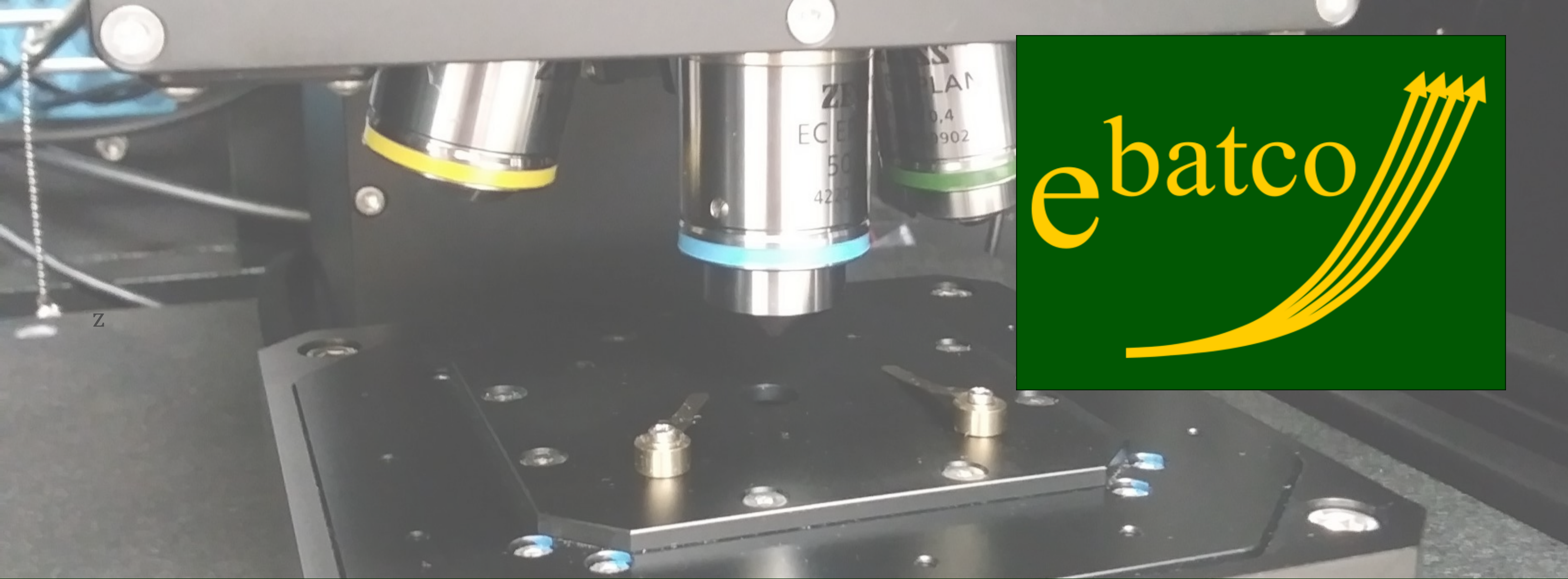

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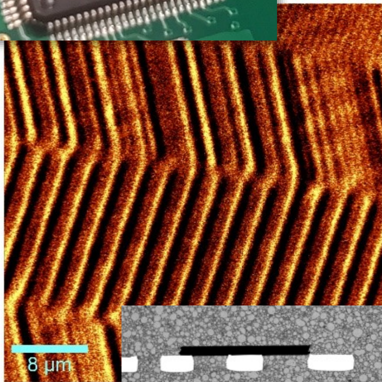

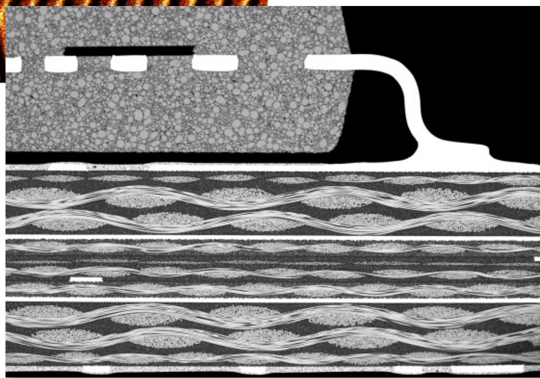

Nano/Micro Scale Analysis, Characterization and Testing of Semiconductor and Microelectronic Materials

Summary

This white paper focuses on nano and micro scale analysis, characterization, and testing of semiconductor and microelectronic materials. Encouraged by the Biden Administration's CHIPS and Science Act and the booming private semiconductor manufacturing investments, Ebatco, an established and well-recognized lab service power house for nano and microscale materials and device testing, has analyzed the testing needs of semiconductor and microelectronic industries and responded with suites of contract lab service solutions.

Introduction

Semiconductor and microelectronic material development and production are demanding processes. Ensuring you have a quality, certified testing lab at your side guarantees that you can respond in a timely manner to the demands—both expected and unexpected—of the development and production processes. Having provided the materials community with over 16 years of support, Ebatco is your choice to streamline your development process and to alleviate your production pressure. In addition to possessing an ISO/IEC 17025:2017 accreditation for its lab services, Ebatco consistently strives to improve the quality of its contract lab service offerings through the acquisition of advanced instruments, newly issued testing standards, and novel analysis methods.

There are five main technical disciplines in which Ebatco is prepared to serve the semiconductor and microelectronics community: mechanical, chemical, surface/interface, thermal, and particle and liquid discipline. The following provides you with an illustration of the types of work we can do to address common analytical and testing needs of the semiconductor and microelectronics community.

There are five distinct suites of analyses, characterizations and tests that are available at Ebatco for the five technical disciplines: mechanical analysis, chemical analysis, surface/interface analysis, thermal analysis, and particle and liquid analysis. These analysis suites and associated techniques are briefly explained below and are further exemplified in Table 1:

As an ISO-certified nanoindentation lab, Ebatco's nanomechanical analysis offerings are uniquely situated to deliver top-quality and high-resolution nanoscale characterization of ultrathin films and coatings, providing you with information regarding material hardness, elastic modulus, fracture toughness, film interfacial adhesion and other mechanical properties.

The chemical analysis suite offers powerful and rapid analysis techniques covering surface contamination, microstructure and elemental compositions, chemical and molecular imaging, unknown identification, degree of crystallinity, and a host of other characterization techniques.

The surface/interface analysis suite provides you with information pertinent to surface roughness, surface defects, surface charging, liquid contact angles, hydrophobicity, coating adhesion, pore size, and friction and wear.

The thermal analysis suite provides customers with varied data such as glass transition temperature, stability of materials, melting temperature, latent heat, contamination, and coefficient of thermal expansion.

The particle and liquid analysis suite delivers particle size distribution, particle zeta potential, liquid viscosity, and other properties that are vital to examining particle runoff, evaluating sanitation protocols, contamination, and failure analysis.

Table 1. Examples of Ebatco Analysis and Testing Suites

Lab Service Suite	Techniques
Mechanical Analysis	Nano/micro indentation, nano/micro scratch, Nano/micro compression, XPM, UTM
Chemical Analysis	FTIR microscopy, Raman microscopy, ICP-OES, SEM/EDS, XRD
Particle & Liquid Analysis	Particle/surface zeta potential, rheology, particle sizing, particle counting, laser diffraction, pore size, viscosity, refractive index, density, dielectric constant
Thermal Analysis	DMA, TMA, STA, DSC, mDSC, TGA
Surface/Interface Analysis	Optical profilometry, SEM, AFM, EFM, MFM, Microcontact angle, XRR, surface/interfacial tension, friction and wear

Mechanical Analysis

The mechanical properties of semiconductors are important for both performance and durability. Mechanical testing allows for the examination of properties that influence the durability of a thin film semiconductor during device fabrication. The properties determined by mechanical testing can further ensure performance of devices produced from a semiconductor wafer by confirming that the wafer has no quality issues and matches the intended specifications.

Case Study #1

Nanoindentation is a technique frequently used to determine the nanohardness and reduced elastic modulus of thin film semiconductors. Figure 1 shows a typical nanoindent made with a Berkovich indenter tip on a silicon wafer surface. Nanohardness and modulus can be used to assess how the film will react to stresses during the device fabrication processes such as chemical mechanical planarization (CMP). It is also known that integrated circuit (IC) devices have numerous layers of structures, and thermal expansion coefficient mismatch between layers can lead to significant thermal stresses during service. Excessive stresses can lead to cracking, warping, film dislocation, and delamination of the film layers. Nanoindentation helps to assess IC resistance to these stresses during usage.

