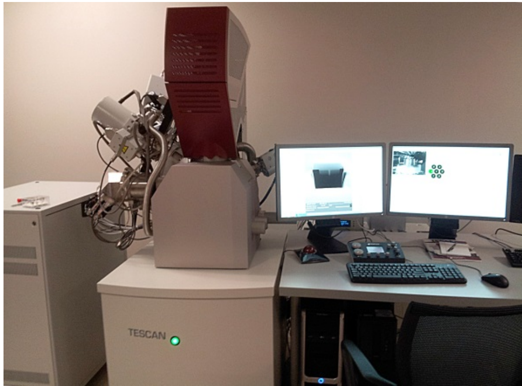
Capabilities of the Materials Characterization Facility (MCF) at TAMU

The Materials Characterization Facility (MCF) at Texas A&M University is a core user facility supported by the Office of the Vice President for Research, the College of Engineering/TEES, and the College of Science. The MCF provides researchers in the TAMU community with access to high-end instrumentation essential for fundamental studies of the surface and interfacial properties of materials, such as ion and electron based spectroscopies, and electron, optical and scanning probe microscopies. The MCF is staffed by research scientists with expertise in these areas, and they provide fundamental research training to students and faculty on our instrumentation, as well as consultation on measurement needs and data interpretation. Beyond TAMU, the MCF also supports collaborative research projects with outside industrial users. In addition to research training, the facility also supports educational activities involving lab tours, workshops, hands on demonstrations, and STEM outreach through our open house and lunchtime seminar series. The capabilities at the MCF include:

Electron Microscopy:

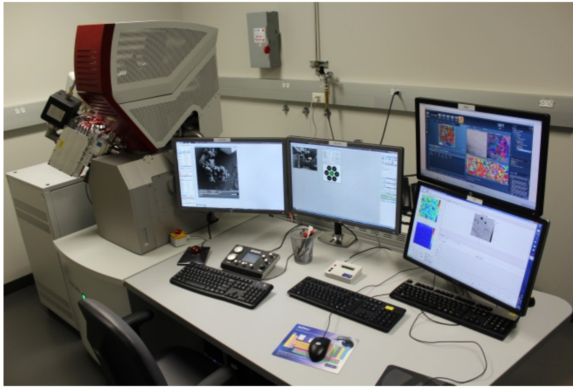
- ***Scanning Electron Microscope:*** The JEOL JSM-7500F is an ultra-high resolution cold field emission scanning electron microscope (FE-SEM) equipped with a high brightness conical FE gun and a low aberration conical objective lens; conventional in-chamber Everhart-Thornley and through-the-lens secondary detectors, low angle back-scattered electron detector (LABE), IR-CCD chamber camera, Oxford EDS system equipped with X-ray mapping and digital imaging.

- ***Focused Ion Beam (Xe plasma source, Tescan FERA-3 Model GMH):*** Dual beam Focused Ion Beam Microscope equipped with: Schottky Field Emission Electron Source; SE, BSE detectors; Integrated Plasma Ion Source (Xe) Focused Ion Beam (FIB); DrawBeam Basic Electron and Ion Beam Lithography Software; Motorized Retractable Panchromatic Cathodoluminescence Detector (350-650 nm spectral range); MonoGIS Gas Injection System (Platinum); Standard EBSD with a NordlysNano high sensitivity camera and 3D EBSD capabilities; Integrated Time-of-Flight Mass Spectrometer (TOF-SIMS).

