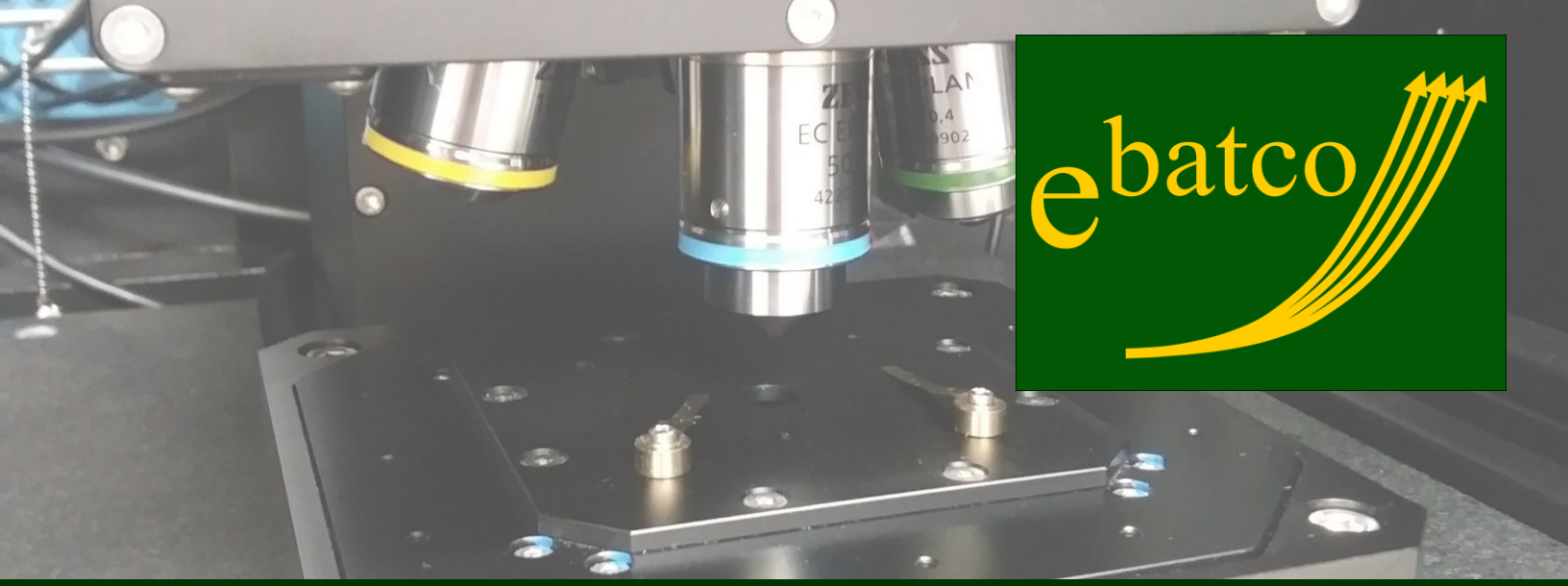

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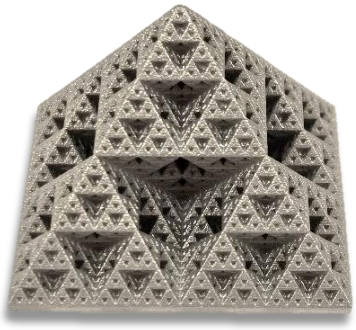

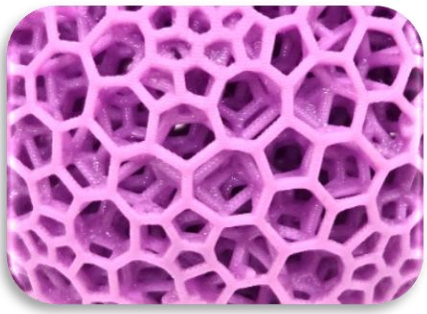

Additive Manufacturing:

Characterization, Testing, and Analysis from Raw Materials to Finished Goods

Summary

This white paper focuses on the characterization, testing, and analysis that are critical to the development and implementation of additive manufactured (AM) products from raw material inspection, qualification of finished goods specifications, verifications, and failure analysis. AM provides a route to rapidly and autonomously fabricate complex parts, and the choice of infill design can enable macroscale material properties that might be otherwise impossible or prohibitively expensive. Polymer, metal, ceramic, and composite AM processes also bring the respective properties that are unique to each material class, ensuring that products can have the ideal properties for a given application. The implementation of AM for each material class, however, can be drastically different, and the techniques that are needed to develop processes and control product quality can be similarly variable. To meet these challenges and help assess raw materials, processes, and final products, Ebatco can provide the testing solutions you need for any process or product using our expansive instrumentation suites and experienced personnel.

Introduction

Additive manufacturing 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 18 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 AM community: mechanical, thermal, particle and liquid, surface/interface, and chemical analysis suites. The following provides an illustration of the types of work we can offer to address common analytical and testing needs for AM products. These suites, along with relevant techniques, are correlated to the different AM material classes in Table 1.

1. Ebatco's *mechanical analysis suite* offerings are uniquely situated to deliver top-quality and high-resolution mechanical properties for individual layers, infill patterns, and final products, in addition to nanoscale characterization of ultrathin films, coatings, and interfaces using our ISO-certified nanoindentation lab. This suite will provide you with information regarding material hardness, elastic modulus, fracture toughness, film interfacial adhesion and other mechanical properties.
2. *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.
3. *The particle and liquid analysis suite* delivers particle size distribution, particle zeta potential, liquid viscosity, and other properties that are vital to examining flowability, uniformity, suspension stability, contamination, and failure analysis.
4. *The surface/interface analysis suite* provides information pertinent to surface roughness, surface charging, liquid contact angles, hydrophobicity, coating/interface adhesion, pore size, and friction and wear.
5. *The chemical analysis suite* offers powerful and rapid analysis techniques covering surface contamination, microstructure and elemental compositions, chemical imaging, unknown material identification, degree of crystallinity, and a host of other characterization techniques.

Table 1. Brief summary of Ebatco's material analysis suites and the associated techniques that are relevant to additive manufacturing. A technique's relevance to an additive manufacturing material class is indicated by a **o**.