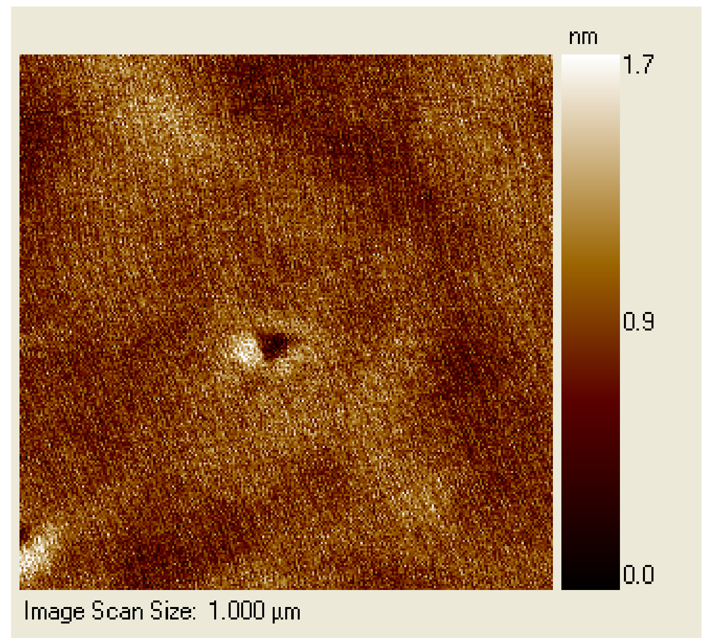

Figure 1. A nanoindent on a silicon wafer made with a Berkovich tip under a 30 µN load.

Case Study #2

XPM (Express Property Mapping) high-speed nanoindentation is a new technique used for 2D mapping the mechanical properties of a surface, such as the one displayed in Figure 2. XPM can be used on thin film semiconductors, ICs, or their wafer substrates to form a micron-spaced property map of the nanohardness or reduced elastic modulus across the surface. The map not only provides statistically significant results for individual phases, but also visualizes their high-resolution spatial distributions. It also offers unprecedented insight into nonuniformity in mechanical properties which may indicate defects/flaws that may or may not be visible on the surface. Identifying and characterizing these defects early can prevent problems during the device fabrication process or issues with inconsistencies in conductivity that can cause data transfer delays, processing errors, and decreased efficiencies.

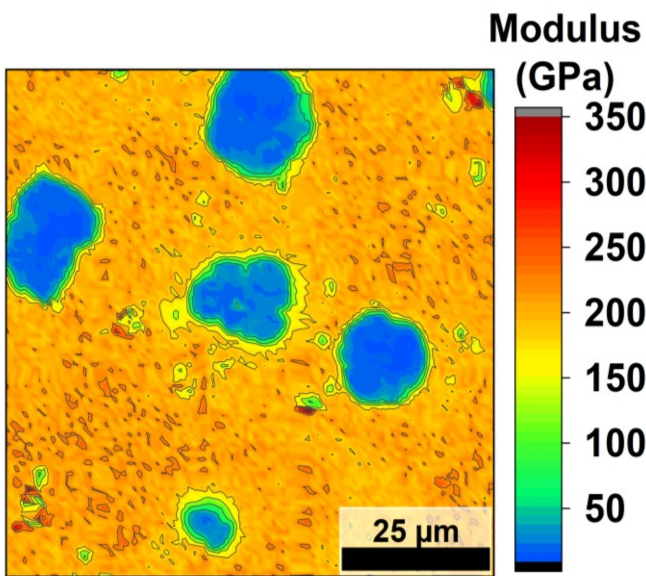

Figure 2. XPM map of cast iron, displaying the variation in reduced elastic modulus between the soft carbon nodules and the surrounding pearlite.

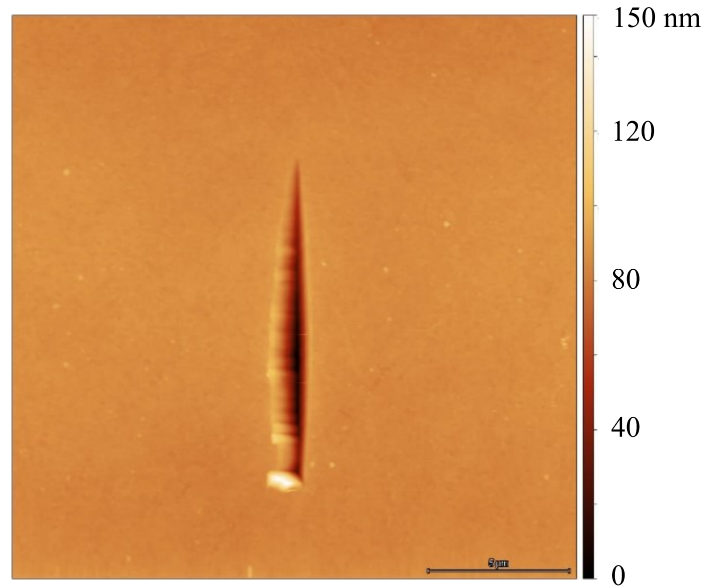

Figure 3. SPM image of a representative nanoscratch test on an ITO thin film semiconductor.

Case Study #3

Nanoscratch is used to probe the scratch resistance and interfacial adhesion of thin film semiconductors. A scratch is generated with either a constant or ramping force until the film begins to delaminate or endure a detectable scratch track. This allows for the determination of the force required to mechanically induce damage or delamination in the film. A representative scratch with film delamination can be seen in Figure 3.

The critical force of interfacial adhesion failure is primarily useful for determining how well a semiconductor film layer adheres to its underlying layer and its resistance to delamination. Stresses placed on a film during or after device fabrication may cause the film to delaminate if the adhesion between the film and its substrate or underlayer is not sufficiently strong. Nanoscratch is a widely accepted method for film interfacial adhesion strength evaluation. Nanoscratch can also be used for the determination of surface scratch resistance or understanding on the material removal efficacy of CMP process.

Chemical Analysis

Chemical analysis allows for the determination of composition in semiconductors. This kind of analysis is useful for quality control, as it can ensure that the overall composition of a film is correct and that the film is chemically uniform across the wafer surface. Ebatco has several chemical analysis tools to meet different application needs. Non-destructive techniques, like FTIR (Fourier Transform Infra-Red), Raman spectroscopy, and EDS (Energy Dispersive X-ray Spectroscopy) can evaluate uniformity, identify contaminants, and investigate fracture surfaces. ICP-OES (Inductively Coupled Plasma Optical Emission Spectroscopy) can identify dopants or trace elements at concentrations >10 ppb. With XRD (X-ray Diffraction), crystallographic phase identification\quantification, impurity phase concentration, heavy element concentrations, lattice strain, residual stress, and nanoparticle sizing can be determined.

Case Study #4

FTIR and Raman spectroscopy are vibrational spectroscopy methods that allow for the analysis of chemical and structural characteristics in thin film semiconductors. Figure 4 illustrates this capability, showing an image of a contaminated surface and IR data on each region. FTIR microscopy enhanced by the new focal plane array (FPA) detecting technique can collect 2D compositional data of IR-active compounds resolved across the surface of a thin film semiconductor with micron spatial resolution and fast speed. This can be used to assess the consistency of the film composition and chemical molecular distribution or to identify contaminants.