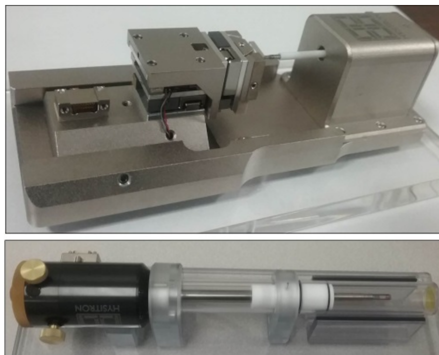


- ***In-situ Tensile Stage:*** *In-situ* thermo-mechanical testing module for SEM with EBSD. The Kammrath & Weiss *in-situ* thermo-mechanical testing module allows dynamic microstructural observations in SEM at high temperatures under different mechanical loading conditions. The loading stage is equipped with gear boxes, covering the range of 1-150 µm/s velocities. The loading stages are capable of performing tension, compression and bending tests using a 10KN as well as 500N load cells. The stage is equipped with the adaptation for EBSP measurements and also has a heating sub-stage capable of heating specimens mounted on the loading apparatus up to 1000 ^{o}C.

- ***Focused Ion Beam (Ga source, Tescan LYRA -3 Model GMH):*** Dual beam Focused Ion Beam Microscope equipped with: Schottky Field Emission Electron Source; SE, BSE detectors; STEM (dark and bright field imaging); EBIC imagining system (electron beam induced conductivity); fully integrated Canion Ga LMIS Focused Ion Beam column; 5-Reservoir Gas Injection System: W deposition, Pt deposition, Insulator (SiO_x) deposition, Enhanced Etching (H_2O), Enhanced or selective etching of Si, SiO_2, Si_3N_4, W (XeF_2); SmarAct 3-axis (XYZ) Piezo Nanomanipulator and controller; Beam Deceleration Mode for imaging at low voltage; Standard EDS Microanalysis System with X- MaxN 50.



- ***Picoindentors:*** The *in situ* PI 95 TEM/PI 85 PicoIndenters are full-fledged depth-sensing nanoindenters capable of direct-observation of nanomechanical tests inside the TEM and SEM respectively. Both PicoIndenters provide quantitative force-displacement data which can be time-correlated to real-time events in the TEM/SEM videos.



PI 85 SEM transducer assembly (top); and PI 95 TEM Transducer (bottom)

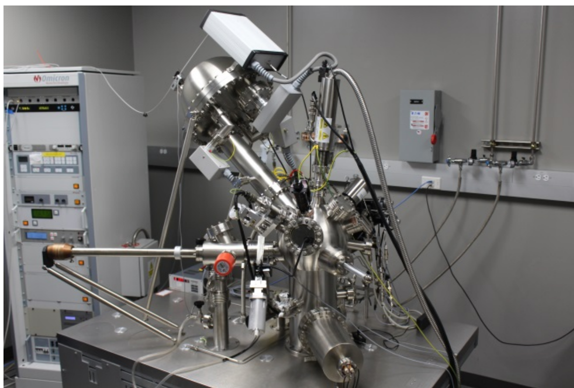
- ***Electron Microprobe:*** The Cameca SXFive has an LaB_6 source and is equipped with EDS,and CL detector. The instrument has five spectrometers with the following crystal configuration: (1) LTAP and LPET; (2) TAP, PET, PC0, and PC2; (3) LPET and LLiF; (4) PET, LiF, PC1, and PC3; (5) LPET and LLiF

- ***Themis Titan TEM:*** The Titan Themis[3] 300 S/TEM is a high resolution transmission electron microscope with spherical aberration correctors (Cs) for both the image and probe optics system, resulting in resolution limits below 1 Å between 60 and 30 kV in both TEM and STEM mode. The high brightness electron gun (X-FEG) is equipped with a monochromator to improve energy resolution in

combination with a high-sensitivity SDD X-ray spectrometer (Super-X) and a high-resolution post-column energy filter (GIF Quantum). Additional capabilities: energy filtered TEM (EFTEM) imaging, high-resolution electron energy-loss spectroscopy (EELS), energy-dispersive X-ray spectroscopy (EDXS), and electron tomography. The Titan Themis^3 300 can also be used to perform *in situ* experiments using special TEM specimen holders.

