

Using Light and Electron Microscopy, Computed Tomography, and Light Scattering to Evaluate Additive Manufacturing Powders and Parts

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ABSTRACT

Customized test techniques using combinations of light and electron microscopy, computed tomography, and light scattering have been developed to evaluate the characteristics of both the powder feedstocks used in additive manufacturing and the finished parts. By combining these techniques, a more accurate assessment of additive manufacturing products is possible because each provides specialized benefits to the evaluation. Some are limited to individual types of data, while others are more versatile and provide information in several areas of interest. The high-resolution capabilities of the microscopy techniques increase the accuracy of linear and areal measurements and the scanning electron microscopy enhancement of performing chemical analysis provides additional unique benefits. Computed tomography has the ability to view particles and parts as three-dimensional features, finding internal defects, porosity, etc. The light scattering methods are valuable in being able to estimate particle size into the range from nanometer to millimeter. This technical paper will present both benefits and drawbacks of these techniques in hopes of finding the best combination of tests to characterize the additive manufacturing materials and products efficiently and accurately.

INTRODUCTION

Multiple test techniques are employed to characterize the powders used as the raw materials for additive manufacturing (AM). The intent of this testing is to evaluate the powders for product consistency and suitability for use. Examples of the properties typically measured are; particle size and shape, the amount of entrapped gas (porosity) within the particles, the metallic and non-metallic inclusion content, contamination of chemically dissimilar powders, and many others. Additionally, some of the same test equipment, although using different methods, is used to evaluate the finished AM parts. In this case,

product quality and consistency, as evidenced by defect content, microstructure both overall and localized, conformance to part design, in addition to other attributes, are evaluated.¹⁻³

Information on most of these characteristics can be accessed using visual techniques; consequently, digital imaging and automated image analysis are effective in their evaluation, however, where particle size is of primary importance, a light scattering approach can be effective. A short list of the most-used evaluation methods are:

- Visible light scattering (laser diffraction and dynamic light scattering)
- Light optical microscopy (dynamic and static image analysis)
- Computed tomography
- Scanning electron microscopy (imaging and chemical analysis)

While multiple means are available to access different types of data, it is advisable to utilize the best technique for the specific property or attribute. Combining methods is usually the best approach to characterizing the important traits in both the parts and powders. A brief description of each of these techniques is described below.

COMPARISON OF ANALYSIS METHODS

Visible Light Scattering Methods

Light scattering particle analysis consists of two techniques, e.g., laser diffraction and dynamic light scattering. The two are fundamentally different in what they see and detect in addition to how their respective measurements are made. Although unique in their approaches to particle characterization, both methods are limited to estimating the size of individual features.

Laser Diffraction

Laser diffraction, also known as laser light scattering, laser diffractometry, Fraunhofer diffraction, and/or Mie scattering, uses a beam of light, usually from a laser, to illuminate individual particles in either a wet or a dry environment. When the light interacts with particle edges, as shown in Figure 1(a), it scatters and creates a pattern of light waves behind the particle. The waves overlap and with the overlapping, there is a distribution of intensity fringes. This distribution contains maxima and minima in light intensity, where the intensity and spread of the pattern is directly proportional to the size of the particle. The angle of scatter is narrower and more intense for large particles, while wider and less intense for smaller particles, as seen in Figure 1(b).

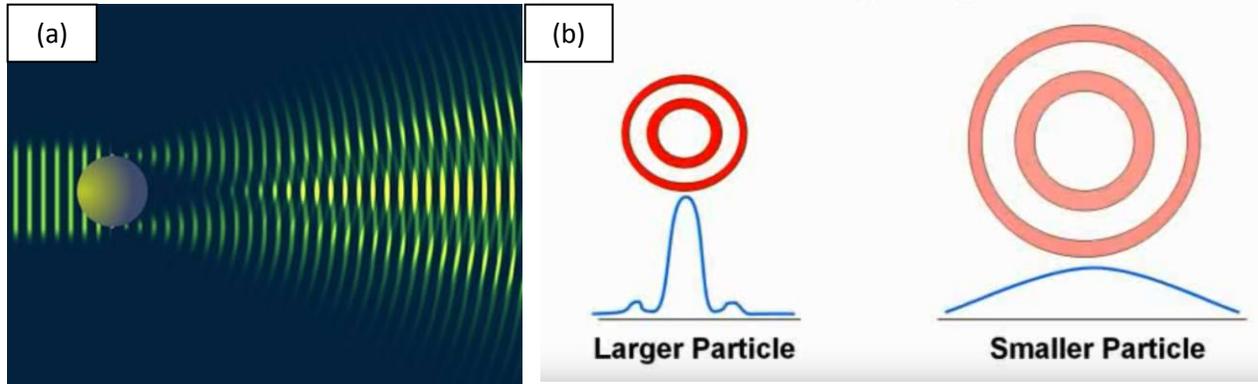


Figure 1. (a) Light approaching the spherical particle from the left in with the resulting wave pattern created on the right side. (b) The relative intensity and spread difference caused by particle size.^{4,5}

To generate, capture, and analyze these scatter patterns, laser diffraction uses two substantially different theories as the basis for their designs. They are the Fraunhofer and Mie theories, with both used successfully in the commercially available analyzers. The design differences in the two result in a variation in the particle size distributions in which they are effective. Fraunhofer diffraction is best used with particles larger than 20-50 μm while Mie scattering can analyze particles into the nanometer range. The upper size limit for both systems is normally in the millimeter range.

