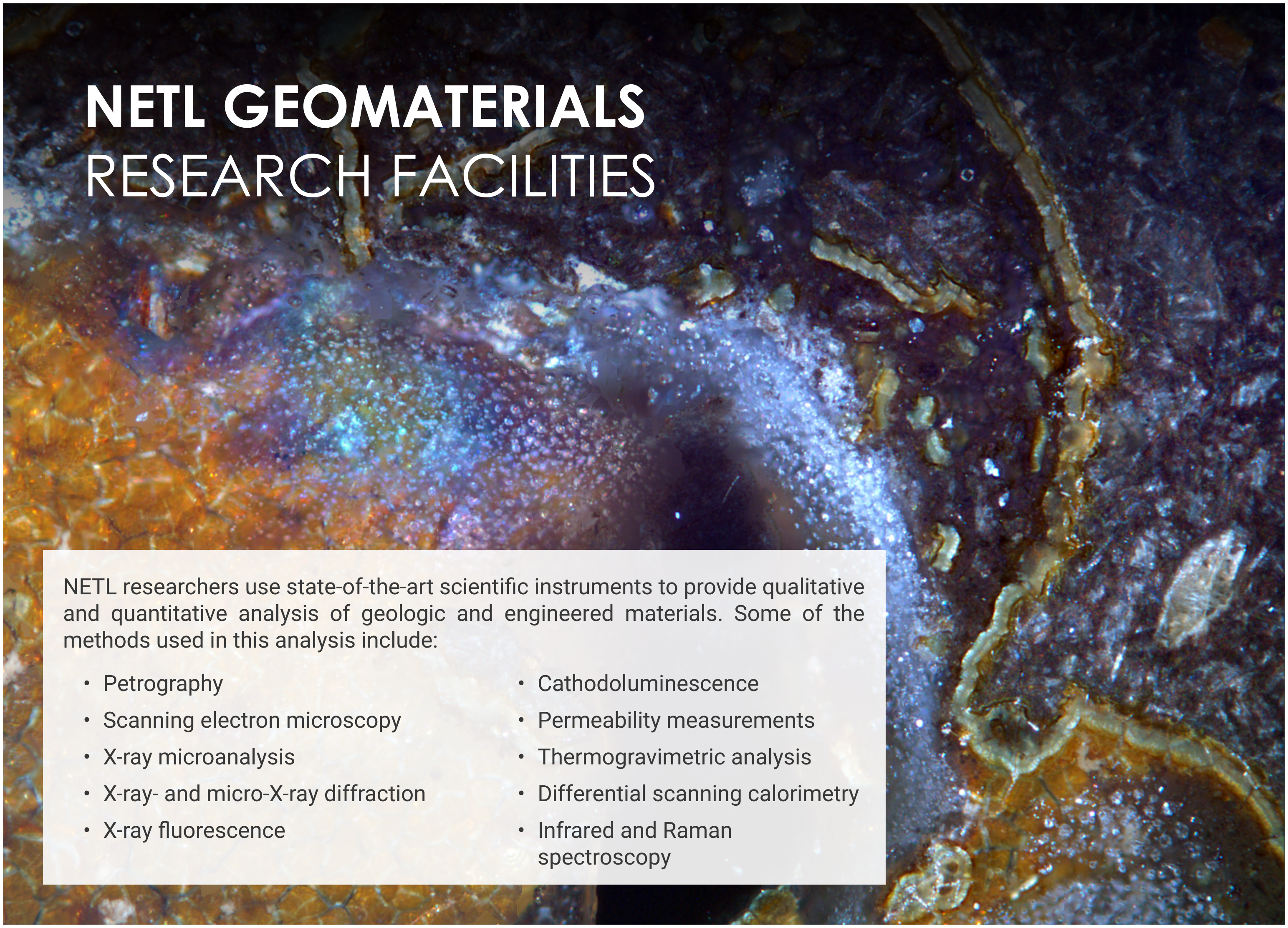
NETL GEOMATERIALS
RESEARCH FACILITIES



NETL researchers use state-of-the-art scientific instruments to provide qualitative and quantitative analysis of geologic and engineered materials. Some of the methods used in this analysis include:

- Petrography
- Scanning electron microscopy
- X-ray microanalysis
- X-ray- and micro-X-ray diffraction
- X-ray fluorescence
- Cathodoluminescence
- Permeability measurements
- Thermogravimetric analysis
- Differential scanning calorimetry
- Infrared and Raman spectroscopy

R&D176, February 2024

NATIONAL ENERGY TECHNOLOGY LABORATORY

As our nation transitions to greater reliance on renewable energy sources, NETL is tackling the grand challenges facing the safe and efficient use of our nation's fossil energy resources. Our researchers are coupling the aforementioned tools with their expertise in material science, geology, fluid-rock geophysics and fluid-rock geochemistry to examine geologic materials such as shales, coals, clays, limestone, sandstone, igneous rocks, carbonates and basalts — all to better understand the subsurface. Researchers also perform analyses on cements, ceramics, nanomaterials, catalysts, corrosion deposits, metal alloys, synthesis products and biological materials to understand the relationships that govern material performance.