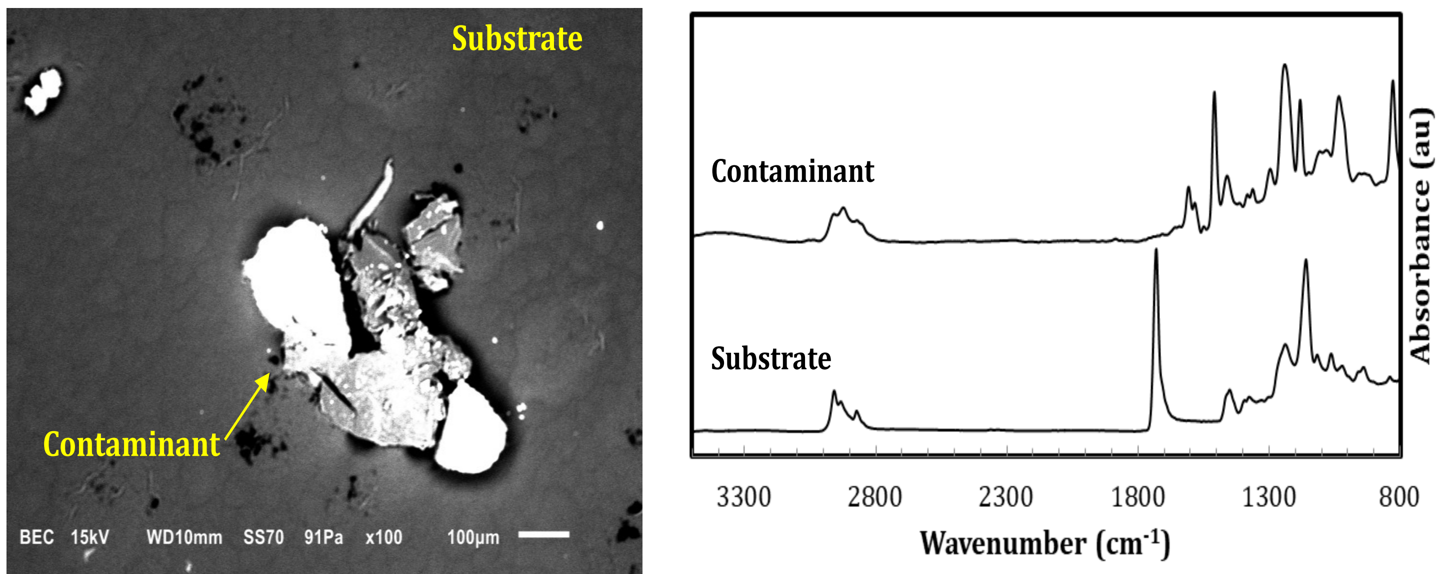

Figure 4. SEM image of a contaminant on a black carbon tape substrate (left). IR data for the contaminant and the substrate (right).

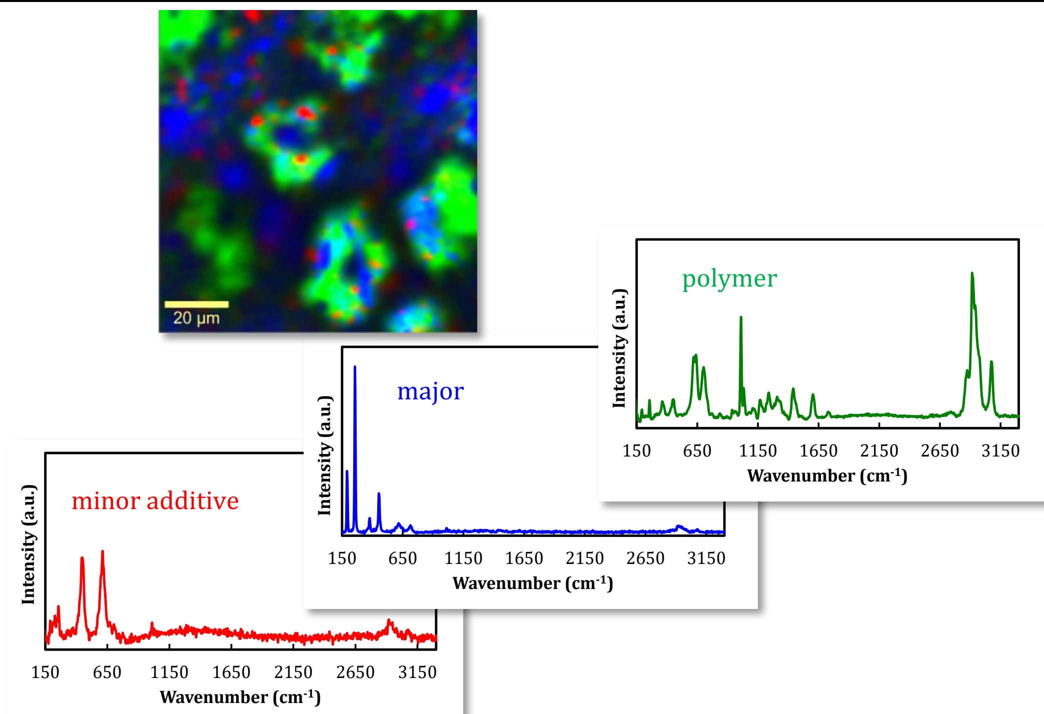

Figure 5. 2D Raman map of a polymer composite material (top) and color-coded spectra for each of the three compounds present in the composite (bottom).

Case Study #5

Confocal Raman spectroscopy has gained popularity in recent years for semiconductor analytical applications. It has functions similar to FTIR microscopy for molecular-level chemical analysis and 2D compositional mapping. Figure 5 shows a 2D map of a polymer composite with the Raman spectra corresponding to each of the three phases in the map. Because of its confocal microscopy setup, confocal Raman microscopy can allow for depth scanning to obtain data on subsequent layers of a sample. Figure 6 shows a depth scan of a layered polymer specimen with the Raman spectra of each polymer layer. When combining the 2D XY mapping with the Z Axis scanning capability of the confocal Raman microscopy 3D chemical imaging is possible, which is certain to provide useful 3D chemical information on semiconductor materials and devices!

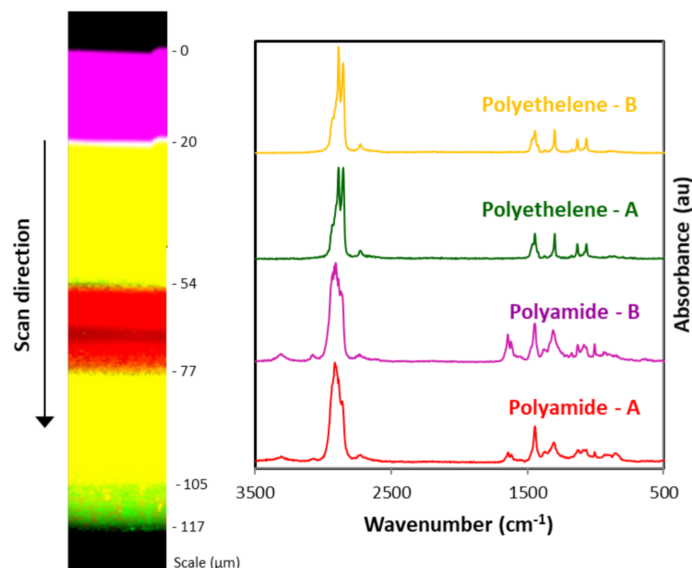

Figure 6. Depth scan of a layered polymer material (left) and Raman spectra for each of the polymers (right).

Case Study #6

ICP-OES enables simultaneous detection and quantification of numerous elements with high precision and sensitivity, offering a comprehensive assessment of an elemental composition in complex matrices. Table 2 demonstrates the effectiveness of ICP-OES when detecting trace metals in various commercial deodorant brands. Aluminum concentrations < 0.1 ppm were detected in "aluminum-free" deodorant brands #1 and #2.

Semiconductor fabrication requires precise and accurate control of dopant concentrations to deliver optimized electronic properties in each component or layer. To determine processing effectiveness or to diagnose conductivity inconsistencies, ICP-OES offers a reliable route to finding highly accurate dopant concentrations. Silicon wafer fabrication also relies on extremely high levels of cleanliness, often utilizing ultrapure water (UPW) for cleaning processes, cutting, and polishing. Trace element analysis can verify the quality of your UPW, ensuring that washing away residual ions doesn't leave new, unwanted ions behind.

