SEM Technology
Advances Energy Research

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FEG-SEMs enable fast and accurate analysis of solar thin films, oil shale, and catalysts.

High-resolution field-emission scanning electron microscopes (FEG-SEMs) have proven to be very powerful tools for energy-related research. Developments in such areas as solar thin films, oil shale, catalysis, and fuel cells require sub-nanometer resolution SEMs with a versatile set of detectors. They also require advanced sample preparation and handling techniques, such as argon ion polishing and FIB (focused ion beam).

Several advanced technologies in FEG-SEMs are now available:

- LABe (low-angle backscattered electron) detector allows unprecedented imaging of specimens at both high and ultra-low kVs
- Application of specimen bias (Gentle Beam mode) allows examination of charging specimens without additional coating
- STEM detectors in SEM allow observation of sub-nanometer features

Furthermore, the cross-section polisher (CP), a defocused Ar^+ ion beam specimen preparation device, allows preparation of well-polished cross sections of challenging materials without smearing, strain, mechanical deformation, or other common artifacts.

This article discusses incorporation of both advanced sample preparation and handling techniques, and the newest SEM detectors and imaging capabilities to advance energy research.

Solar Thin Films

A solar cell converts the energy of sunlight directly into electricity via the photovoltaic effect. The holy grail of the industry is to produce low cost, highly efficient solar cells. Current research efforts focus on thin film growth, incorporation of smaller and cheaper crystals (CuInGaSe), and investigation of defect structures in silicon that reduce efficiency.

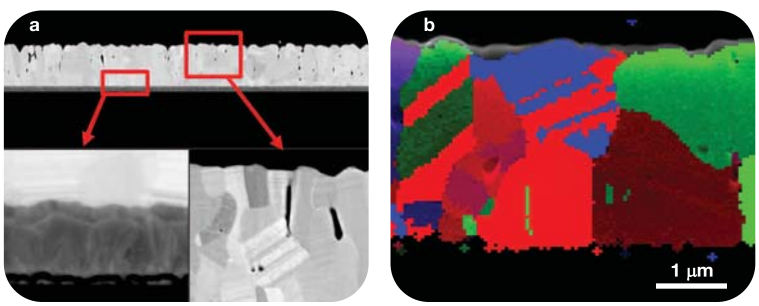

Fig. 1 — *(a) Solar panel thin film cross-section prepared with the CP. Insets show higher magnification images of the different layers of the film, with a clear view of the layer thickness and grain structure. (b) EBSD grain orientation map of the film structure.*

Over the last few years, we have successfully exploited the Cross Section Polisher for preparation of samples of these materials, and the FEG-SEM for study of solar thin film growth mechanisms. In particular, we have combined low kV high-resolution backscatter SEM imaging of film cross-sections **(Fig. 1a)** with EBSD (Electron Backscatter Diffraction) analysis (**Fig. 1b**) to study crystal growth and orientation of the films.

We have also developed a technique in which we can polish the top surface of the film in a grazing incidence configuration to investigate not only the crystal orientation, but also the porosity and grain packing of the film **(Fig. 2).**

Film growth direction, grain orientation, layer thickness, and porosity impact the ultimate performance and efficiency of the solar film, and are therefore significant for both R&D and quality control on the manufacturing side.

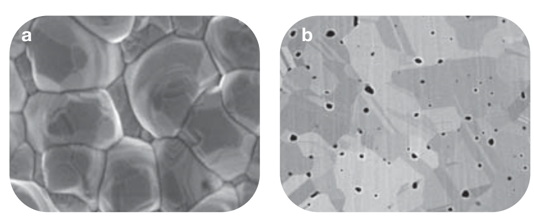

Fig. 2 — *Imaging top surface of a solar thin film: (a) as received; (b) after CP preparation, grain structure and porosity are clearly observed on the ion-polished surface.*

Oil Shale and Natural Gas

Oil shale is a fine-grained sedimentary rock that contains significant amounts of kerogen, a solid mixture of organic chemical compounds. The overall composition of the shale

**Member of Electronic Device Failure Analysis Society, an affiliate society of ASM International* determines the potential ease of oil or gas extraction. Researchers investigate shale porosity at both the macroscale and nanoscale to determine the potential of deposits to produce economically viable sources of oil and natural gas.

However, flat shale samples are notoriously difficult to prepare for SEM by standard mechanical methods, because they tend to crumble, and the features are often obscured by smearing. To overcome these challenges, we combined several techniques, including CP, FIB (Focused Ion Beam), and FEG-SEM.