The analysis techniques described in this fact sheet are used to help characterize the fundamental properties of both conventional and unconventional natural gas and oil reservoirs for both the onshore and offshore ultra-deepwater reservoirs. These properties provide insight into these reservoirs' geothermal properties, enhanced oil or gas recovery potential, and carbon and hydrogen subsurface storage potential. Information gained helps NETL and its partners better understand subsurface processes in the field, provide information for computational models or simulations and perform risk assessments from laboratory experimental studies.

Figure 1. Brightfield photomicrograph of Columbia River Basalt vug filled with iron-hydroxide.

PETROGRAPHY

NETL makes several petrographic microscopes accessible to researchers:

- The Laboratory's optical **petrographic and visible light microscopes** are capable of transmitted light petrographic analysis using polarized light on transparent rock sections cut and polished to 30 micrometers. Standard features include crossed-polars, a Bertrand lens and a first-order red plate. Digital cameras on these scopes send captured images to computers for detailed analyses
- A **reflected light microscope** illuminates polished opaque rock samples in a variety of wavelengths to induce fluorescence in organic or mineral components. Pore structures impregnated with fluorescent-dyed epoxy can be viewed under the correct illumination. The microscope has been modified in-house for particle analysis, allowing the shape and size of solid particles to be rapidly measured

Image analysis software and computer-controlled stages allow for high-powered scanning; creation of image mosaics; enhancement of contrast, color, features and illumination; and digital analysis of data. Rapid data analyses are achived through automated detection and volumetric assays of porosity and mineral content.

ELECTRON MICROSCOPY

NETL's Geological and Environmental Systems (GES) Directorate uses **scanning electron microscopes (SEM)** to analyze geomaterials by scanning them with a high-energy beam of electrons in a raster scan pattern. The beam interacts with the sample's atoms, and the signals provide information about the sample's surface topography, composition, electrical conductivity and other properties. GES uses electron microscopy to gain information about material morphology, elemental composition and distribution, crystalline phase orientation, and distribution, particle size, thermally induced morphological changes and mineral-ceramic characterization. Examples of multiuser electron microscopes include the JEOL JSM-IT700HR and FEI Quanta 600 field emission gun (FEG), which are used to image and analyze material surface structure to 10 nanometers, identify elemental concentrations qualitatively or quantitatively with detailed elemental maps, spot analysis, and line profiles. Software allows for the automated collection of X-ray data for preselected locations and enables data from multiple locations to be stitched together to produce a high-resolution image of an entire sample. The SEMs are equipped with three primary detector types: (1) secondary electron (SE), (2) backscattered electron (BSE), and (3) energy dispersive spectroscopy (EDS). In addition, the Quanta 600 FEG operates in three vacuum modes. High vacuum mode is used for the imaging and microanalysis of typically prepared, electrically conductive samples. Low-vacuum mode is used for the imaging and microanalysis of non-conductive specimens without special preparation, such as coating with a conductive material. Environmental (ESEM™) mode is used for high-vacuum-incompatible samples, such as hydrated materials. The sample chambers can accommodate large samples, allowing non-destructive investigations of a variety of sizes and materials. The JEOL JSM-IT700HR and Quanta 600 FEG are also equipped with electron back-scatter diffraction (EBSD) detectors to allow for the analysis of material structure and crystallographic orientations. In addition to the JEOL JSM-IT700HR and FEI Quanta 600 FEG, the microscopy capabilities also include a JEOL JSM-7600F, a high-resolution semi-in-lens Schottky electron source field emission SEM. The JSM- 7600F is equipped with an in-lens and Everhart-Thornley SE detector; co-linear retractable in-lens and low-angle BSE detectors; a transmission electron detector (TEM), an in-chamber infrared camera, r-filtering and gentle beam mode to enhance imaging. The JSM-7600F is interfaced with an EDAX EBSD system, a Thermo-Noran NS-7 microanalysis system (EDS), and a Gatan MonoCL-4 Cathodoluminescence system (CL). The CL system is especially suitable for geochemical analysis as CL is especially sensitive to specific types of trace element distributions and geochemical effects that allows geochemical processes to be reconstructed.