Mechanical | Thermal | Particle and Liquid | Surface and Interface | Chemical

Property	Technique	Polymers AM	Metals AM	Ceramics AM	Composites AM
Strength, Modulus	UTM, DMA, Nanoindentation	o	o	o	o
Fatigue, Creep	UTM, DMA, Nanoindentation	o	o	o	o
Hardness	Nanoindentation, Microindentation	o	o	o	o
Decarburization Depth	Nanoindentation, SEM		o		
Degradation	Friction and Wear	o	o	o	o
Thermal Expansion	TMA	o	o	o	o
Glass Transition, Phase Transition	STA, DSC, DMA, TMA	o	o	o	o
Enthalpy of Fusion	DSC	o	o		o
Decomposition	STA, DSC, TGA	o		o	o
Particle Size	SEM, DLS, Coulter, Laser Diffraction, Light Obscuration, Optical Microscopy	o	o	o	o
Particle Shape, Morpohology, Porosity	SEM, Optical Microscopy	o	o	o	o
Zeta Potential	ELS, Streaming Potential	o		o	o
Flow Properties	Rheometer	o		o	
Surface Topography	AFM, Optical Profilometry	o	o	o	o
Coating Thickness	SEM, XRR, Scratch	o	o	o	o
Layer & Coating Adhesion Strength	Scratch, Nanoscratch, Peel Strength, Stud Pull, Tape Pull	o	o	o	o
Friction & Wear	Friction, Abrasion	o	o	o	o
Crystal Structure, Crystallinity	XRD	o	o	o	o
Residual Stress	XRD		o		
Polymer & Molecule Identification	FTIR, Raman	o		o	o
Element Identification	EDS, ICP-OES	o	o	o	o

Mechanical Analysis

The mechanical properties of additive manufactured parts are often a primary area of interest for manufacturers and consumers. The processing needed to 3D print a new product can drastically modify the materials properties, for better or for worse, relative to a bulk material that is molded or machined to shape. Additionally, the layer design, from the layer height, print orientation, infill pattern, extrusion temperature and rate, can influence the specific strength and directional properties of a product. Identifying the directionality of materials properties like stiffness and strength can be critical to ensuring that a single particular infill pattern, or a repeating collection of patterns, behaves as the design intends.

Case Study #1: Universal Testing Machine (UTM)

The UTM is a versatile instrument that is capable in a range of mechanical testing modes, including tension, compression, shear, and bending. Mechanical testing is critical to ensure a product and its components will survive the stresses they experience in an intended application. While mechanical properties for common materials are well documented and are easily accessible, there are a variety of factors that can affect the real-world performance of a material. Processing routes and material history, with factors like starting materials, heat treatment, extrusion parameters, layer dimensions, and technique can all heavily influence mechanical properties.

Figure 1 shows three-point bending test results from two identical, 3D printed bars made with a carbon filled PET/Nylon blend. One bar was tested with the load perpendicular to the layering direction while the second bar was rotated 90° and tested so the load was parallel to the layering direction. This change in orientation resulted in a moderate change in yield stress (69.59 vs 55.57 MPa), but there was a very significant change in the elastic modulus (4.68 vs 2.64 GPa). This change in modulus, or stiffness, can be easily visualized by the difference in slope for the linear segment at the start of each curve. Additive manufactured materials are well known for having anisotropic material properties as a result of the layer-by-layer deposition method. Anisotropic properties can be used to great effect when a part is loaded in the correct orientation, but understanding the loading limits in different orientations is an important step to proper implementation.

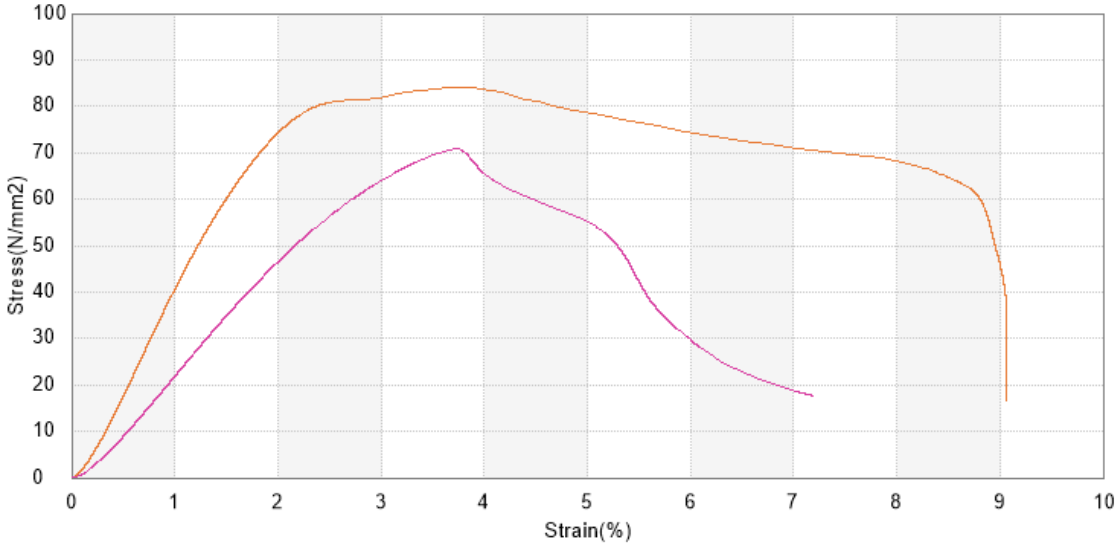

Figure 1. Three-point bend test results for 3D printed parts in the directions parallel and perpendicular to the layer-by-layer deposition direction.

Case Study #2: Nanoindentation

Nanoindentation is an excellent tool to determine the nanohardness and elastic modulus for materials with small dimensions and small volumes (films, powders, wires, single grains) or nonuniform surfaces and cross-sections. With a force resolution below 1 nN and displacement resolution of 0.2 nm, nanoindentation testing allows for highly accurate and repeatable results, even at the nanoscale. Additional capabilities, like nanoscratch for interfacial adhesion, property mapping on nonuniform surfaces, nanoDMA, and *in-situ* SPM imaging make this instrument versatile and excellent for investigating mechanical properties at the micro and nanoscale.

Figure 2 demonstrates how Accelerated Property Mapping (XPM) functions in the Bruker Hysitron TI 980 to spatially resolve the properties of a laser powder bed fusion (PBF) additive manufactured product. From the energy dispersive X-ray spectroscopy (EDS) composition map on the left, there are clearly Fe-rich and Co-rich regions that result from the incomplete fusion of the starting powders. Ni and Cr are also present, but they are more well distributed throughout. The XPM mechanical property maps, seen in the middle and right images of Figure 2, were acquired in a single test with 10,800 total indents. While cobalt and iron have very similar elastic moduli (typically 190 - 210 GPa), cobalt typically has a higher hardness than iron for similar alloys. From the images in Figure 2, higher cobalt concentrations from EDS clearly correlate to higher hardness regions from XPM. These results demonstrate how mapping with nanoindentation can resolve subtle changes in mechanical properties that might arise from compositional differences, phase separation, coating nonuniformity, or underlying porosity.