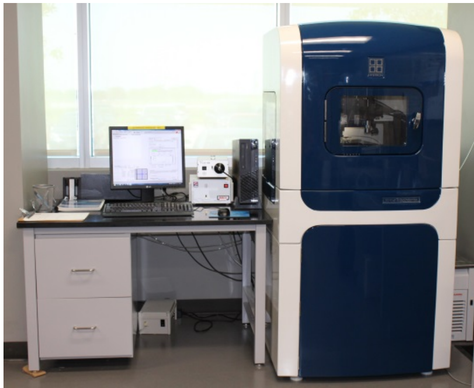
Surface analysis:

- ***XPS/UPS:*** Omicron XPS/UPS system with Argus detector uses Omicron's DAR 400 dual Mg/Al X-ray source for XPS measurements and the HIS 13 He UV source for UPS measurements. Electron analysis can be done with Omicron's 124 mm mean radius electrostatic hemisperical dispersive energy analyzer with the 128-channel micro-channelplate Argus detector with 0.8 eV resolution. This system is also equipped with a CN10 charge neutralizer to reduce charging on samples such as polymers and an NGI3000 Argon ion sputter gun for surface cleaning.



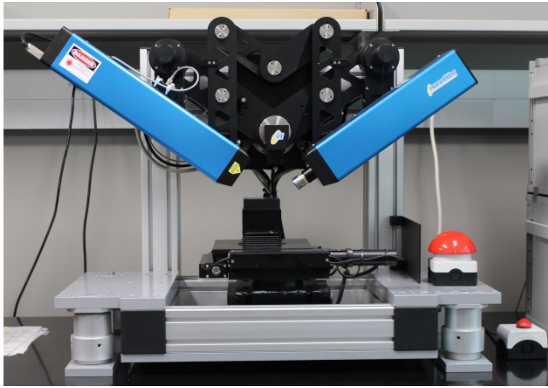
- ***Nanoindenter:*** The TI 950 Triboindenter is equipped with performech Advanced Control Module which provides great performance for nanomechanical testing. It is equipped with integrated dual head testing for low load and high load performance that enables testing at the nano/micro scale levels for both hard and soft materials. It has improved lateral measurements for thin film samples, xSol high temperature stage having the range of 20 °C up to 800 °C, extended displacement stage –

suited for testing adhesive and compliant samples. In addition, it has a fluorescence microscope option capable of performing both standard bright-field and fluorescence imaging, NanoDMA and Modulus Mapping for quantitative measurements of viscoelastic nanomechanical properties from the in situ SPM imaging and TriboEA for acoustic emission signals from fracture or deformation.



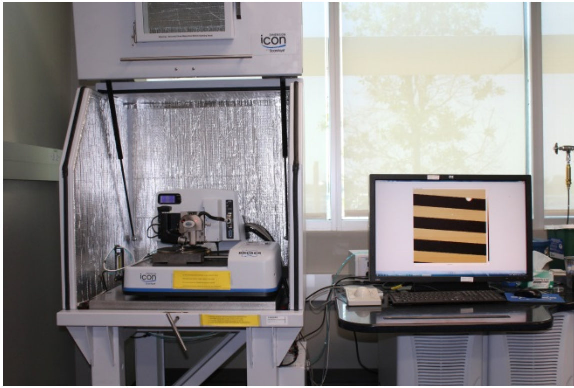
- ***AFM-IR:*** The Anasys Instruments nanoIR2-s™ combines nanoscale chemical characterization AFM-IR (Atomic Force Microscopy–Infrared Spectroscopy) with optical property mapping s-SNOM (scattering Scanning Near Field Optical Microscopy). AFM-IR provides the spatial resolution of AFM with chemical analysis capabilities of infrared spectroscopy (IR). An AFM probe is used to locally detect the thermal expansion of sample(s) resulting from absorption of infrared radiation at the resonant wavelength. IR spectra are then collected by measuring the cantilever oscillation amplitude as a function of IR wavelength, creating a unique chemical fingerprint with nanoscale spatial resolution. The s-SNOM technique uses a metallized AFM tip to enhance and scatter radiation from the tip in proximity to the sample. The scattered radiation carries information about the complex optical properties of the sample under the metallized tip.