One drawback with laser diffraction is the assumption that the particles are hard spheres. The mathematics of the technique are based on spherical shapes and more information on the particles is required if the shape deviates from that of a sphere. Consequently, estimation of shape is difficult.

Laser diffraction also does not require calibration. It uses first principles to determine the size estimations. Therefore, making compensation or 'zeroing in' adjustments to the system to coordinate a measured value with a calibration standard value is not needed. However, it is advisable to analyze a sample of a known particle size distribution and compare the results to either historical performance or published data to observe if the results match. In reality, either the system is working or it is not.

Dynamic Light Scattering

Dynamic light scattering, also known as Photon Correlation Spectroscopy or Quasi-Elastic Light Scattering, uses the behavior of light scattered by suspended particles moving in a liquid as the basis for the estimation of particle size. In practice, a sample of fine powder is dispersed in a liquid, creating movement of the particles by Brownian motion. The suspended particles are illuminated using a laser and a photon detector placed at a fixed angle to the light source measures fluctuations in the scattering of light caused by movement of the particles. The amount and speed of particle movement are controlled by size, where smaller particles move faster and cause the fluctuations to occur more rapidly when compared with particles larger in size (Figure 2). These data are further processed to provide a size distribution estimate of the dispersed sample.

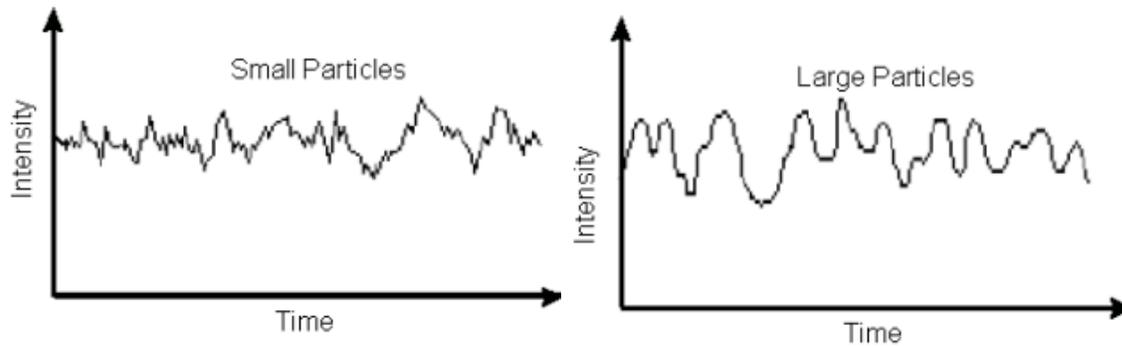


Figure 2. Typical intensity fluctuations for large and small particles.⁶

Dynamic light scattering has the same drawback as laser diffraction in that the particles are assumed to be spherical. In addition, since the technique assumes a particle shape to perform the analysis on the acquired data, no measured or calculated shape information is available. The technique has the advantage of being able to analyze particles in the nanometer range, considerably smaller than the capabilities of laser diffraction and the other size measuring methods.

Digital Imaging Methods

In each digital imaging method, an image is produced through the interaction of the sample with an energy source, e.g., light, X-rays, or electrons. The result of the exposure to the specific energy type is projected or reflected onto a detector sensitive to this energy. The detector is covered with an array of 'square', space filling, energy sensitive picture elements, aka, pixels, and it captures a single planar image of the illuminated sample. The captured image contains information at either the actual or magnified feature size, depending on whether the image is altered through optical or electronic magnification. In a given system, the pixel size on the detector is constant. It is what is presented to the detector that determines the resolution.

Each pixel, as it relates to the actual or virtual feature or part size, determines how much information can be used to evaluate a single feature or detail. Also, each pixel contains digital information of a single gray shade or, in the case of light optical microscopy, possibly a color. The overall planar images are constructed of the information in the entire pixel array.

The size of each pixel, again either as part of a magnified or actual-size feature, is known as the resolution of the system. The smaller the size of a pixel in relation to the size of a feature, the higher the resolution and the greater the accuracy of the measurements. In order to extract information on individual features, the gray or color digital data for each pixel is compared with a predetermined scale where a separation is made for those pixels within the scale and those outside.