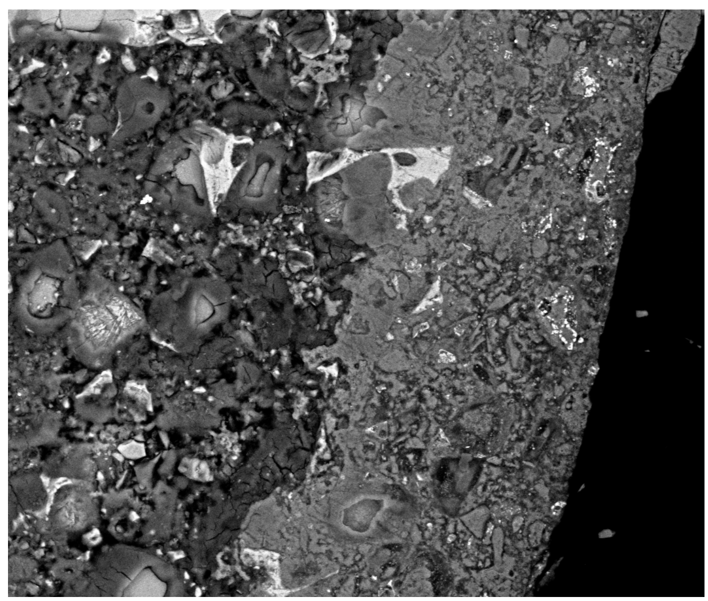


Figure 2. SEM backscattered electron image of Class H cement exposed to a CO_2-H_2S acid gas mixture and showing carbonation and pyrite formation.

ELECTRON MICROPROBE ANALYSIS

NETL's field emission electron microprobe (JEOL JXA-85030F) is outfitted with both wavelength dispersive spectroscopy (WDS), energy, EDS and CL to support a wide range of research projects. The instrument is capable of an image resolution of 3 nanometer (nm) at 30 kilovolts (kV)/10 nm at 1 kV, which is optimal for analyzing light elements (e.g., boron, carbon, oxygen and nitrogen), as well as trace element analysis. This specialized WDS system has five wavelength spectrometers that are configured to provide microanalysis of elemental composition simultaneously with the EDS detector. The CL detection of ultraviolet, visible and infrared wavelength spectrum (~250-800 nm) can be obtained by the CL detector concurrently with integrated backscatter analysis. All these detectors are calibrated to give hyperspectral elemental mapping, phase analysis, luminescence spectra and automatic montaging for samples up to 90×90 mm in size.

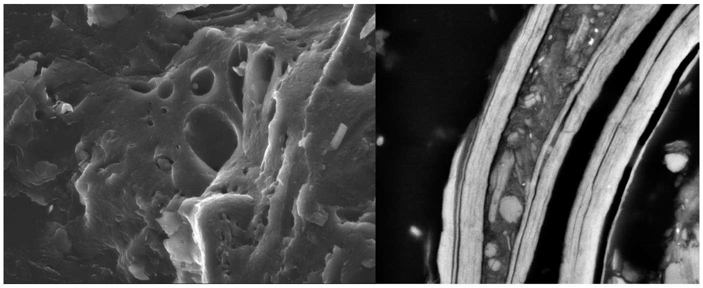


Figure 3. SEM images of the pore structure of coal (left) and clay in shale (right).

X-RAY DIFFRACTION

X-ray diffraction (XRD) is used to identify and quantify phases in crystalline materials as well as to investigate other characteristics such as crystallite size. The crystal's atomic structure determines how the crystal diffracts an X-ray beam. The material is identified by matching its diffraction pattern to like patterns found in extensive databases. XRD is an integral tool for robust characterization of materials both pre- and post-experimentation in a wide array of fields including geology, chemistry and materials science.

GES researchers use a Malvern Panalytical Empyrean diffractometer, two PANalytical X'Pert Pro powder diffractometers, a Malvern Panalytical Aeris bench-top diffractometer and a Rigaku Ultima III x-ray diffractometer to characterize materials utilized in experiments related to natural samples from the field or materials developed in the lab. The instruments are also used for in-situ experiments determining the effect of temperature on crystal structure and phase reactions. High-temperature stages are available for in situ analyses up to ~1200° Celsius and, under some conditions, above ~2000° Celsius. Depending on the specific stage selected, samples can be subjected to various atmospheres, for example hydrogen or carbon dioxide, and pressures to investigate the interactions of gases with solid samples. Other capabilities available on NETL's diffractometers include micro-diffraction, automated sample changers, Eulerian cradles, low angle and grazing incidence capabilities, pair distribution function analysis, a version of computed tomography analysis, an automated x,y,z stage, transmission, capillary and spinning stages.