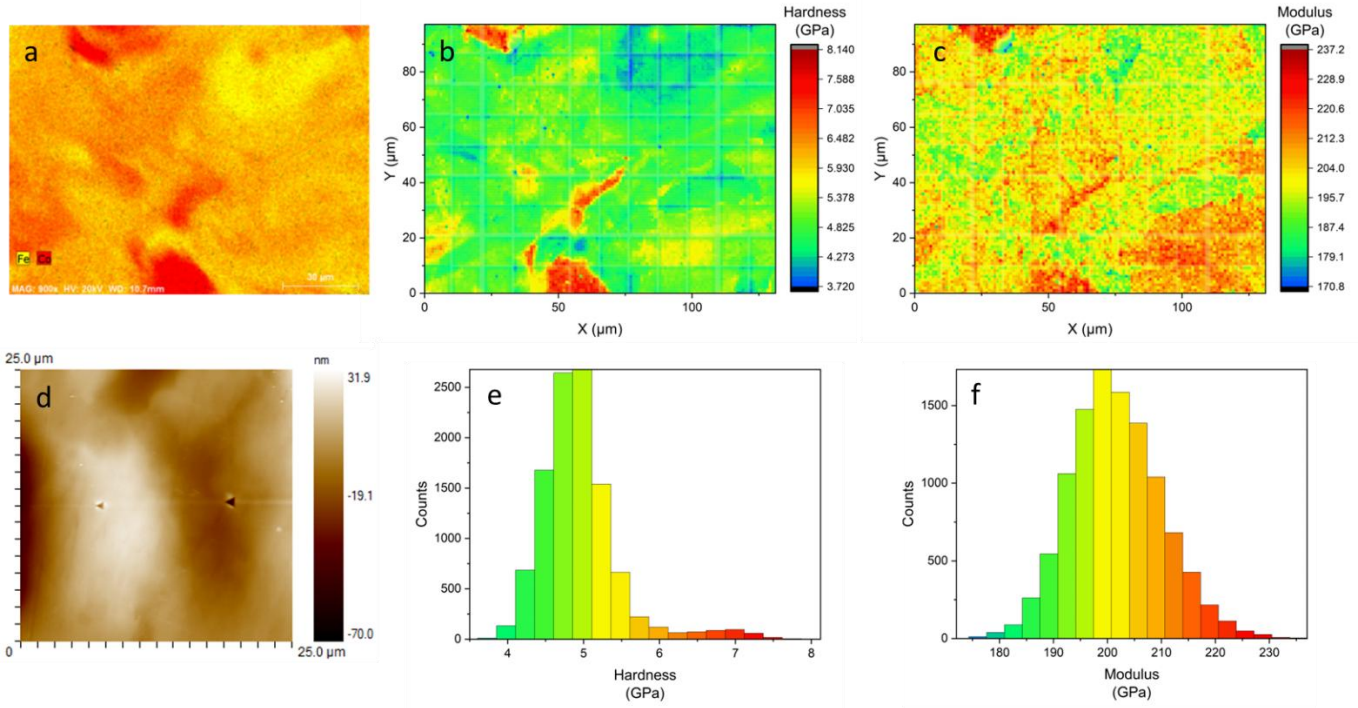

Figure 2. EDS composition map (a) compared with a XPM hardness map (b) and a XPM elastic modulus map (c), SPM image of intents on Co-rich (small indent, left) and Fe-rich (large indent, right) regions (d), nanohardenss histogram (e), and elastic modulus histograms (f) for a product manufactured via laser PBF.

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, degree of cross-linking, melting temperatures, axis-resolved coefficients of thermal expansion, or decomposition behavior, Ebatco has the instrumentation and expertise to meet your needs. Additive manufacturing (AM) methods generally rely on heat or energy input to create a product. Whether it's softening or melting a polymer filament, fusing metal powder, sintering ceramics, or burning out organic additives, understanding the thermal properties of printing materials is essential for designing and optimizing AM processes

Case Study #3: Thermomechanical Analysis (TMA)

TMA offers accurate characterization of thermal expansion in materials, and the tests can be performed in tension for thin films and fibers, or in compression for bulk materials. One side of a sample is fixed, while a probe makes contact with the opposite side that is free to move, and dimension changes are measured under a controlled temperature and gas environment. While the expansion of many materials will be isotropic, layered structures like 3D printed parts will tend to have anisotropic expansion. This anisotropic expansion can arise from regions of the sample that are constrained vs regions that are free to expand, or areas that have preferred orientations and differing bonding characteristics.

Figure 3 demonstrates how the coefficient of thermal expansion (CTE) can differ among the different axes of a 3D printed part. From the dimension change curve on the left, broad glass transition peaks are observed from about 75 – 140° C with nearly linear segments on either side. These linear segments can be used to determine the CTE in the solid state (pre-transition) and in the rubbery state (post-transition). Before the glass transition, Axis 1 and Axis 2 have a CTE that is about 6 times greater than the CTE for Axis 3. After the glass transition, Axis 1 and Axis 2 have a CTE about 11 times greater than that of Axis 3. For these measurements, Axis 3 corresponds to the primary tool path direction, or the direction parallel to the deposited lines of material. Preferred orientation of polymeric molecules and filler materials, as well as weaker bonding between layers of deposited material along Axis 1 and Axis 2 are probable reasons for the significant differences in CTE. These differences underscore the importance of characterizing CTE in 3D printed parts, especially when they will be exposed to variable temperature conditions.

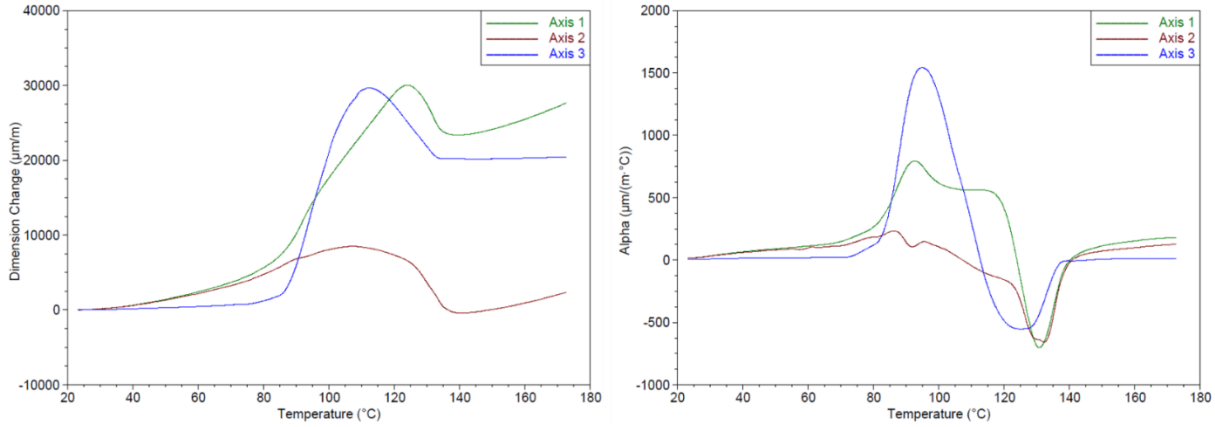

Figure 3. TMA measurements on a carbon-filled PET/Nylon blend 3D printed part showing dimension changes (left) and the first derivative of dimension change (right).