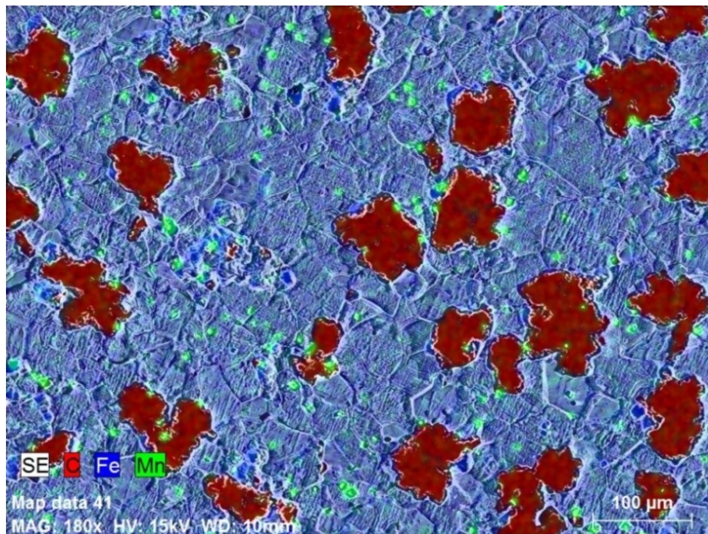

Figure 7. EDS elemental hypermap overlaid with an SEM image of a malleable cast iron surface. The distributions of three elements: C, Fe, and Mn are shown in red, blue, and green, respectively.

Table 2. Elemental Concentrations of Commercial Deodorant Samples

Sample / Element	Deodorant #1 (ppm)	Deodorant #2 (ppm)	Deodorant #3 (ppm)
Aluminum (394.4 nm)*	0.02	0.04	122.63
Copper (324.7 nm)*	0.00	0.00	0.00
Iron (239.5 nm)*	0.03	0.02	0.11
Manganese (257.6 nm)*	0.01	0.00	0.00
Nickel (231.6 nm)*	0.00	0.00	0.00
Zinc (213.8 nm)*	0.02	0.01	0.06

Case Study #7

EDS is equipped within Ebatco's SEM instrument, identifying and quantifying elemental concentrations by measuring characteristic X-rays produced as the SEM's electron beam interacts with the sample material. In addition to SEM imaging observations, the EDS system can further assist when analyzing micro areas of interest, like fracture surfaces, scratches, pits, and surface defects.

Figure 7 demonstrates the effectiveness of elemental mapping when identifying regions of interest that may or may not be visible in optical or SEM imaging. The red regions are graphite aggregates that were formed after casting by carbide decomposition during an annealing heat treatment. Manganese inclusions are identified by the scattered green regions; these features would be difficult to detect without EDS.

In addition to fracture and surface defect observations, voltage control of the incident electron beam can provide depth control for subsurface composition data. High or low voltages can be chosen to include elements ~1 μm deep or to limit the detection to the near surface layers, respectively.

Case Study #8

The primary use of XRD is determining the phases present in a sample. Each family of lattice planes within a crystalline phase produces an XRD spectral peak; lattice plane spacing determines the peak position, while chemical composition and crystal orientation influence peak intensity.

XRD also allows for the determination of crystallite size. In a polycrystalline material, the width of the peaks can be used to calculate the average grain size for that phase. Figure 8 presents XRD scans analyzed for phase identification of alumina powder with varying crystallite sizes. Peak broadening can be clearly observed for crystallites < 100 nm in diameter. The peak shifting and absence of certain peaks in the 50 nm sample indicates a size-dependent phase transition from α- to γ-phase alumina. In addition, XRD can investigate the lattice parameters and stable crystallographic phases in thin films that are heavily influenced by the underlying substrate; these substrate-dependent factors can drastically influence electrical and mechanical properties that are critical to semiconductor operation.

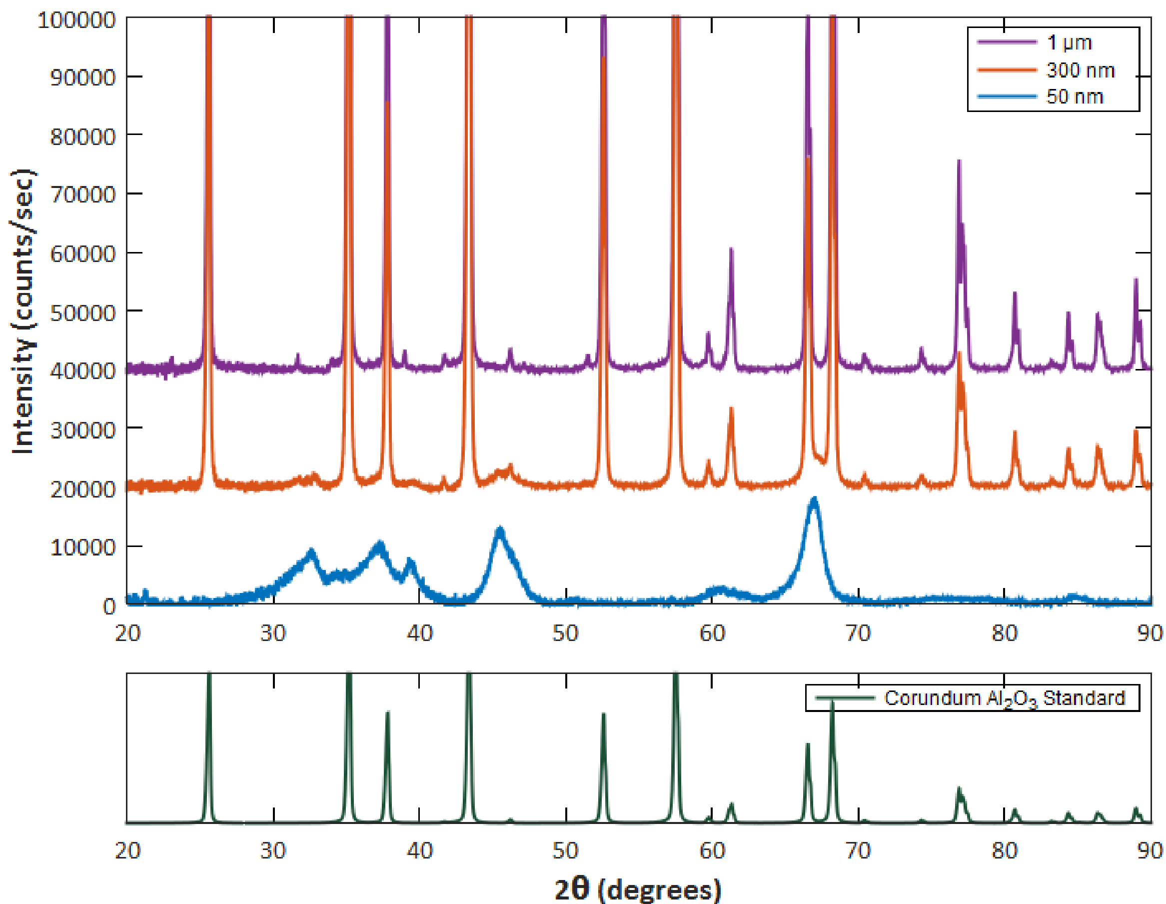

Figure 8. XRD scans of alumina powder of 50 nm, 300 nm, and 1 μm crystallite sizes (top) along with the literature results for aluminum oxide (bottom).

Particles and Liquids

Ebatco offers an array of methods to obtain precise particle sizes, to describe aggregation behavior, and to characterize solution or slurry flow properties. The ability to measure both particle and surface Zeta potentials can help to optimize dispersion behavior related to Chemical Mechanical Planarization (CMP) slurries and polishing pads. Rheology is used to describe flow properties at different shear rates, which can help to create slurry formulations or to ensure ideal spin coating behavior of a photoresist solution.

Case Study #9

Quantifying Zeta potential as a function of pH can provide crucial information related to particle aggregation in solution or on a surface. Figure 9 shows an example of this measurement on bleached and unbleached paintbrush fibers. Particles in slurries should maintain a Zeta potential > 30 mV absolute value to avoid aggregation. Zeta potential data can inform which slurry formulation will have only primary particles of a desired size to avoid a polishing slurry that would cause undesirable scratches and blemishes.

The measurement of particle size along with particle Zeta potential measurement allows for an even more complete characterization of a slurry suspension. Particle sizes are expected to reach their minimum at Zeta potential >30 mV absolute value as agglomeration of particles is significantly slowed down. Additionally, the measured particle size distributions and Zeta potential data can be compared with the data provided by a supplier to ensure quality.