GES uses micro-X-ray diffraction on the Empyrean diffractometer to analyze small amounts of material on reaction rims and in pore spaces in a variety of materials, both ex situ and in situ. Applications include powder diffraction of very small sample volumes, micro-diffraction of specific features, trace mineral analysis, in situ analysis, aggregate analysis, and measurements of weakly diffracting, disordered materials. In addition, experiments can be conducted in situ to monitor crystal structure and phase transitions at elevated temperature dry or in the presence of liquid utilizing one of the high-temperature stages.

PERMEABILITY MEASUREMENTS

The **Randolph Steady-State Core Analysis Laboratory (RASSCAL)** allows researchers to measure flows of gas through very low-permeability rocks under net pressures approximating those encountered underground. Temperature-controlled gas reference pressures, stable to about one part in 500,000, allow for actual steady-state gas flow measurements as low as one millionth of a standard cm^3 per second. Effects of increased net stress on gas permeability, such as those experienced during drawdown, can be duplicated, and the hysteresis of gas flow under stress cycling can be investigated.

Data gained through the RASSCAL are helping researchers better understand gas shale reservoir properties and the ability of a drained shale to accept CO_2 for storage. Measurements of rocks partially saturated with liquids are contributing to the knowledge of how shales behave as reservoir seals and how retrograde condensate shuts off gas flow in some shales.

THERMOGRAVIMETRIC ANALYSIS, DIFFERENTIAL SCANNING CALORIMETRY, INFRARED AND RAMAN SPECTROSCOPY

Using **thermogravimetry** and **differential scanning calorimetry**, GES researchers test geological and environmental material samples to determine degradation and decomposition temperatures, absorbed moisture content, solvent residues, levels of various components and reactivity toward CO_2. Materials routinely tested include formation rocks, various minerals, hydrocarbon-rich source rocks, coal and coal byproducts, soil samples, formation and produced waters, and production additives and modifiers for recovering unconventional fossil fuel resources.

NETL's **Fourier-Transform Infrared (FT-IR) Spectroscopy**, with high-temperature/high-pressure in-situ attenuated total reflectance (ATR) accessories (figure 4), helps researchers characterize the chemical and structural changes of a range of materials using a Nicolet IS-50 4700 FT-IR. It provides researchers the capability to study reaction mechanisms by identifying intermediates and reaction products formed in-situ during gas-liquid-solid reactions.

The Laboratory's **Fourier-Transform Infrared/Raman (FT-IR/Raman) Spectroscopy**, with microstage and high-temperature/high-pressure diffuse reflectance accessories, features a Nicolet 8700 with capability for rapid scanning, an FT-Raman configured with the research-grade 2.5 W Nd:YVO4 laser, multiple spectral ranges, advanced FT-IR research tools, are used for sample mapping, screening applications and kinetic analysis readily accessible with FT-Raman spectroscopy.

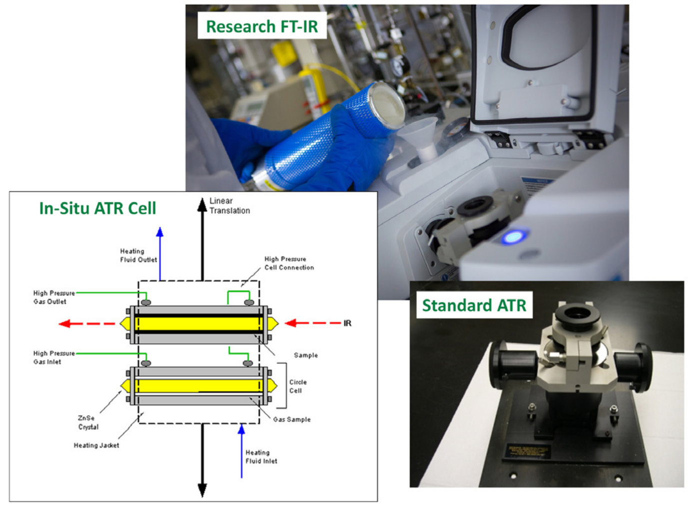


Figure 4. NETL researcher adds liquid nitrogen (top image) to FT-IR instrument using a standard ATR cell (right image) capable of quickly analyzing solid samples. Powdered/liquid samples can be analyzed In situ (left image) allowing control of pressure, temperature and gas type exposure.

For more information on evaluating geologic materials at NETL, please see the NETL Geoimaging Characterization Fact Sheet (R&D178)

NETL is a U.S. Department of Energy (DOE) national laboratory dedicated to advancing the nation's energy future by creating innovative solutions that strengthen the security, affordability and reliability of energy systems and natural resources. With laboratories and computational capabilities at research facilities in Albany, Oregon; Morgantown, West Virginia; and Pittsburgh, Pennsylvania, NETL addresses energy challenges through implementing DOE programs across the nation and advancing energy technologies related to fossil fuels. By fostering collaborations and conducting world-class research, NETL strives to strengthen national energy security through energy technology development.

Research Partners

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