Case Study #4: Simultaneous Thermal Analysis (STA)

STA is a technique that combines thermogravimetric analysis (TGA) with differential scanning calorimetry (DSC). TGA is used to determine the mass change of a sample over time as it is heated, which is useful for characterizing decomposition reactions, moisture content, volatile components, oxidation, and more. DSC measures the change in heat flow in a sample, relative to a reference, under varying temperatures to reveal thermal events like melting and solidification, glass transitions, phase transformations, chemical reactions, and decomposition. Combining these techniques is invaluable in certain applications, such as differentiating between phase transformation and decomposition, or recognizing pyrolysis, oxidation and combustion reactions.

Figure 4 shows STA results for a 17-4PH powder intended for additive manufacturing, which has been heated through its melt and to 1550 °C. The TGA results show steady mass gain from about 200 °C to 1100 °C, and the DSC results show a series of convoluted exothermic reactions over the same range. Mass gain with an accompanied exotherm often indicates oxidation in metals, although additional reactions with organic additives are also likely over this temperature range. Knowledge of the starting and final chemistries, along with the mass change and heat flow data from STA, can all be used to assign intermediate reaction steps throughout the heating process. When DSC is performed in conjunction with a standard reference material, like sapphire, specific heat (C_p) can also be calculated, as seen from the results in Figure 4. Knowing the temperature-dependent values for C_p will allow calculations of the power needed to change the powder's temperature to a specific value. Additionally, knowing the total heat required to melt a metal powder is especially useful for laser fusion additive manufacturing routes, so laser power can be tuned in an efficient manner.

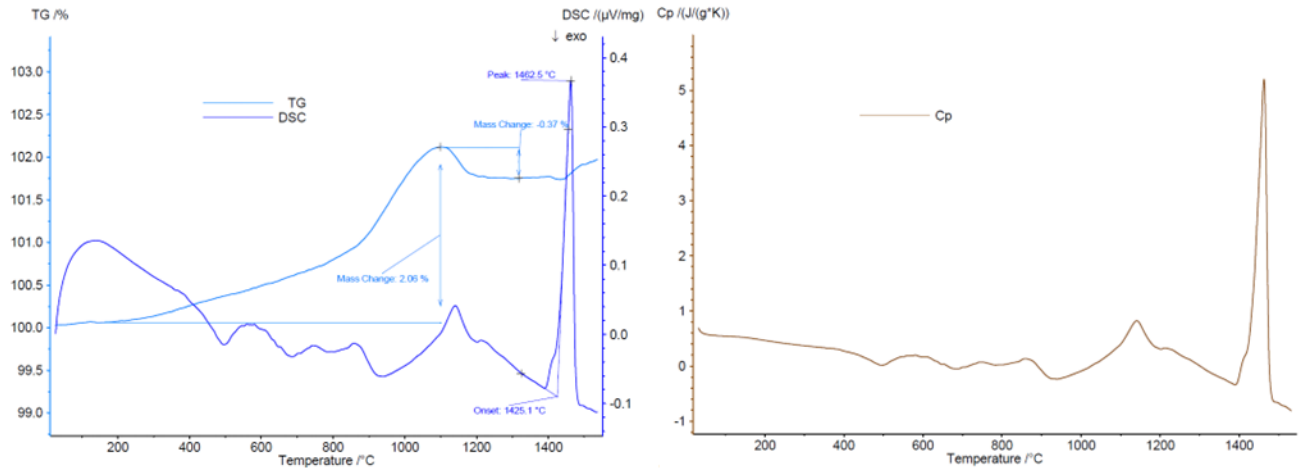

Figure 4. STA data (left) and specific heat data (right) for 17-4PH additive manufacturing powder.

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. Sizing and size distributions for particles between 0.1 nm and 2000 µm can be achieved. This sizing capability, and the ability to measure both particle and surface Zeta potentials can help to optimize dispersion and flow behavior that is critical to reliable material delivery during AM fabrication. Rheology is often used to describe flow properties at different shear rates, which can help to create slurry formulations and to identify ideal flow or extrusion rates during printing. Temperature-controlled tests can also help to optimize melt flow behavior for thermoplastic materials and blends.

Case Study #5: Particle Sizing

Particle sizing is a critical factor for optimizing flow and densification behavior in additive manufacturing processes that rely on powder and slurry supply feeds. Metal AM processes, like Powder Bed Fusion and Directed Energy Deposition, can utilize dry powders, and characterizing these powders without forming a suspension first provides data that most closely relate to the real-world implementation. The Beckman Coulter LS 13 320 is capable of measuring particles suspended in a liquid medium or in a dry powder form by using specially designed sample modules. The Tornado Dry Powder System (DPS) module can measure powders from 0.4 µm to 2 mm in a dry environment.

Commercially available 17-4PH steel powder was investigated for particle size distributions using the DPS module, and the results are provided in Figure 5. The manufacturer specifications state a particle size range from 0 – 25 µm, which appears to be accurate based on the size distribution based on number percentage. Viewing the same sizing data based on surface area and volume percentages, however, reveals the existence of larger sized particles. Particles with larger sizes will contribute more to the overall surface area and volume of the powder, compared with smaller particles, making their presence more noticeable in these plots than the plot for number density. Particle size distributions influence packing density and flowability, making this an important design factor, especially for high performance and high-stakes parts, like those used in aerospace and infrastructure.