First, a shale sample is prepared in the CP, producing a flat surface that allows easy and straightforward imaging in SEM because it has no smearing or mechanical deformation. Backscatter SEM images show unambiguously the location of various constituents such as kerogen, pores, clays, quartz, pyrites, etc. (**Fig. 3a**). Moreover, macro- and nano-pores are easily identifiable in CP-prepared specimens (**Fig. 3b).**

The sample can then be taken to a FIB. A specific location that has pores can be accurately sectioned and subsequently reconstructed in three dimensions to represent the pore network structure in a given volume (**Fig. 4**).

The combination of the above methods, as well as additional compositional analysis, enables researchers to create a comprehensive analysis of the shale deposits.

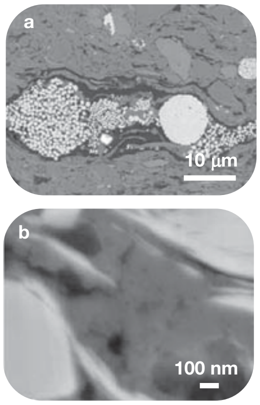

Fig.3 — *(a) Backscatter SEM image of the CP prepared shale specimen taken using LABe detector. Dark contrast shows location of kerogen, white contrast shows pyrite crystals, gray contrast corresponds to areas of clay, carbonates and quartz. (b) nano-pores in kerogen-rich area.*

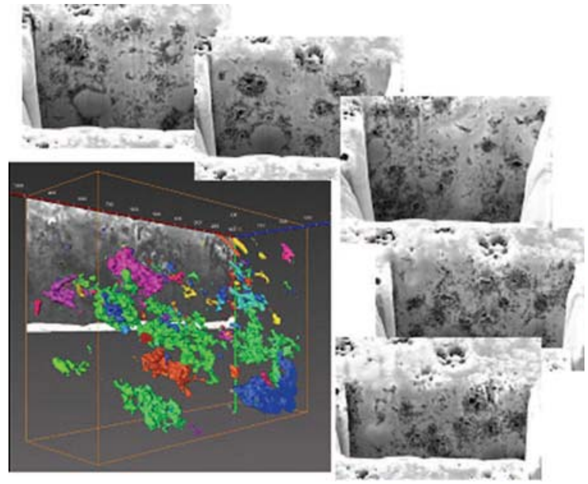

Fig. 4 —*Five out of 40 slices used to reconstruct a 3D volume of shale with the JIB-4600F FIB-SEM. A 3D rendition of the pore structure was done with Avizo software (from VSG).*

Catalysis

In the past, catalytic materials have been examined in SEMs at relatively low magnifications to observe overall morphology and perform EDS analysis of its constituents. Higher magnifications have been traditionally reserved for TEM and dedicated STEM analyses, because of the inherent higher resolving power of such instruments.

Current state-of-the-art FEG-SEM technology allows examination of catalysts using in-lens detectors (SE and BSE) as well as STEM detectors. For example, the in-lens detector technology allows observation of a catalyst with one to five nanometer metal particles on a carbonaceous support **(Fig. 5**).

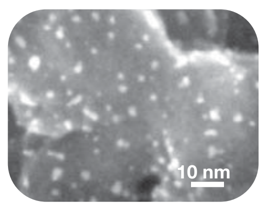

Fig. 5 — *Energy filtered image of a catalyst with sub 5 nm Pt/Pd metal particles imaged at 1,000,000X magnification.*

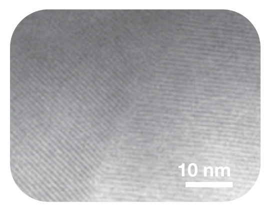

Fig. 6 — *STEM in SEM image of Faujasite showing 7.4 Å pore spacing.*

Furthermore, the STEM detector enables researchers to begin to image well into the sub-nanometer range – **Fig. 6** shows STEM image of Faujasite (zeolite Y) clearly resolving 0.74 nm pores.

Sensitive Sample Handling

Many of the sample types encountered in the fields described above require special sample handling prior to imaging. The samples are often highly chemically reactive due to their small size and high surface area. The samples can potentially react with oxygen or water, and therefore cannot be exposed to ambient conditions. These specimens can be transferred into the SEM chamber via atmosphere-isolated transfer vessels that in many cases serve as a reaction chamber for *in situ* experiments. JEOL recently developed a special air-lock chamber to allow transfer of air-sensitive specimens such as fuel cells to be imaged in the SEM without atmospheric exposure**.**

Summary

New detectors in conjunction with advances in FEG-SEM column and gun technologies allow researchers to expand the use of SEM for current energy related projects that require not only extreme resolution capabilities, but also versatility in sample handling and preparation. The ease of use of the FEG-SEM gives it a clear advantage over TEM or other traditional characterization methods for increasing characterization and analysis throughput. ❍

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