Figure 3 shows a resolution simulation using a high-resolution image from a light microscope as the base, (a), and having two arrays of square features, i.e., simulated pixels, (b) and (c), overlaid onto it. A grayscale was chosen to represent the brightest simulated pixels, those contained entirely inside the particles. To include the information at the feature edges, the grayscale would need to be widened to include a darker gray since those pixels are partially inside the particle and partially inside the mounting material. This is illustrated in Figure 4 where Figure 3(c) is shown with green squares representing the

pixels containing edge-related information. The perimeters of the particle cross-sections are seen in the green pixels giving an idea of the gray value of these pixels. The number of simulated pixels in each particle cross-section is what the computer sees and uses for making the measurements of the size, shape, etc. of the individual particles. In addition, at the lower simulated resolution, several particles contain no simulated pixels since they are either smaller than the simulated pixel size or there is no coincidence of the particles with whole pixels due to the placement of the detector array. It is apparent, as the simulated pixel size is reduced; the number of simulated pixels contained within each particle is increased and approaches the actual size and shape of the features. The importance of image resolution cannot be overstated.

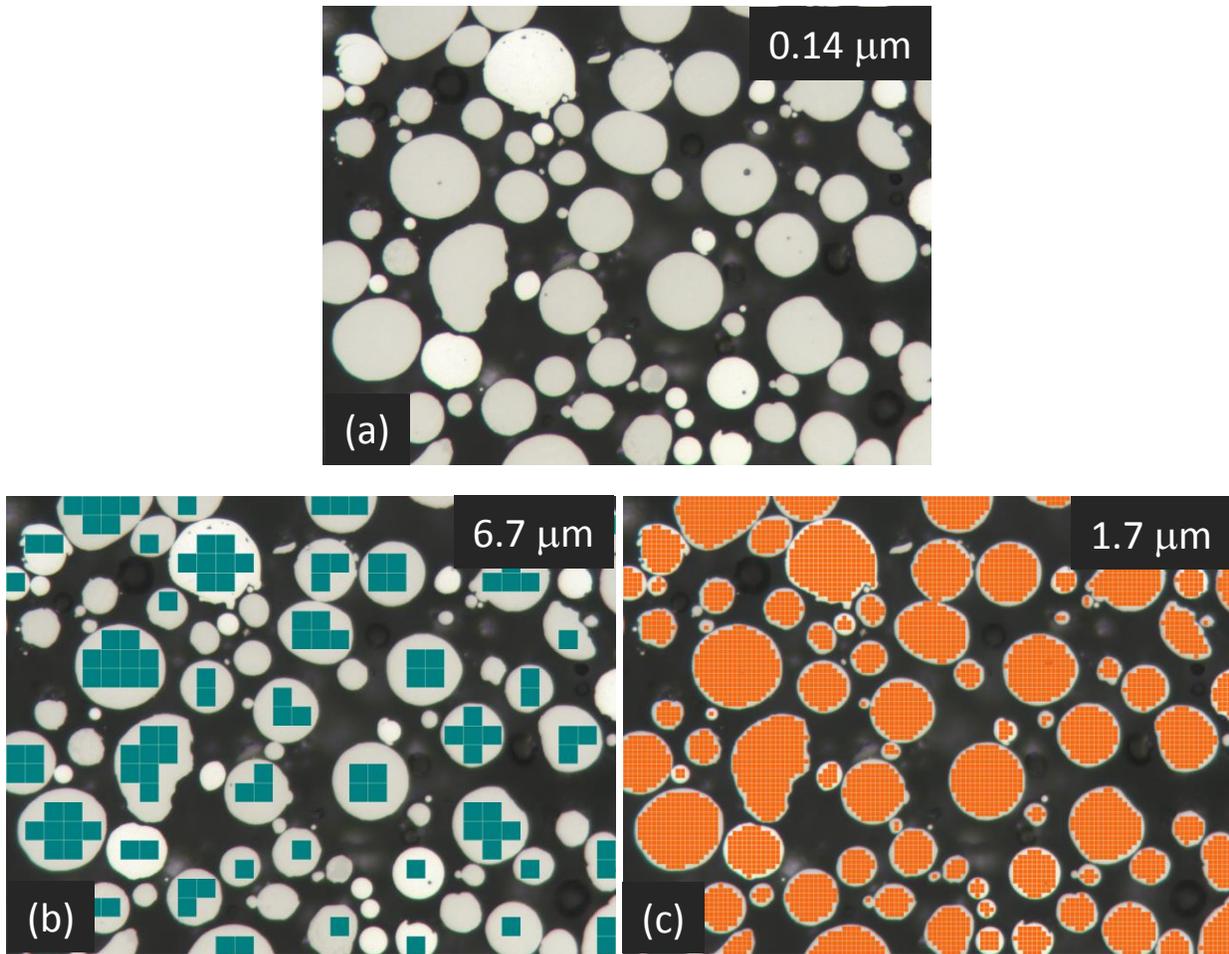


Figure 3. Cross-sections of an AM powder comparing pixel size of a light optical microscope image with overlaid pixel simulations. The (a) image is from a light optical microscope at a resolution of $0.14 \mu\text{m}/\text{pixel}$, (b) and (c) show blue and orange squares at the two lower resolution simulated pixels sizes. Simulated pixel size is contained in the upper right box.