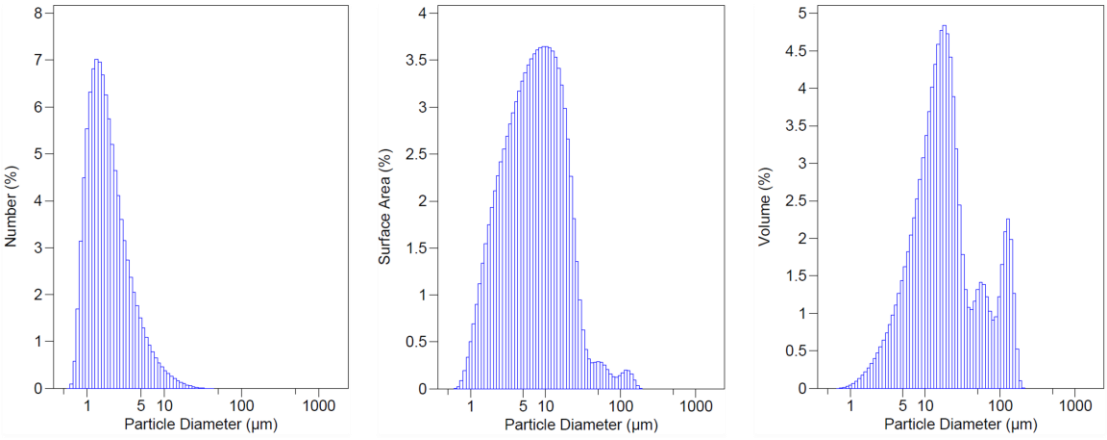

Figure 5. Particle size distributions based on differential volume, surface area and number for the 17-4PH powder for additive manufacturing.

Case Study #6: Zeta Potential

Quantifying Zeta potential as a function of pH can provide crucial information related to particle aggregation in solution or on a surface. Particles in slurries should maintain a Zeta potential > |30| mV to avoid aggregation. Zeta potential data can predict which slurry formulation will have only primary particles of a desired size and which will have larger aggregates. When optimizing the flow of a slurry, which is common for ceramic additive manufacturing processes, particles that tend to aggregate will result in unpredictable flow behavior. This, in turn, can cause uneven layer lines and upstream blockages.

Figure 6 demonstrates Zeta potential results for silica particles in suspension over a 3 – 12 pH range. These results show that the suspension should be stable at 8.0 pH and above, and the suspension will aggregate the most when the pH is equal to the isoelectric point, 5.69 pH. The measurement of particle size along with particle Zeta potential allows for an even more complete characterization of a slurry suspension. The measured particle size distributions and Zeta potential data can also be compared with the data provided by a supplier to ensure quality.

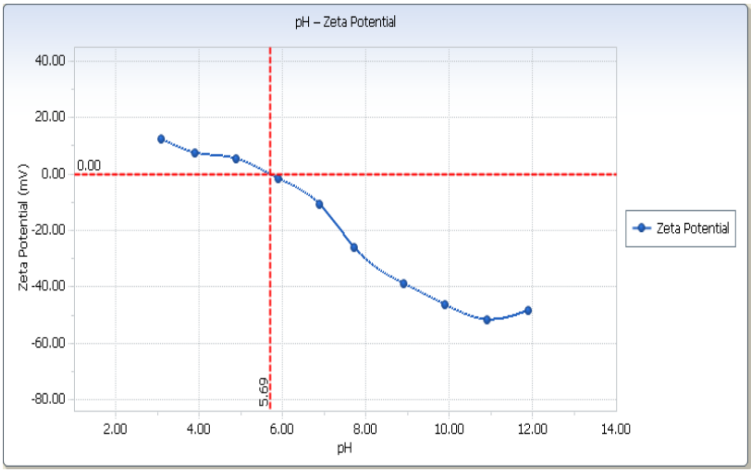

Figure 6. pH-dependent Zeta potential measurement of silica particles in suspension.

Case Study #7: Rheology

Rheology measurements are most often used to determine absolute viscosity under different shear rates to find ideal processing parameters, or to understand materials properties like thixotropy, yield stress, and aggregation behavior. Additionally, viscoelastic measurements can determine storage and loss modulus, which can be used to describe temperature-dependent flow characteristics like glass transitions and melting points and frequency- or amplitude-dependent properties like flow points, structural breakdown, and structural recovery.

For ceramic additive manufacturing, the fabrication process relies on a powder slurry to be reliably delivered through tubing and a dispensing nozzle, before being deposited in a layer that must be firm enough to hold its shape. Determining a slurry recipe with an optimized combination of flowability and rigidity is a delicate balance. The amounts of binders, plasticizers, powders, and liquid, along with the particle size, polymer molecular weight, and liquid viscosity will all influence flow properties. Figure 7 demonstrates how particle size can affect a slurry's viscosity at different shear rates. The linear power law relationship for all slurries indicates excellent particle dispersion, yet there is a notable difference in the flow behavior of the 50 nm Alumina sample. The increased viscosity at low shear rates indicates that this slurry is more likely to clog tubing and nozzles, especially if dispensing flow rates are inconsistent.

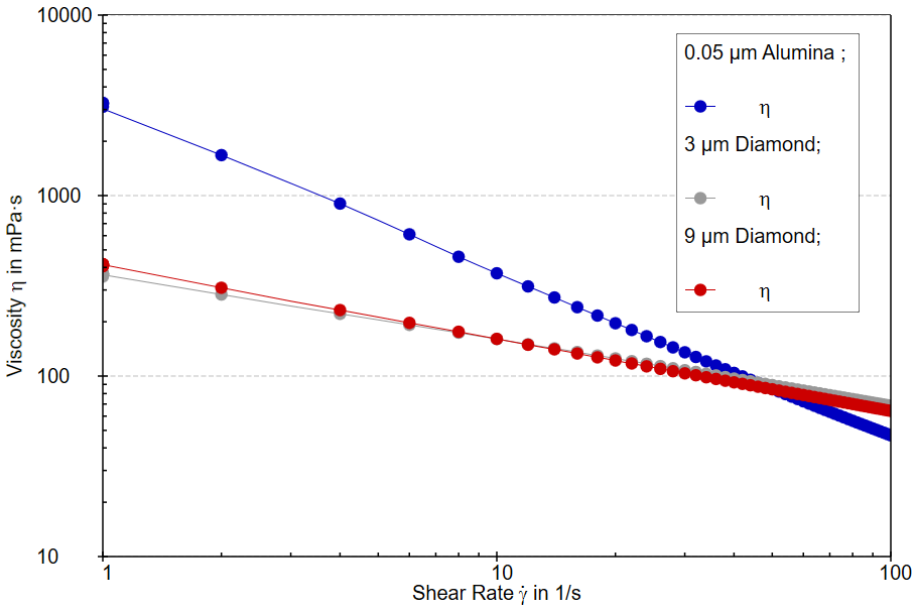

Figure 7. Viscosity vs. shear rate measurements demonstrating shear thinning behavior in particle suspensions.

Surface and Interface Analysis

Surface analysis techniques can identify nanoscale and microscale features that can influence compatibility, hydrophobicity, adhesion strength, and friction/wear. For AM products, the interfaces between layers, the orientation of the layers, and the layer thickness can be just as critical as the layers themselves, so proper materials properties and adherence at interfaces is necessary to ensure desired product performances. Ebatco's vast surface and interfacial testing and analysis capabilities can provide interfacial adhesion strength, sub-nanometer surface roughness, and precise imaging of surface structures, defects, and features.