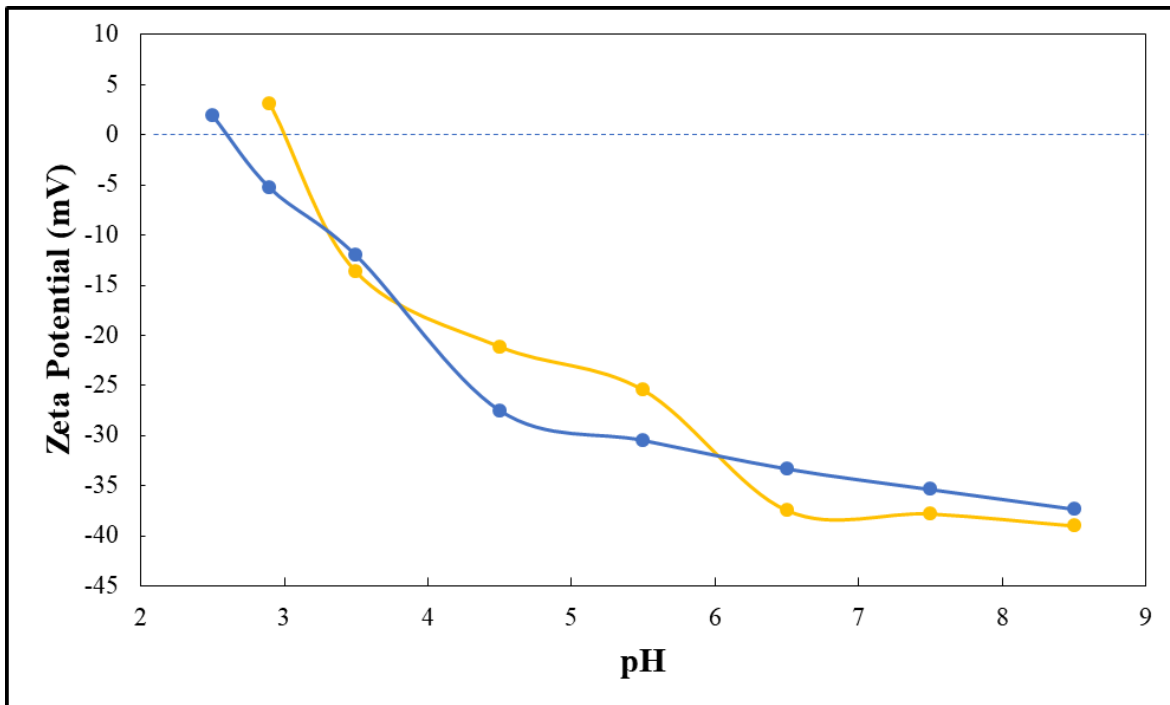

Figure 9. pH-dependent Zeta Potential measurement of untreated paintbrush fibers (yellow) and bleached paintbrush fibers (blue).

Case Study #10

Newtonian fluids, like air and water, have a constant viscosity with increasing shear rate (spin speed between parallel plates), but more complex fluids and solutions can exhibit a range of different behaviors. Well-dispersed polishing suspensions, like those shown in Figure 10, will show a linear shear thinning behavior. When a force is applied, the decrease in viscosity allows the particles to flow and mix homogeneously, and when the mixing stops, viscosity increases and prevents particles from settling quickly. Aggregated suspensions will show non-linear behavior as additional forces break apart clusters of particles. Rheology measurements on polishing suspensions can help to ensure uniform surfaces after each polishing stage.

Rheological measurements can also be useful for photoresist applications. The photoresist solutions used for lithography are often coated onto a surface using spin coating. Maintaining a desired thickness while spreading to coat uniformly requires optimized viscosity and flow properties. Further, temperature-dependent rheology studies can help to understand how lamp heat and/or nearby exothermic curing reactions can influence photoresist curing processes.

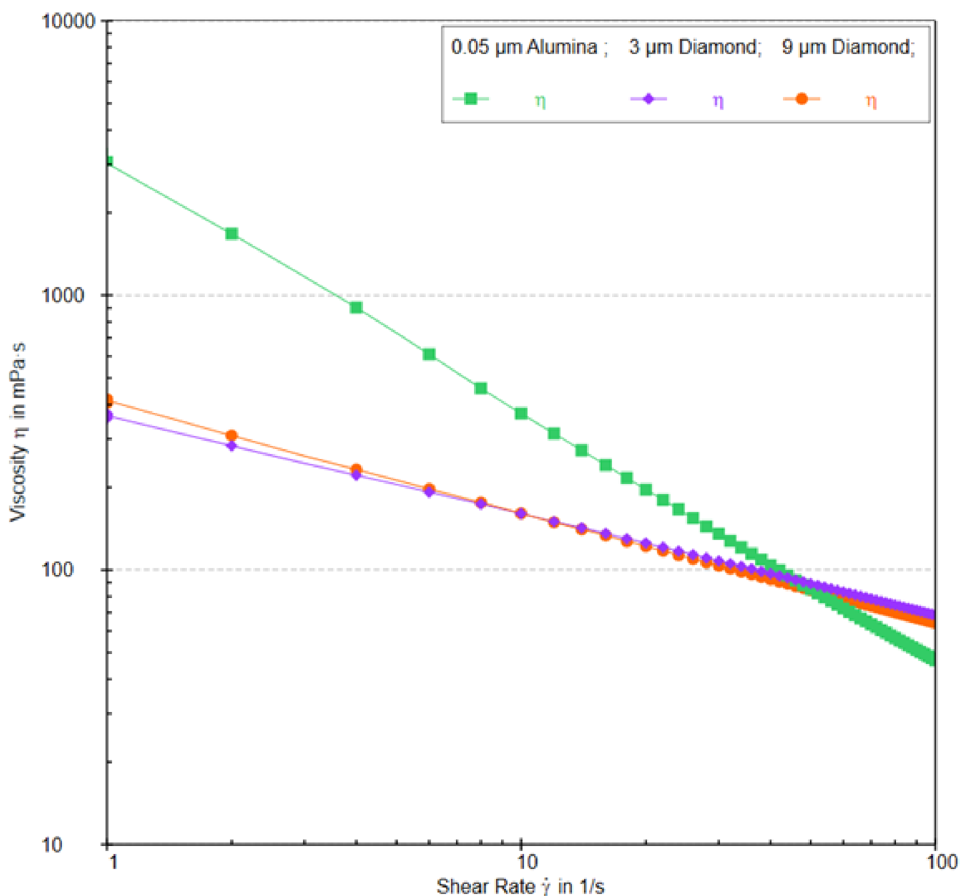

Figure 10. Shear thinning flow behavior in commercial grade polishing suspensions.

Case Study #11

Ebatco's suite of particle sizing instrumentation offers an excellent degree of accuracy, resolution and reproducibility for particle sizes as low as 0.6 nm and for solids loadings between 0.001 – 40 vol%, as well as dry powders. The accuracy and resolution of dynamic light scattering for particle sizing is demonstrated in Figure 11. Size distribution plots such as this can be used to easily identify whether a desirable, normal distribution of particle sizes has been achieved, which can deliver ultrasmooth surfaces when as a polishing slurry. The reproducibility of these measurements is represented by the standard deviation of repeated tests, shown in Table 3.

Particle sizing and size distribution measurements, combined with zeta potential and rheology, will provide the information necessary to ensure the quality and effectiveness of polishing solids, liquids, and of final slurry formulations. These factors are key to producing ultrasmooth surfaces reliably and with minimal defects.