With detection and feature definition determined by the selection of a gray or color level corresponding to the features of interest and since pixels represent a single gray or color level, the selection of the digital feature must be carefully made to produce a binary image as close to the actual feature as possible. The result of careful planning is the collection of data higher in accuracy. It should be noted that some of the

digital imaging systems permit or require a determination of this gray or color scale range to be made by an operator while others use predetermined settings.

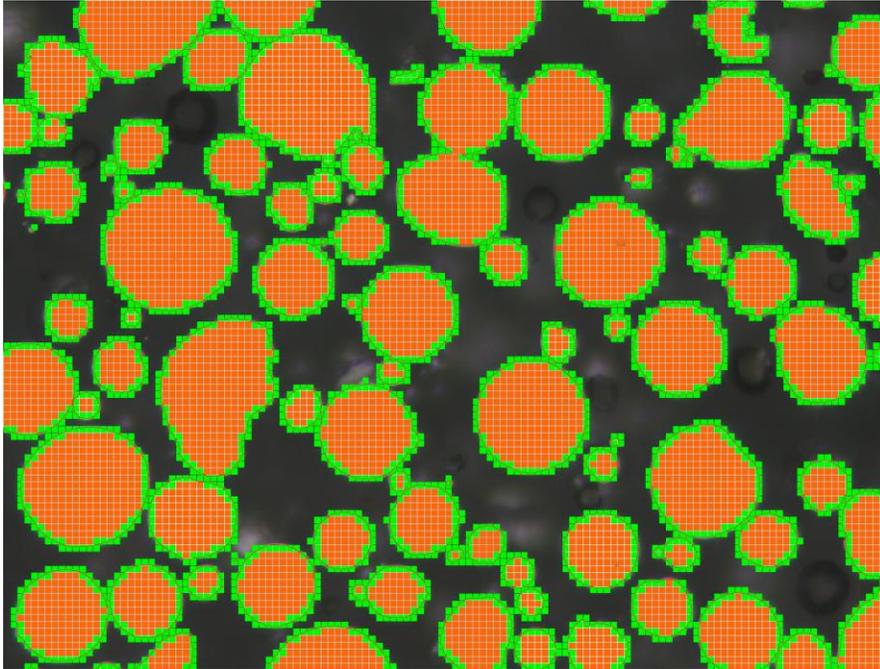


Figure 4. Green particle edges showing the pixels at the surfaces containing edge-related information.

The various digital imaging techniques have fundamentally different resolution capabilities and consequently, the accuracy of their respective measurements will vary. Further, not all measurements are performed in the same manner on the digital systems. Different algorithms are used to make basic distance and area estimations. One of the most difficult parameters to measure is the perimeter of a feature.⁷ This is notoriously difficult to measure and the accuracy at lower resolutions is often inadequate and ineffective in the creation of an effective shape descriptor.

Dynamic Image Analysis

In dynamic image analysis (DIA), a sample of loose powder is flowed between a pulsed light source and the digital detector. The detector acquires digital images as the illuminating light is pulsed. The image seen by the detector is a projection of the individual particles and appears as a dark shadow against a bright background. As an example, transmitted light images of two particle types are displayed in Figure 5. These are light optical microscope images and, in a comparison of particle size, the appearance of the particles in a DIA system is considerably smaller than what is shown in these images. Figure 6 is a schematic of a DIA system. While Figure 6 shows a single camera, the design can be modified to contain two cameras/detectors for image acquisition. In this two-camera design, a large field camera at a lower resolution captures images of the larger particles from the entire field and a second camera optically magnifies the particle flow, sees a smaller portion of the dispersed flow, and captures images of the smaller particles.⁸

In the DIA systems, the resolution can be between >10.0 and $0.8 \mu\text{m}/\text{pixel}$,⁹ although it is unlikely the larger particles are analyzed using the higher resolution. In addition, depending on the sample and particle size, hundreds of particles can be imaged per minute of acquisition time. Another interesting capability of the system is being able to store and match the grayscale image of each particle with the data that was acquired from it.