Case Study #8: Optical Profilometry

When a sample has sufficient reflectivity, optical profilometry can generate height maps over millimeter-sized regions with 0.1 nm height resolution. The large area of analysis allows for the obtained roughness values to be more representative of an entire surface than other methods that are more limited in their analysis windows. Additionally, this method does not rely on a contact stylus rastering across a surface, so height maps are obtained very quickly and without the risks that come with physical contact (or near contact) with the surface.

Figure 8 displays optical profilometry height maps for two samples made with different metal additive manufacturing fabrication routes. For the two samples that were measured, the results indicate that the powder bed fusion route results in a surface that is less rough than its counterpart, which was fabricated using a metal binder jetting route. Surface roughness can have a significant influence on friction, surface interactions, optical properties, and more, making it an important aspect of materials characterization in many different fields. When optimizing an additive manufacturing process, optical profilometry can serve as a fast and accurate way to investigate how processing parameters can influence surface topography.

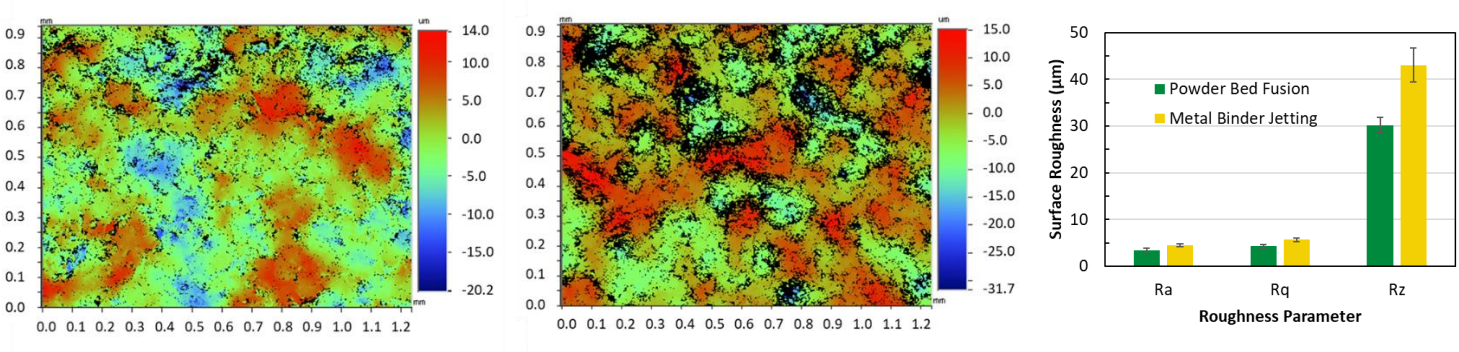

Figure 8. Optical profilometry height maps from samples made by powder bed fusion (left) and a metal binder jetting (middle). A summary of relevant roughness values is provided in the plot at the right.

Case Study #9: Contact/Microcontact Angle

Contact angle measurements provide an indicator of wettability and this technique has been widely adopted in industrial fields to evaluate surfaces. For a water droplet on a solid surface, a contact angle greater than 90° indicates a hydrophobic surface, and a contact angle less than 90° indicates a hydrophilic surface. Additionally, microcontact angle measurements utilize micron sized glass capillary, fine liquid dispensing control, and a high-speed, high-resolution CCD camera to analyze samples and surface features, like fibers and micro patterns, that are smaller than a conventional liquid droplet.

3D printed polymeric products are often built line by line, layer by layer, with each layer solidifying before the neighboring one is deposited. There are several possible roughness patterns at different faces, with top surfaces (0°), or the x-y face, having line-line interfaces, side surfaces (90°) along the z-direction having layer-layer interfaces, and surfaces between 0 and 90° having steps of varying size that achieve a particular shape. These different faces and step sizes result in significant differences in surface roughness at positions on a sample, which have a direct impact on surface wettability. Figure 9 demonstrates how increased surface roughness can cause a material to transition from having hydrophilic behavior to hydrophobic behavior. These measurements were performed using microcontact angle on thin 3D printed components that are less than 1 mm wide.

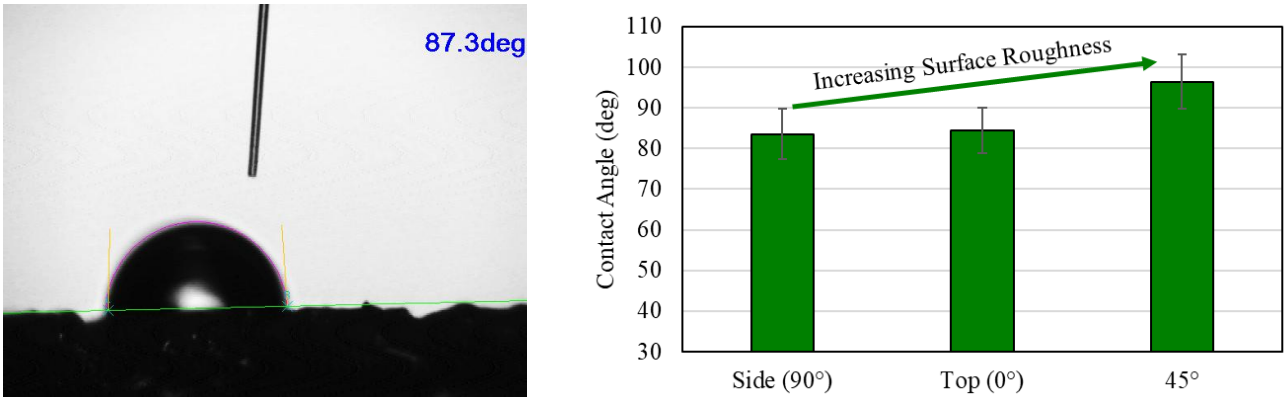

Figure 9. Representative microcontact angle measurement image (left) and results (right) of a 3D printed polymeric product, taken at positions on the sample where the test surface is 0°, 45°, and 90° to the layering planes.

Case Study #10: Porosity with SEM

Increasing porosity percentages can inhibit many material properties, like density, strength, thermal conductivity, electrical conductivity, and more. Additive manufactured products often suffer from higher porosities than their bulk counterparts, resulting from poor layer adhesion, filament quality, powder packing, or unoptimized fabrication parameters. The high contrast and resolution obtained from SEM images allows for pore size analysis using automated software that can detect pores and calculate individual pore statistics, pore size distributions, and total porosity.

Figure 10 shows an SEM image and the processed result showing color masks to indicate the detected and analyzed pores. This AM part built through powder bed fusion has significant porosity that can be easily seen as black shapes within the cross-section. From the image analysis, the results indicate the total porosity is 3.35 %, and the largest pore, identified by a purple mask, has a maximum Feret diameter of 271.37 µm. Information on total porosity can be used to predict changes to material properties, and maximum pore size is critical to understanding and predicting fracture strength.