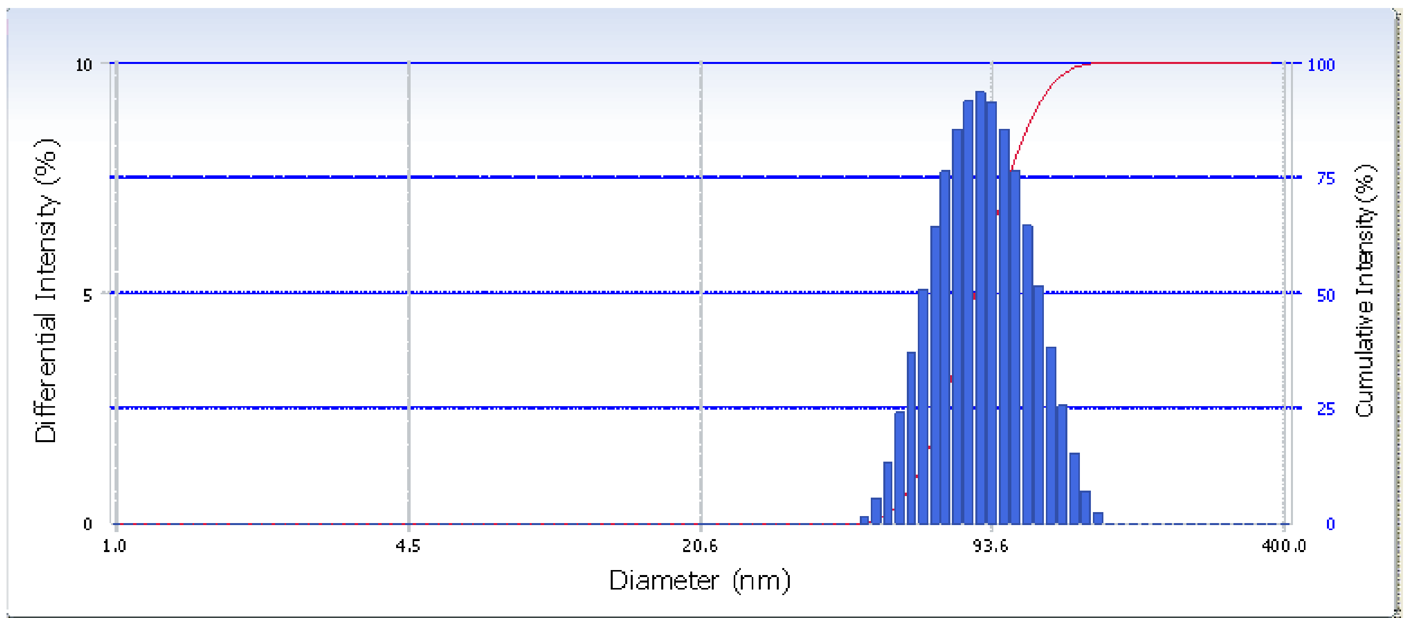

Figure 11. Size distribution of silica nanoparticles in suspension.

Table 3. Average particle sizes and standard deviation between tests for two commercial polishing slurries

Specimen	Diamond in Water (nm)	Diluted Silica (nm)
Test 1	4630.2	128.5
Test 2	4836.1	131.0
Test 3	4885.9	129.0
Average	4784.1	129.5
Standard Deviation	135.6	1.3

Thermal Analysis

Thermal analysis is crucial for any material or system that will encounter temperature changes or thermodynamic events during fabrication, transport, or operation. Whether it is glass transition characterization for dielectrics or packing materials, coefficients of thermal expansion to limit thin film interfacial strain mismatch, or melting temperatures for soldering joints, Ebatco has the instrumentation and expertise to meet your needs.

Case Study #12

In addition to first-order phase transitions such as melting and vaporization, there are second-order transitions that occur in certain materials. One of these second-order transitions is the glass transition exhibited by amorphous materials. When amorphous materials are cooled below their glass transition temperatures, they become hard and brittle, like glass; when the amorphous materials are heated above their glass transition temperatures, they are in the rubbery state, where they are softer and more flexible. Dynamic Mechanical Analysis (DMA) measures viscoelastic properties of a material, determining storage modulus, loss modulus and tangent delta values at different temperatures, as shown in Figure 12. These values not only provide information on the material elastic and viscous behaviors, but they can also help to find glass transition temperatures.

Applications of DMA in semiconductor and microelectronic materials could include measurements of changes in the mechanical properties of packaging materials due to operational or environmental temperature shifts. Besides, dielectrics for high temperature applications require a manufacturer to consider glass transition temperatures due to notable changes in Young's modulus, dielectric permittivity, and dielectric dissipation factor associated with glass transition of the dielectrics.

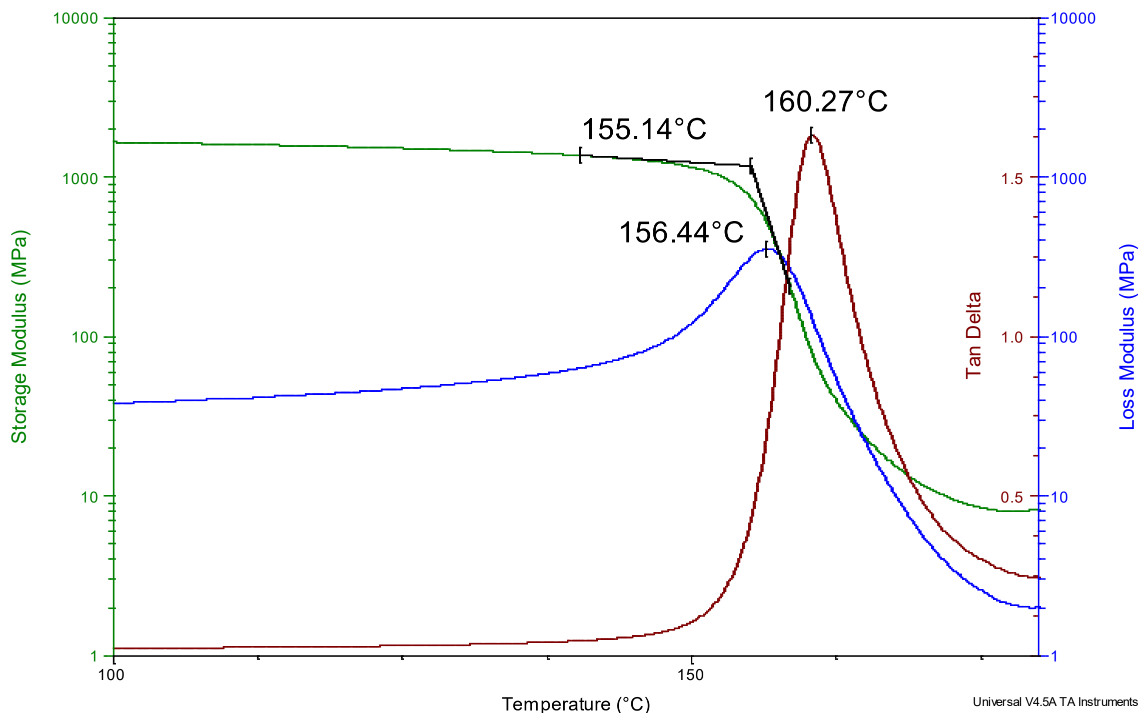

Figure 12. Dynamic mechanical analysis for the glass transition of polycarbonate.

Case Study #13

Simultaneous Thermal Analysis (STA) combines the capabilities of Thermogravimetric Analysis (TGA) with Differential Scanning Calorimetry (DSC). TGA measures very small mass changes with high accuracies, which is relevant to finding decomposition (outgassing) temperatures of potential epoxy packaging materials. DSC identifies temperature-dependent exothermic and endothermic reactions, which can be related to decomposition, epoxy curing onset and time, and degree of cross-linking in epoxies, glass transitions in epoxies and dielectrics, phase transformations in crystalline solids, and melting temperatures for soldering materials. Figure 13 shows STA decomposition data with both TGA (green) and DSC (blue) contributions.

Combining STA with other techniques in the thermal analysis suite can help to provide a complete picture of events like decompositions and glass transitions that can affect mechanical properties and strains. These events are important for semiconductor packaging or for semiconductor stability and performances.

Figure 13. STA Data for the Decomposition of Calcium Oxalate Monohydrate.

Case Study #14

Thermal expansion and thermal expansion mismatch at interfaces are crucial for layered materials, as temperature changes can result in warping, cracking and delamination at mismatched interfaces. Thermomechanical Analysis (TMA) is used to quantify a material's coefficient of thermal expansion (CTE) in a controlled gas and temperature environment. With a displacement resolution of 0.5 nm between -150 ºC and 1000 ºC, highly accurate values can be obtained for your area of interest. While CTE is often represented by a single number, indicating linear expansion behavior, nonlinear or complex expansion/contraction behavior is observed for some compositions, as shown in Figure 14. As excessive thermal expansion, complex expansion, and expansion mismatch for delicate IC components can lead to premature failure or performance degradation. CTE should be understood, determined and monitored.