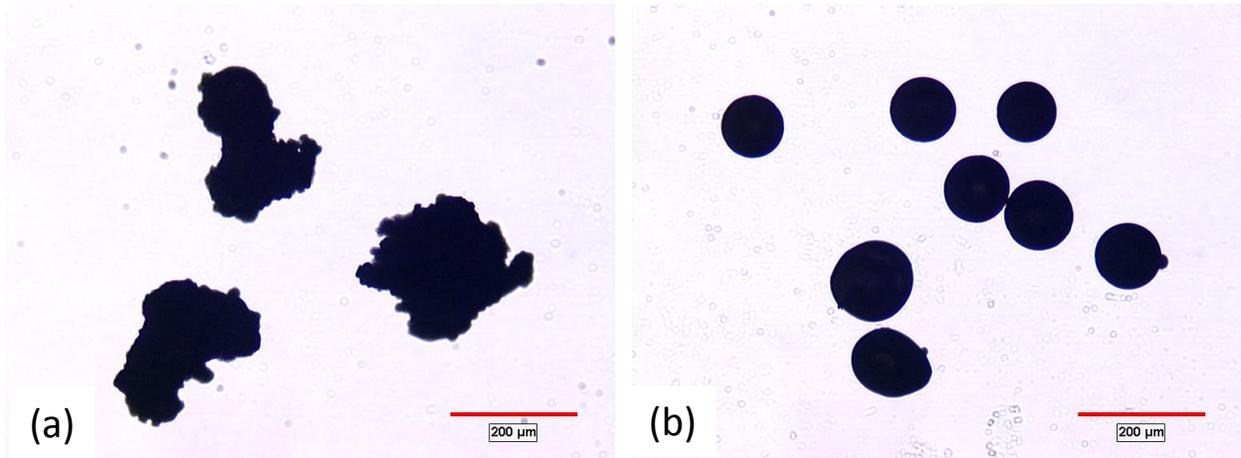


Figure 5. Transmitted light images of an atomized Fe (a) and gas atomized Ni-based alloy (b).

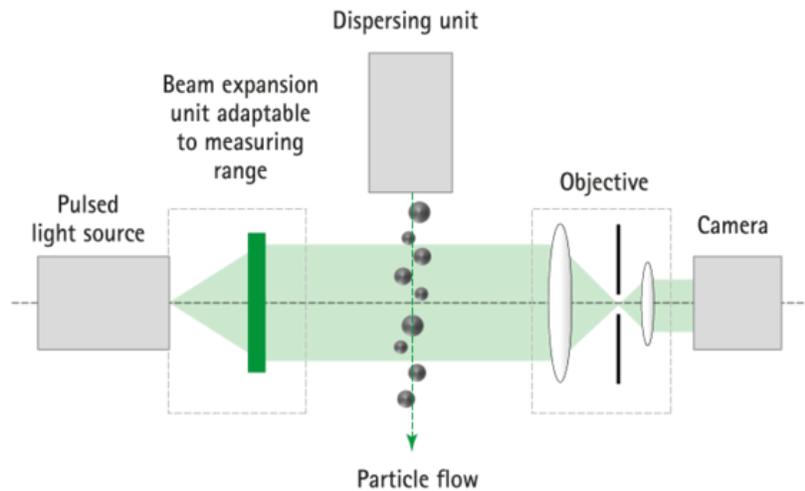


Figure 6. Schematic of a dynamic image analysis system.¹⁰

Static Image Analysis

The more traditional automated image analysis (AIA) is designed as a static system, where the particles and features being analyzed are mounted on either a transparent or translucent microscope slide or stationary cross-sections of particles or features visible in a prepared metallographic mount. Detection and image acquisition are the same as other digital imaging systems, with the graylevel or color of the features of interest used to make the separation of what is of interest and what is ignored. The resolution issue is also important with a static system because of the edges, which are a different gray or color compared with the feature interior.

Measurements in the static system are similar to what is performed with other digital systems. The advantage with a microscope-based system is the image is magnified, enlarged to increase the resolution to a higher level than many other systems where enlargement of the image is either not possible or substantially lower. In these AIA microscopy-based systems, the sample is often prepared or fixed on the microscope stage and an appropriate magnification is selected to ensure the features of interest are enlarged sufficiently to visualize the important details. Using AIA with the AM powders, cross-sections are usually used to provide the images for analysis. For example, Figure 7 shows cross-sections of loose powder particles at two magnifications. It is obvious the accuracy of measurements made on these images improve with an increase in magnification since the same detector is used in image acquisition and the virtual image at the higher magnification presents larger representations of the individual features.

