit. The information is gathered by "feeling" or "touching" the surface with a mechanical probe. Piezoelectric elements that facilitate tiny but accurate and precise movements on (electronic) command enable very precise scanning.

Principle: The AFM consists of a cantilever with a sharp tip (probe) at its end that is used to scan the specimen surface. The cantilever is typically silicon or silicon nitride with a tip radius of curvature on the order of nanometers. When the tip is brought into proximity of a sample surface, forces between the tip and the sample lead to a deflection of the cantilever according to Hooke's law.^[5] Depending

ATOMIC FORCE MICROSCOPY (AFM)

on the situation, forces that are measured in AFM include mechanical contact force, van der Waals forces, capillary forces, chemical bonding, electrostatic forces, magnetic forces (see magnetic force microscope, MFM), Casimir forces, solvation forces, etc. Along with force, additional quantities may simultaneously be measured through the use of specialized types of probes (see scanning thermal microscopy, scanning joule expansion microscopy, photothermal microscopy, etc.). The AFM can be operated in a number of modes, depending on the application. In general, possible imaging modes are divided into static (also called *contact*) modes where the cantilever is vibrated or oscillated at a given frequency.

Application/User's purpose: The AFM has been applied to problems in a wide range of disciplines of the natural sciences, including solid-state physics, semiconductor science and technology, molecular engineering, polymer chemistry and physics, surface chemistry, molecular biology, cell biology, and medicine.

1. Solid state physics: (a) the identification of atoms at a surface, (b) the evaluation of interactions between a specific atom and its neighboring atoms, and (c) the study of changes in physical properties arising from changes in an atomic arrangement through atomic manipulation.

2. Molecular biology: AFM can be used to study the structure and mechanical properties of protein complexes and assemblies.

3. Cellular biology: AFM can be used to attempt to distinguish cancer cells and normal cells based on a hardness of cells, and to evaluate interactions between a specific cell and its neighboring cells in a competitive culture system. AFM can also

PARTICLE SIZE/ZETA POTENTIAL/MOLECULAR WEIGHT ANALYZER

Introduction: This instrument is flexible analytical tools for characterizing the physical properties of small particles. Depending on the configuration and application the system can be used as a particle size analyzer, or also used to measure zeta potential, molecular weight, (MW) and second virial coefficient (A2).

Principle: Particle size is measured by using dynamic light scattering (DLS). The sample particles in the cell experience Brownian motion. A light source is introduced into the cell, and the scattered light is collected at either 90° or 173° as shown in the diagram below. The system automatically selects the optimum scattering angle and cell position depending on the sample concentration and intensity.

Zeta potential analysis is made either in a disposable plastic cell, or a dip cell. The system measures the sample conductivity, applies an electric field, and then measures the motion of the particles using electrophoretic light scattering.

Molecular weightis determined by performing static light scattering measurements on a sample at several different concentrations. A Debye plot of KC/R vs. concentration produces results for both molecular weight and second virial coefficient.

Application/User's purpose:

1. Particle size analysis: It is performed by dynamic light scattering (DLS). Depending on the physical properties of the sample, the dynamic range is 0.3 nm – 8 μ m. The lower limit is influenced by concentration, how strongly the sample scatters light, and the



presence of large, unwanted particles. The upper limit is influenced by the density of the sample since DLS is modeled on all motion coming from Brownian motion, not gravitational settling.

2. Zeta potential: Charge on the surface of particles is characterized by measuring the zeta potential of a suspension. The sample is injected into a disposable cell and a measurement of the particle electrophoretic mobility results in the calculated zeta potential. The zeta potential of the sample is most often used as an indicator of dispersion

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