Figure 14. Thermal expansion of silicon nitride rod.

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Surface Analysis

Surface analysis techniques can identify nanoscale features that may impact the properties of semiconductors. Ebatco's vast surface and interfacial testing and analysis capabilities can provide precise imaging of surface structures, defects, and features, can detect single layer contamination, and can measure sub-nanometer surface roughnesses.

Case Study #15

The ability to identify surface defects is crucial for semiconductor production, as even minor surface defects can impact the performance of integrated circuits and microelectronics. Defects such as pits, inclusions, impurities and scratches greatly increase the surface roughness of the affected semiconductors and act as sites for processes such as boundary scattering to occur, decreasing conductivity and performance.

Optical profilometry serves as a technique for identifying these defects at any stage of the fabrication process. Optical profilometry can take significantly less time in imaging surfaces of samples, relative to other surface mapping instruments. With other techniques, triangulation or line scanning is done as opposed to area-based measurements. Optical profilometry is advantageous because it needs very little to no sample preparation, making measurements performed on the profilometer very fast and simple. Figure 15 shows an optical profile of a coated wafer with pitting and other surface defects.

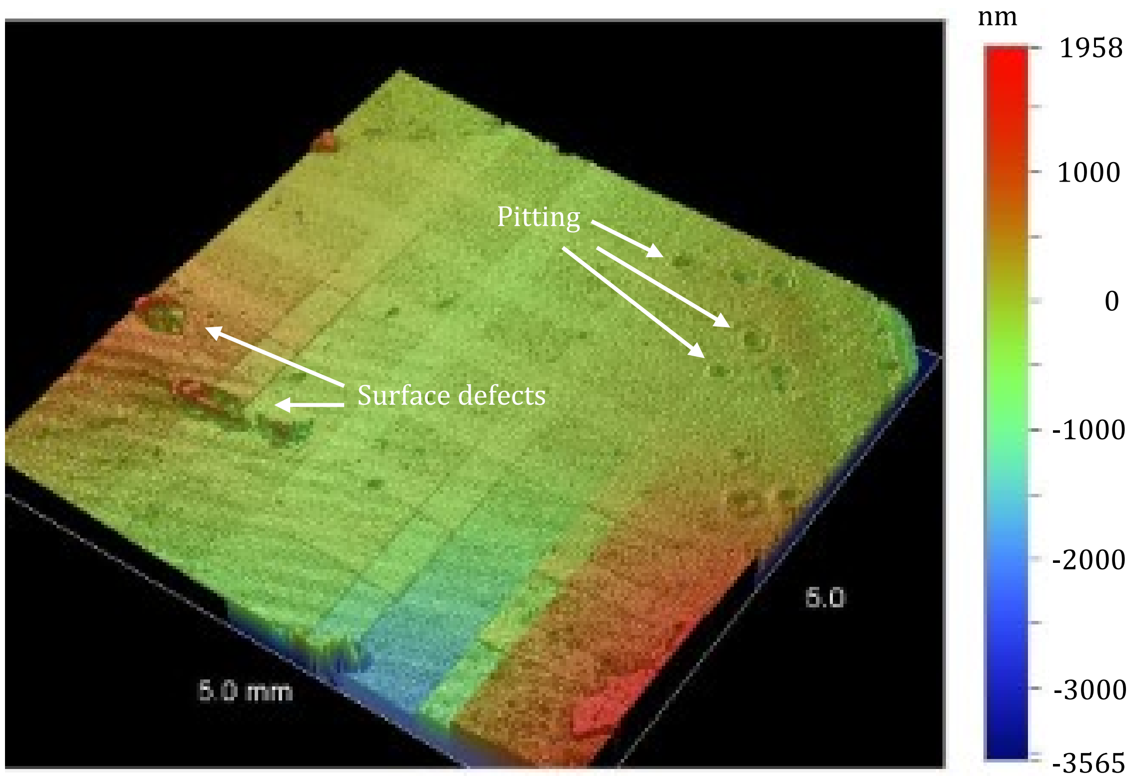

Figure 15. 3D optical profile of a coated wafer sample with pitting and other surface defects.

Case Study #16

Complimentary to optical profilometry, SEM (Scanning Electron Microscopy) allows for precise imaging of defects at the nanometer level. Relative to optical imaging, the higher magnification and increased depth of field from SEM can provide more insights into small features, like scratches and microchip cross-sections, and into complex topologies, like fracture surfaces. For example, Figure 16 displays an SEM image of a soldering contact between a microcontroller and a circuit board.

This technique is particularly useful in the semiconductor industry for quality assurance, helping identify small defects that have led to a failure, or that might lead to poor performance. Additionally, SEM can be used to help determine the potential origin of a defect by closely examining the morphology. Further, combining backscatter electron detection and secondary electron detection can provide insights into compositional and topological variations, respectively, across a sample.

SEM is also useful in the early phases of semiconductor development, as it can be used to examine the surface morphology of freshly deposited semiconductor films to help verify layer thickness, uniformity, and quality. Analysis of these images not only helps to target certain final morphologies, but can help to determine how nucleation and growth proceeded during deposition.