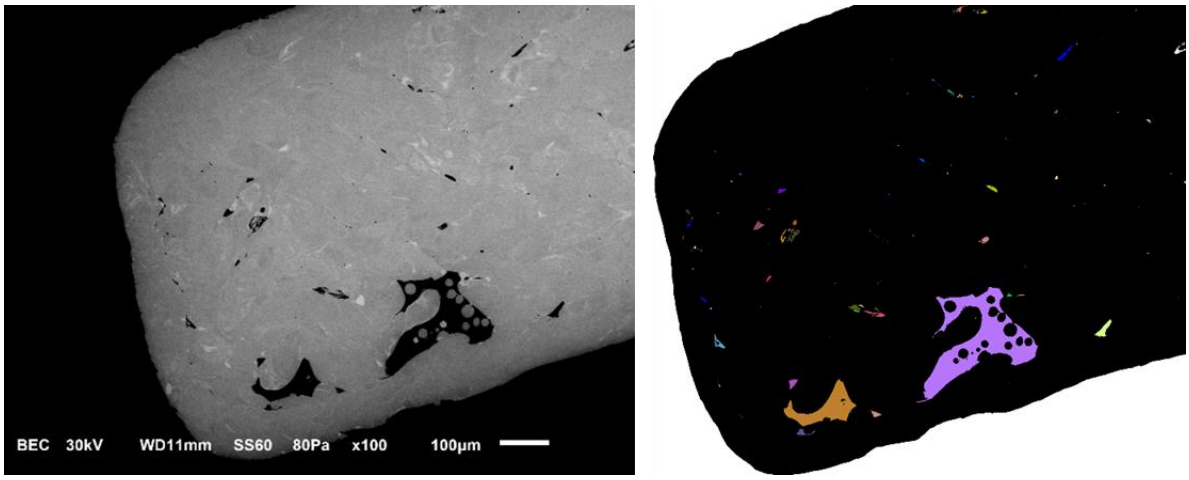

Figure 10. SEM image (left) and processed SEM image with color masks at pore locations (right).

Chemical Analysis

Chemical analysis is used to determine local or bulk concentrations of materials at any stage of a manufacturing process: supply/raw materials, intermediate materials, final products, end of life, and for failure analysis. This kind of analysis is useful for quality control, as it can ensure that the overall composition of a material is correct and that the composition is chemically uniform across a surface or interface. Ebatco has several chemical analysis tools to meet different application needs. Non-destructive techniques, like FTIR (Fourier Transform Infra-Red Spectroscopy), 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 #11: X-Ray Diffraction (XRD)

The primary use of XRD is determining the phases, or repeated atomic arrangements, that are present in a sample. A single composition can have a variety of stable phases, and each phase can exhibit significantly different properties. Each family of lattice planes within a crystalline phase produces a diffraction peak; lattice plane spacing determines the peak position, while chemical composition, crystal orientation, and the frequency of a plane's occurrence influence peak intensity.

For materials made of iron or steel, the thermal history of the sample will largely dictate the resulting phase, microstructure, and mechanical properties. To determine if a heat treatment has resulted in the desired phase or combination of phases, XRD analysis can provide the relative weight percent of phases within a material. Figure 11 shows an XRD pattern of a commercially available 17-4PH steel powder intended for additive manufacturing. Alpha (α) and Gamma (γ) steel phases are both present and quantified using Rietveld refinement. An amorphous phase is also present, seen as a broad peak at low 2θ, which reveals the presence of organic additives that are commonly used to assist with powder cohesion and flowability. Comparing patterns between starting material and final product will elucidate how the metal fusion process has influenced phase transitions, secondary phase formation, and organic burnout.

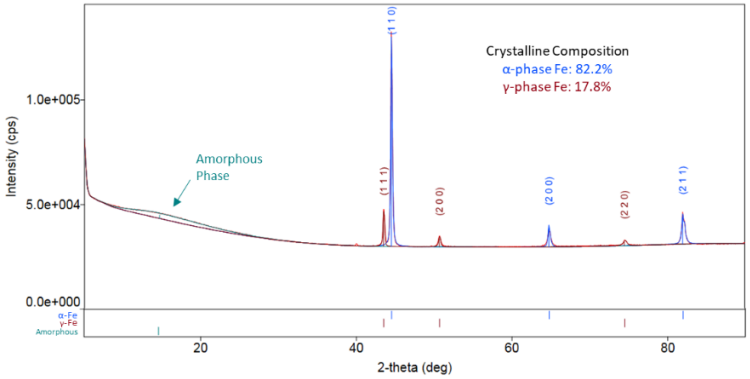

Figure 11. Quantitative phase analysis from an XRD scan on a 17-4PH powder sample. Experimental data are shown as a black line, and Rietveld refinement's individual phase fits are shown with blue, red and green lines.

Case Study #12: Confocal Raman Spectroscopy

Raman spectroscopy is a chemical analysis tool that is complimentary to FTIR since each technique is sensitive to different vibrational modes. While FTIR is the best choice for polymer compositions, Raman is suitable for both polymers and inorganic materials, like metal oxides. Confocal Raman instruments have the additional benefits of fine spatial (x,y) resolution and depth profiling (z) capabilities, allowing for 2D composition maps, film/layer thickness measurements, and 3D composition reconstructions.

Utilizing a polarizer, analyzer, and appropriate sample orientations can also provide information about the directional organization of molecules in a sample, which is well-suited to investigations into failure analysis, polymer extrusion, and degree of crystallinity. Polymer orientation can affect a range of material properties like strength, crystallization temperatures, glass transition temperatures, and wettability. Figure 12 demonstrates how polymer orientations can influence a Raman spectrum with varying angles between polarizer and analyzer. Absorption bands associated with polymer backbones (C-C stretching) are indicated by arrows and are polarization dependent, while bands associated with less ordered side chains (CH_3 stretching) are not polarization dependent. Extrusion-based AM methods are likely to see similar preferred orientation in polymer chains.