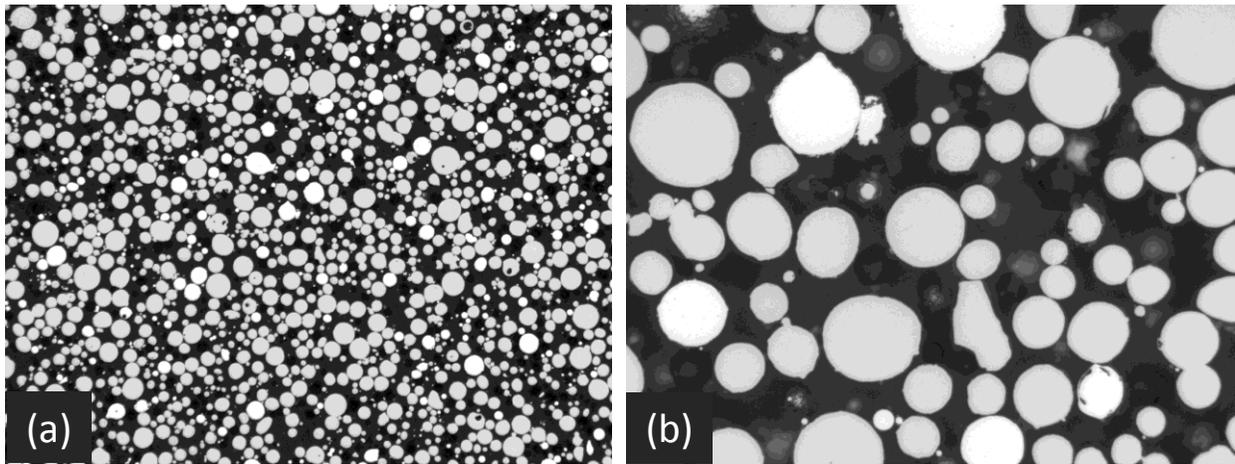


Figure 7. Images of the same sample of particle cross-sections acquired using a 10x (a) and 50X (b) objective lens. Respective resolutions of the images are (a), $0.69 \mu\text{m}/\text{pixel}$ and (b), $0.14 \mu\text{m}/\text{pixel}$.

Computed Tomography

High energy X-rays are used as the energy source for computed tomography (CT) scans. The sample, as small as a vial of powder particles or as large as an entire built part, is placed between the X-ray source and the detector, rotated, and irradiated with X-rays. Digital images are taken at predetermined times as the sample moves, with differences in the local feature density converted into the grayscale image. When possible, the sample is moved closer to the energy source producing a larger projected image of the sample with a small increase in magnification to the image. Where the other digital imaging techniques acquire single planar images for analysis, the CT scanner takes multiple images as the sample rotates. This collection of images is assembled into a 3D representation of the sample. Once constructed, the operator is able to manipulate the 3D image by rotation and tilt to visualize the locations of features that are significantly different in density compared with the base alloy. For instance, low-density features or pores are displayed as dark regions and those high in density, such as tungsten particles in a titanium alloy sample, will be brighter in gray level.

The variation in density between two materials must be relatively large to visualize a change in gray level. For example, a Ti-6Al-4V contaminated with a small amount of 316L will probably not show a noticeable difference in graylevel even though the respective density difference is approximately $4.5 \text{ g}/\text{cm}^3$ and $8.0 \text{ g}/\text{cm}^3$. However, the same Ti-6Al-4V containing a small amount of tungsten at $19.3 \text{ g}/\text{cm}^3$ will show

a large graylevel difference, with the W particles appearing bright against the darker gray Ti-alloy particles. This can be seen in Figure 8, which is a slice through a 3D image of a CT scan. The small bright W particle is shown within the circle.

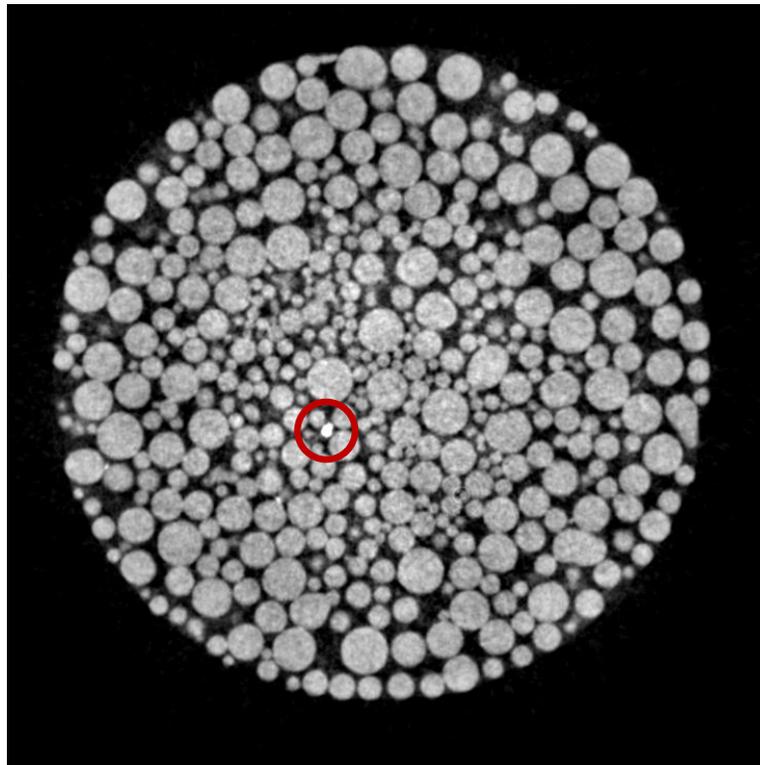


Figure 8. A slice through a CT scan of a Ti powder manufactured using the plasma rotating electrode process (PREP) showing a W particle inside the red circle.