- ***Imaging Ellipsometer:*** The Nanofilm EP3-SE is a high-precision, auto-nulling spectroscopic imaging ellipsometer in the PCSA configuration with ellipsometric resolution of up to Δ ±0.002 deg and Ψ ± 0.001 deg and accuracy of ±0.1 deg. It is equipped with a Xenon arc lamp, allowing spectroscopic ellipsometric scanning from 365-1000 nm at 46 wavelengths—a useful capability for the determination of optical properties for complex films and stacks. Additionally, a CCD camera allows for the capture of optical and ellipsometric images.



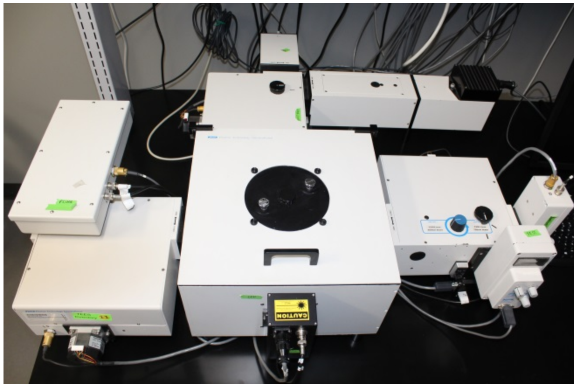
- ***Cameca ion microprobe:*** The CAMECA IMS 4f ion microprobe is a tool for investigating isotopic composition in the chemical, material, geological and biological sciences. All elements (H to U) can be detected in depth profiling, surface, bulk and microanalysis modes. Detection limits are in the ppb range with depth resolution of 10 nm and lateral resolution of ~3 µm. Typical applications include in-depth compositional analysis of high performance materials, isotopic ratios in terrestrial/extraterrestrial specimens, localization/imaging of ^{13}C- and ^{15}N-labeled molecules in biological materials.

- ***Dimension Icon AFM:*** This AFM is equipped with Peak Force Tapping using ScanAsyst for topography and phase images; contact mode; force imaging for elastic properties of materials from force curves plots; intermittent mode (tapping) for topography and phase images; imaging in a liquid environment; peak force TUNA for topography, current images, current-voltage (I-V) plot; Peak Force Quantitative NanoMechanics for modulus, adhesion, deformation and dissipation measurements; magnetic force microscopy for long range magnetic forces on the sample surface and a Peltier heater/cooler stage with the range of -20 °C up to 200 °C.



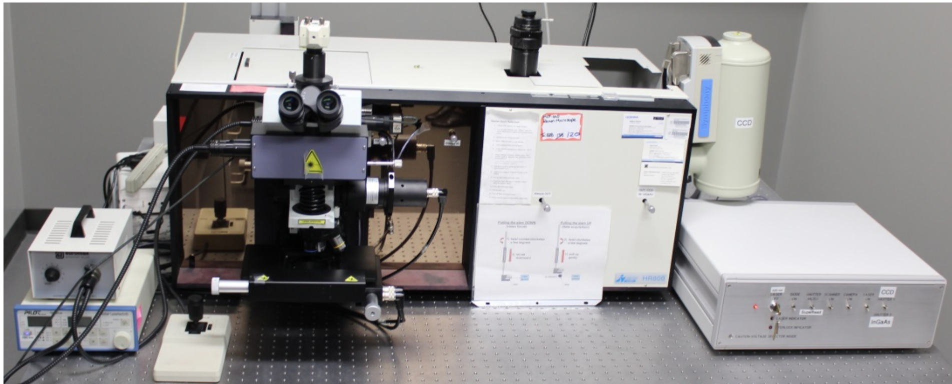
Spectroscopy and Microscopy

- ***Spectrofluorometer:*** The PTI QuantaMaster series spectrofluorometer is a modular system with capabilities for measuring many luminescence phenomena for both liquid and solid (film or powder) samples. It is equipped with a Xenon arc lamp for collecting steady state emission spectra and a pulsing Xenon lamp for measuring phosphorescence lifetimes. Additionally, several LED sources are available at specific wavelengths that can be attached for collecting fluorescence lifetime measurements. FelixGX software can be used to collect and analyze excitation and emission scans, excitation and emission ratios, time-based scans for single samples or up to 10 dyes simultaneously, lifetime measurements, and quantum yield.