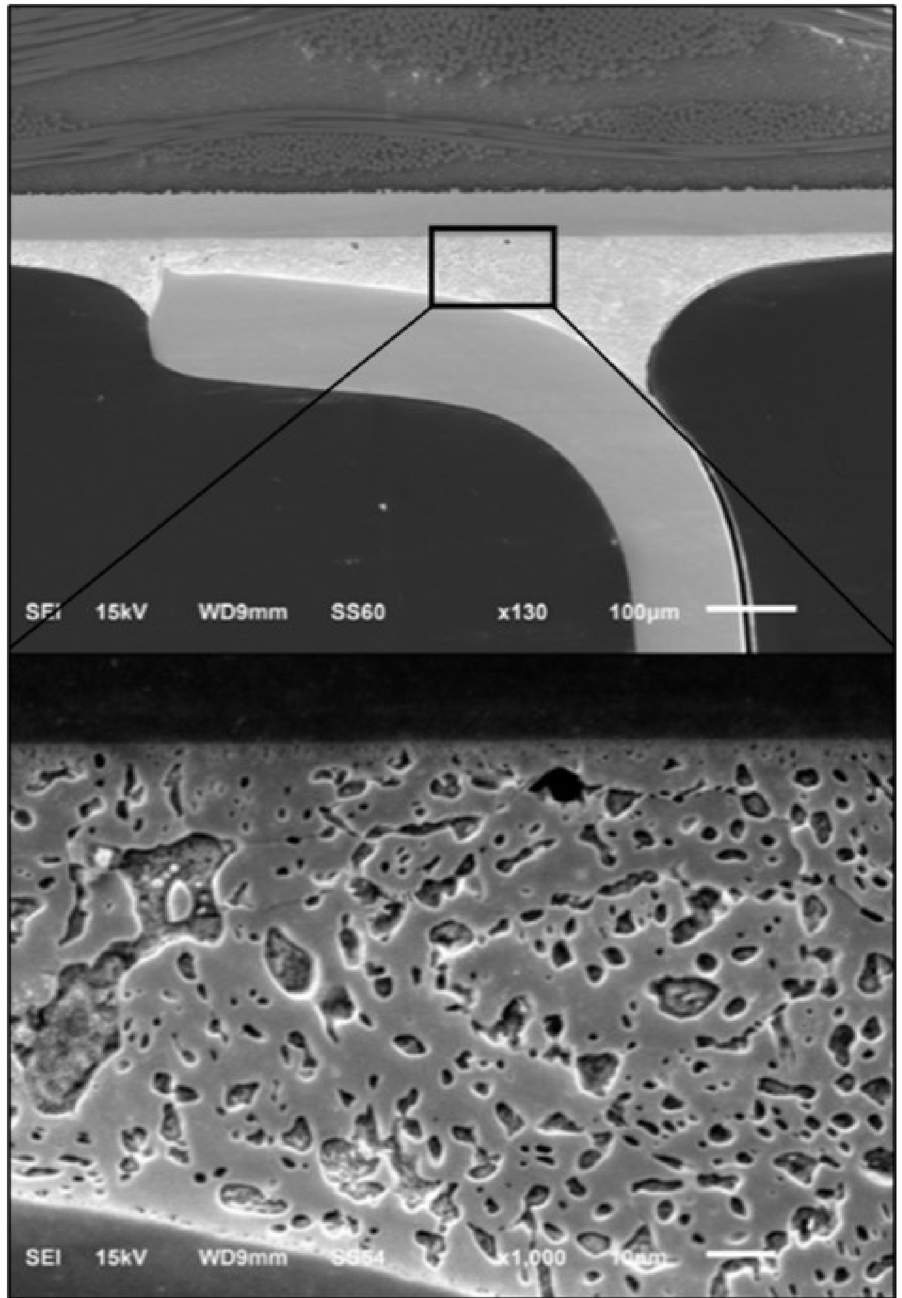

Figure 16. SEM image of a soldering contact between a microchip lead and circuit board. Higher magnification (bottom) shows micro- and macrovoids, and Kirkendall voids within the solder joint.

Case Study #17

AFM (Atomic Force Microscopy) allows for the accurate assessment of the surface roughness on semiconductor wafers, both before and after coating. Figure 17 shows an AFM image of an ITO thin film semiconductor with roughness values included for demonstration.

Surface roughness is an important property for wafers due to its effect on the mechanical, optical, and electronic properties of a semiconductor thin film. Mechanically, surface roughness may influence the durability of a coated wafer. The roughness of the wafer substrate affects the strength of the adherence between the film and the substrate, with rougher substrates decreasing the risk of delamination through increased mechanical interlocking of film and substrate surface. On the other hand, the roughness of a film surface affects the amount of friction the film is subjected to during the device fabrication process.
Electronically, surface roughness especially impacts ultra-thin films through boundary scattering effects, reducing the conductivity and performance of semiconductor thin films. Additionally, boundary scattering reduces the photovoltaic response of thin films for those being used in optical applications.

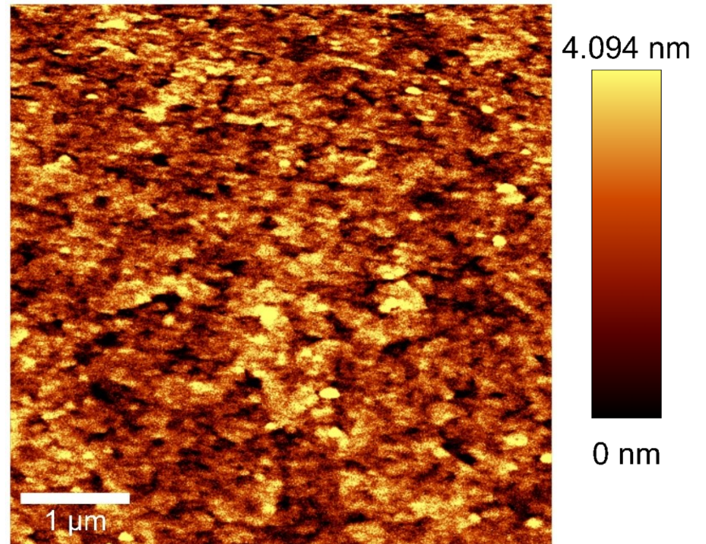

Figure 17. AFM image of an ITO thin film semiconductor. The sample had an RMS roughness of 1.19 nm (Sq) and an average roughness of 0.92 nm (Sa).

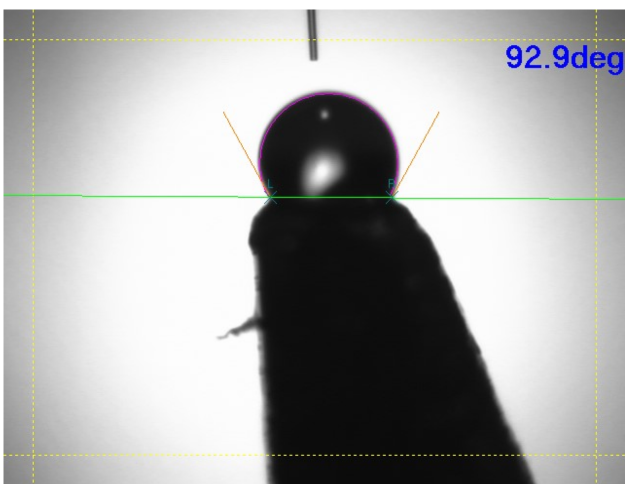

Figure 18. An image of a small droplet contacting a 140 µm hydroxyapatite coated Ti post.

Case Study #18

Micro contact angle measures the wettability of a surface at the micron scale. A representative micro contact angle measurement can be seen in Figure 18, displaying a micrometer scale droplet on a coated metal sample.

These micro contact angle measurements help to better understand the properties of a wafer surface with or without micron-sized patterns, in addition to wettability. They are useful in determining proper cleaning practices, analyzing potential single-molecular-layer contamination, determining a potential coating's adherence, and calculating the surface free energy of a semiconductor thin film. Micro contact angle tests are not limited to using only water as a testing liquid, any fluid that may be relevant to the semiconductor process can be used and studied.

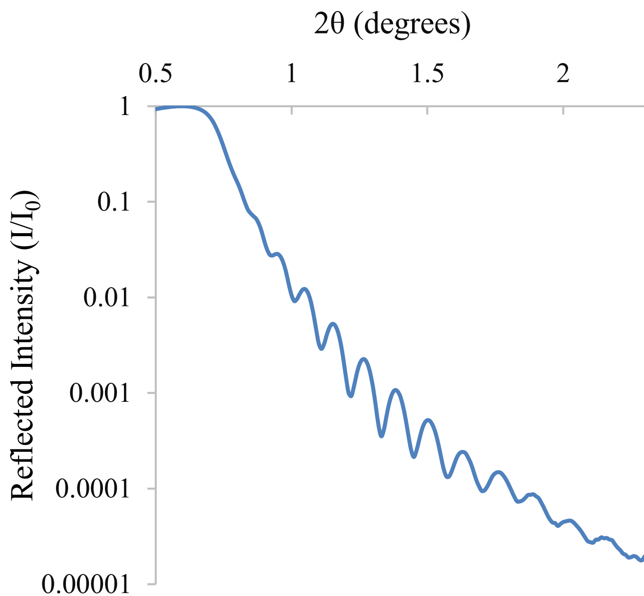

Figure 19. XRR scan of a 65 nm ITO thin film on a glass substrate. ITO thickness was determined to be 63.9 nm based on the scan.

Case Study #19

XRR (X-Ray Reflectivity) is a technique that allows for the determination of film thickness, film density, and surface/interface roughness. As an example, Figure 19 illustrates an XRR scan to determine the thickness of an ITO thin film that is often used in optoelectronics.

Many semiconductors show vastly different properties based on thickness due to changes in band structure. XRR can be used to ensure that the thickness of a thin film semiconductor layer is correct for the desired properties, even in complex, multi-layer structures, as XRR is capable of penetrating hundreds of nanometers into a surface. Surface roughness can influence interfacial adhesion, charge transfer between layers, transparency/optics, and surface reactivity. Surface roughness determination through XRR will be convenient and useful. Moreover, layer density verification via XRR can act as a quick quality check after film depositions, to ensure the formation of fully dense components.

Concluding Remarks

The representative data and case studies in this document are intended to summarize a portion of Ebatco's capabilities that we believe are most relevant to the semiconductor and microelectronics industry. This document covers only a subset of analyses and techniques available to you, a full list of Ebatco technical consulting and contract lab services can be found at **https://www.ebatco.com**; and additional inquiries are welcome via email, at **info@ebatco.com**, or by phone, at **+1 (952) 941-2199**. Our PhD level scientists and experienced staff are ready to assist you with your materials and device characterizations at any time when you have a need. They can advise and recommend techniques and protocols suitable to meet your specific testing and analytical needs. They can also assist you with data interpretation and failure analysis.