Resolution of the CT systems may also be an issue. The enlargement of small samples, such as the AM particles shown in Figure 8, is relatively minimal since the amount of magnification to the image is low. In many cases, the resolution of the CT scanners is $>3 \mu\text{m}/\text{pixel}$, which equates to the smaller particles being defined by a few pixels and any internal porosity or defects seen and measured by even fewer. Depending on the placement of the particles in relation to the graylevel detector, some smaller particles may be ignored from the analysis.

Where small features and particles may be a problem for the CT scanners, some of the scanners have chambers where entire large parts can be imaged and evaluated. Resolution of the small defects or features in the part will still be a problem, but the advantage of having the capability to visualize an entire 3D part provides a distinct advantage compared with other analysis methods.

Scanning Electron Microscopy

Probably the most versatile collection of analysis capabilities is found with the scanning electron microscope (SEM). It uses a beam of electrons to bombard a sample surface, liberating various types of information in the process. The information includes two electron types, secondary and backscattered, which reveal surface details or local atomic number differences in the sample. In addition, chemical

composition information is excited and can be used to determine the composition of features in the magnified areas. The microscope is capable of magnifications hundreds of times greater than what is possible with a light microscope, with resulting resolutions in the nanometer range.

The two electron types used for imaging display different kinds of information. Secondary electron images (SEI) show better representations and more detail of surfaces and backscattered electron images (BEI) show differences in atomic number and variations in local crystal orientation. Regardless of electron type used, the images appear as projections of topographic or planar surfaces. Examples are shown in Figure 9. A secondary electron image of a gas atomized Ti alloy AM powder sample is shown in Figure 9 (a) and in (b), a cross-section from a titanium alloy ingot acquired using backscattered electrons shows the local orientation differences, which appear as variations in grayscale.

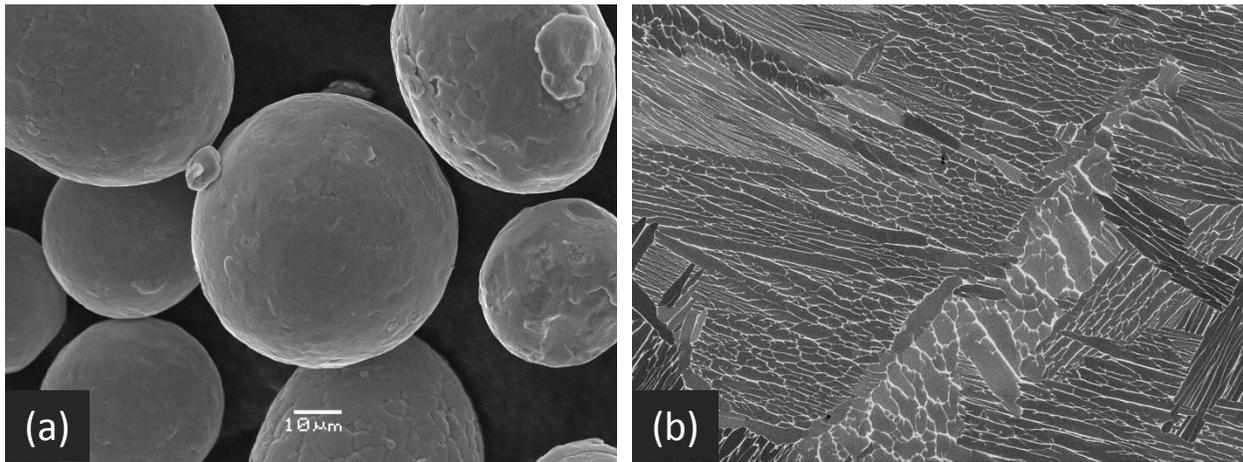


Figure 9. Gas atomized AM powder imaged using secondary electrons in (a) and a backscattered electron image of a cross-section from a Ti alloy ingot etched using Kroll's reagent in (b).

In addition to imaging at a wide range of magnifications, the capabilities of the SEM can be enhanced with the installation of an Energy Dispersive Spectrometer (EDS) to perform chemical analysis on field and feature-based locations in a sample. Chemical analysis of individual particles, phases, transformation products, entire magnified microscope fields, etc. can be performed with accuracy of the results as qualitative, semi-quantitative, or improved to quantitative. This is dependent on the computer software and the use of certified standards.

The chemical analysis capability is particularly beneficial for analyzing phase precipitation, diffusion, defect composition, the presence of cross-product contamination, etc. An example showing the presence of a Ti alloy contaminant in a different Ti alloy base powder is illustrated in Figure 10. In this case, a Ti-6Al-4V is the base powder and a second Ti alloy, commercially pure (CP) Ti, has been unintentionally mixed into it. The (a) image is a SEI of a field selected randomly for analysis and the (b) image, is a combination of elemental maps overlaid on the SEI to pinpoint the location of the CP Ti particles. In the (b) image, the base Ti-6Al-4V particles are colored yellow and the CP Ti particles are red.