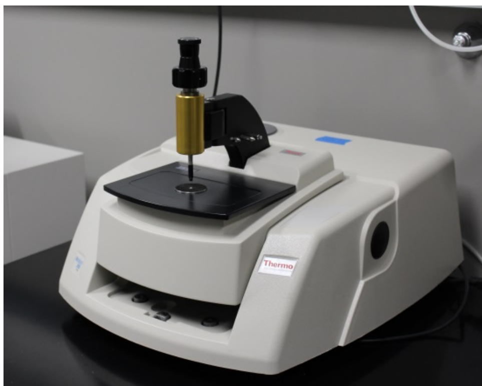


- ***UV-Vis-NIR spectrophotometer:*** The Hitachi U-4100 UV-Vis-NIR spectrophotometer is a high resolution spectrometer capable of measuring absorbance, transmittance, and reflectance of both liquid and solid (film) samples from 175-3300 nm.

- ***Raman confocal microscope:*** The Horiba Jobin-Yvon LabRam IR system provides highly specific spectral fingerprints which enables precise chemical and molecular characterization and identification. It offers optimal confocal spatial and depth discrimination down to 1 µm, two laser options (632 nm and 785 nm), and automated XYZ mapping. The spectrometer is equipped with two gratings and an open electrode CCD with enhanced quantum efficiency in the spectral range 450 – 950 nm.



- ***FTIR spectrometer:*** The Thermo Nicolet 380 FTIR spectrometer is equipped with a standard transmission stage that holds various sample preparations. It has diamond tipped ATR stage for measurements ranging from 3000 to 200 cm^{-1} with spectral range of 7800 to 350 cm^{-1} and 0.9 cm^{-1} spectral resolution.



- ***Fluorescent confocal microscope:*** The Leica TCS SP5 utilizes an inverted DMI 6000 microscope which is equipped with a motorized xy stage and epifluorescence illumination. Filter cubes for blue and

green excitation (I3, N2.1) are available for visual inspection and focusing of samples. The conventional scanner has one transmitted light detector and three reflectance/fluorescence detectors. The excitation laser lines available are 458, 476, 488, 514, 543 and 633 nm and the microscope is equipped with 10x, 40x and 63x dry objectives and a 63x oil immersion objective.

Thermal and Electrical Analysis

- ***Thermal mechanical analysis (TMA):*** The thermo-mechanical analyzer measures changes in the dimensions of a sample as a function of time, temperature and force in a controlled atmosphere. TMA can measure the coefficient of thermal expansion along with the glass transition temperature (Tg). Creep and stress relaxation analysis, and softening and melting points can be measured for solids (soft or rigid samples) under various modes of operation in a temperature range between -150 to 1000 °C, with force range of 0.001 to 1 N.

- ***Differential scanning calorimetry (DSC):*** The capabilities of this DSC include a temperature range from -90 to 400 °C with a sensitivity of 1 µW for measuring the glass transition temperatures (T_g), melting points, crystallization, heat flow, thermal history, kinetics (isothermal crystallization) and degree of cure.

- ***Dielectric spectroscopy:*** The Novocontrol Alpha impedance analyzer is equipped with Quatro cryosystem for dielectric studies with an impedance range of 0.01 ohms to 1014 ohms, a frequency range of 3 µHz to 20 MHz, and a temperature range of -160 to 400 °C with nitrogen gas cooling/heating. It can accommodate solid pellet, foil, and non-volatile liquid samples.

- ***Hot Disk thermal conductivity analysis:*** The Hot Disk Transient Plane Source (TPS) is equipped to measure the absolute thermal conductivity 0.005 to 1000 W/mK, and the thermal diffusivity with auto-calculation of heat capacity of bulk and directional (axial & radial) materials including solid, liquid, paste, and powder.