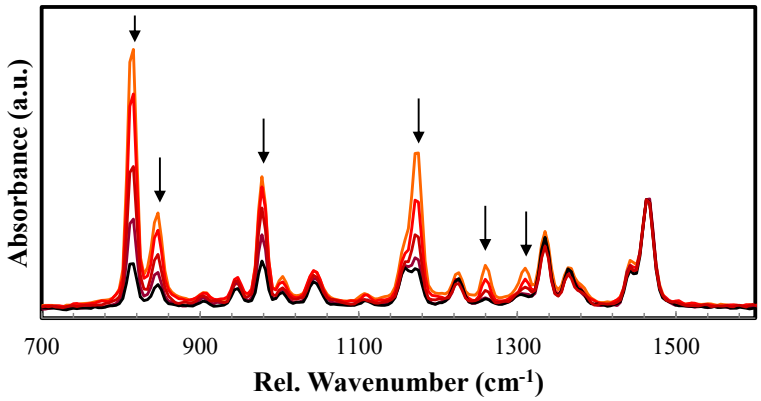

Figure 12. Polarized Raman spectra of transparent packaging tape. The angle between the incident laser light and the analyzer was set to 0° (parallel), 30°, 45°, 60°, and 90° indicated by the lines darkening from light orange to black, respectively. Arrows indicate the peak intensity shifts as the polarization analyzer was rotated.

Case Study #13: Fourier Transform Infrared Spectroscopy (FTIR)

Non-destructive, microscopic, and spatially-resolved chemical analysis capabilities make FTIR an excellent tool for quality control and evaluation of layered structures and coatings. FTIR measures the mid-IR absorption of various chemical bonds and translates them into a spectrum that can be used to identify a compound, making this an especially valuable tool when identifying polymer structures. The combination of a 32 x 32 Focal Plane Array (FPA) detector and selective area imaging enables additional capabilities, like layer sizing and chemical uniformity, that are useful for complex, multilayer materials and composite additive manufactured products.

The multilayer packaging material shown in Figure 13 (left) is composed of three different materials and six layers. Each polymer is identified by comparing the peak wavenumbers and intensities in a spectrum to those in an expansive database. In this way, a composition map can be constructed and overlaid with an optical image.

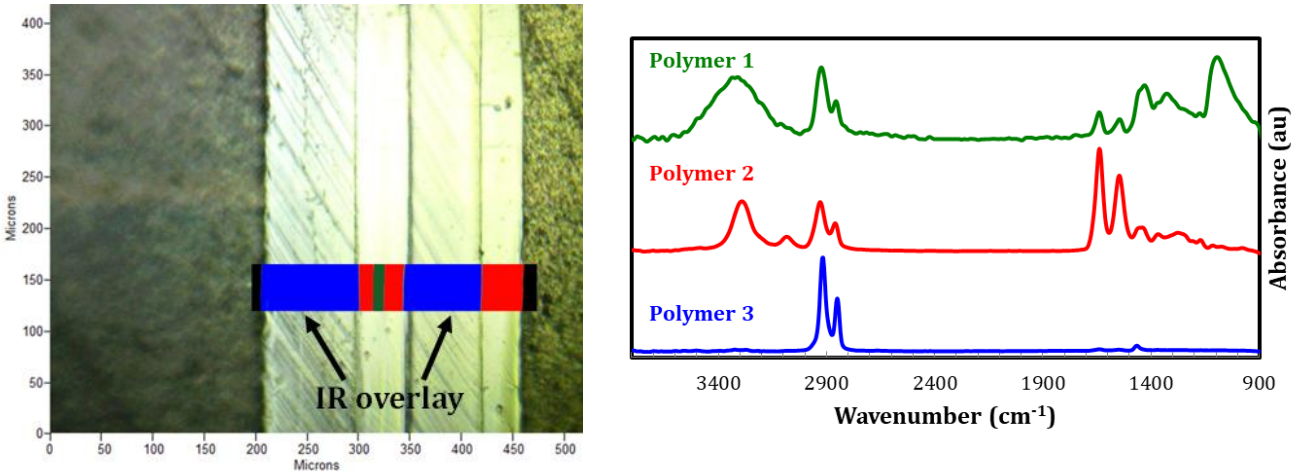

Figure 13. Optical image of a plastic packaging sample with IR image overlay (left) and corresponding FTIR spectra (right).

Case Study #14: Energy Dispersive X-Ray Spectroscopy (EDS)

EDS offers reliable elemental concentrations that are quantified based on the characteristic x-rays emitted from a sample, and concentrations can be measured at a single spot, across a designated line, or over a 2D region. The EDS detector is mounted within Ebatco's Scanning Electron Microscope (SEM); this allows the imaging and focusing capabilities of the SEM to be used in conjunction with the elemental identification of EDS.

An SEM image and EDS map of a pen that has been fabricated using laser powder bed fusion are shown in Figure 14. This additive manufactured process uses a laser to fuse regions of a metal powder bed, layer by layer, until a final product is achieved. While the SEM image reveals a rough topography on the sample surface, there are not obvious indications of an ineffective fusing process. The complimentary EDS map shows regions where the iron and cobalt powders have been intermixed well, but there are also distinct regions that have not mixed and appear to still have the composition and shape of the powder precursors. Depending on the desired application, this laser fusion process may need to be optimized to deliver a more uniform composition for more predictable material properties. The results in Figure 14, and the combined information from SEM and EDS can prove invaluable in the development of an additive manufactured product or process.

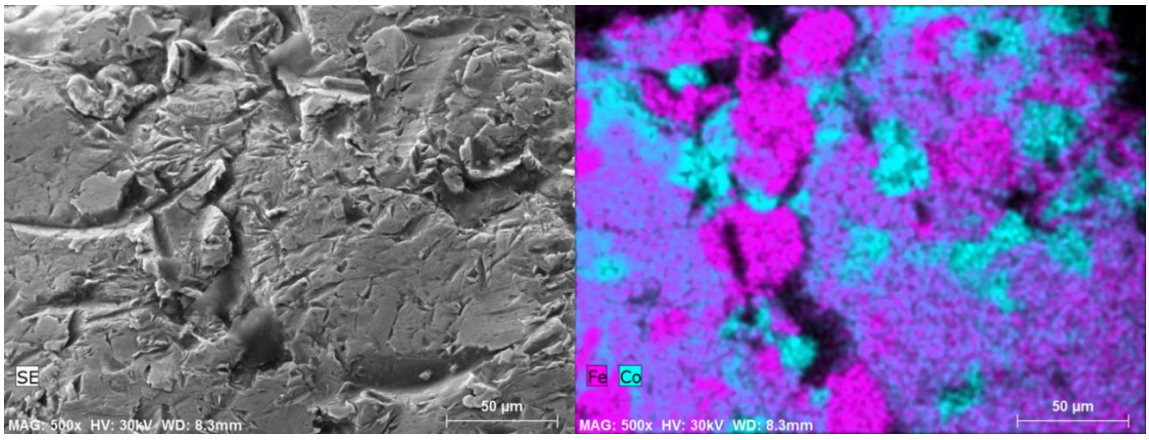

Figure 14. SEM image (left) and EDS map showing iron and cobalt distributions (right) for a pen manufactured by laser powder bed fusion.

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 additive manufacturing 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.