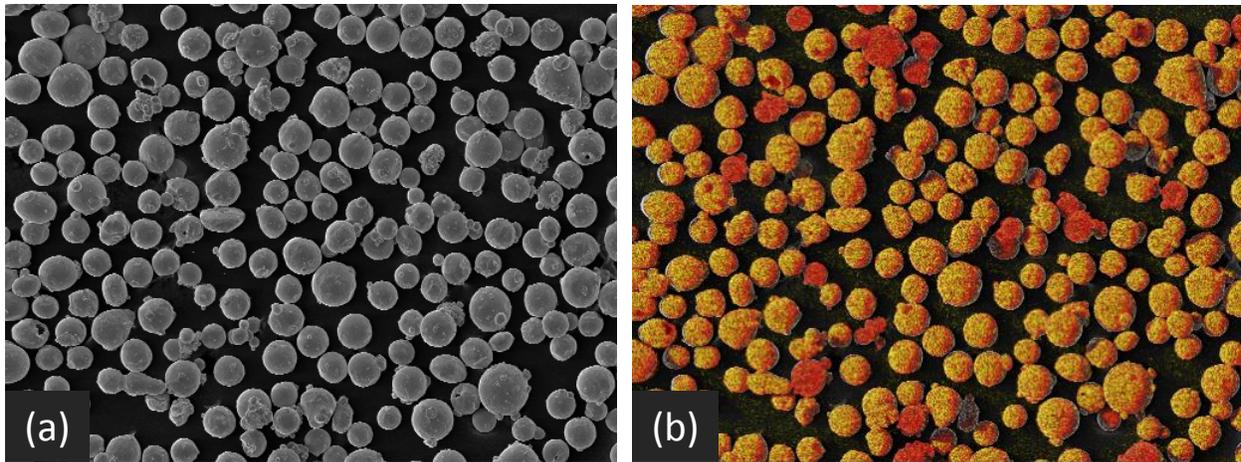


Figure 10. A randomly chosen field of the sample suspected of being contaminated. (a) is a SEI of the particles and (b) is the combination of elemental maps overlaid on the SEI. The base yellow particles are the Ti-6Al-4V and the CP Ti contaminant is red

COMPARISON OF TEST RESULTS

Proper use of the aforementioned test methods helps ensure the AM powders and parts are made to meet and exceed the expectations of the producer, customer, and ultimately, the end-user. Each method has benefits but all also have limitations. The data generated using each is varied, sometimes limited to one type of sample or generated information, and should be evaluated for relevance and accuracy. The user of the devices must determine which feature characteristics require measurement, then choose the appropriate test method that best fits the sample. The following gives a brief review of what is required by each method and what results are produced.

Light Scattering

These techniques are used to measure particle size only and do not use digital imaging to generate the information. The range in applicable size is quite large considering both methods, e.g., laser diffraction and dynamic light scattering, ranging from near nano-scale to several millimeters in size for laser diffraction and moving smaller, to <10 nm to the millimeter range for dynamic light scattering. One serious drawback with light scattering is the assumed particle shape is spherical. In cases where this assumption matches the actual particle shape, the variation from actual to measured size could be negligible. Analyzing particles with shapes deviating from a sphere will probably result in large inaccuracies in the analysis. Also, with the fundamental differences in system design, they will not produce the same results.

Dynamic Image Analysis

Similar to the light scattering methods, dynamic image analysis is also used for characterization of particulates. However, unlike light scattering techniques, it can be used to give more than size-only information in the analysis.

The DIA design detects an outline or shadow image of the particles while in flight without ‘seeing’ the details of the particle surfaces. Essentially, what is imaged is a projection of the particle extremes at a single orientation. Resolution capabilities of DIA are also limited compared with the techniques where the particles are magnified. With the highest resolution as small as 0.8 μm/pixel, the accuracy of analyzing smaller particles may be questionable. In some circumstances, this may be a serious issue when the particle size distribution is in the AM size range of <50 μm. Conversely, while the range of applicable sizes is quite wide, larger particles may be well characterized. Consideration must be given to the importance of small features, especially at the particle perimeters.

As a practical example of the effect of resolution limits and the size of surface details, several AM grade powders were analyzed using one of the DIA systems in hopes of finding a correlation between average particle shape and powder flow using a Carney flowmeter. Two shape estimators were used. They were convexity and roundness, defined as:

$$\text{Convexity} - (A_{\text{actual}}/A_{\text{convex}})^{0.5} \tag{1}$$

$$\text{Roundness} - (4\pi A_{\text{actual}})/P^2 \tag{2}$$

where A_{actual} is the area of the projected particle image, A_{convex} is the convex area of the particle projection; P is the perimeter of the actual particle projection. Looking at the expressions, the maximum value for both is 1, which is a circle for roundness and a shape with a completely smooth surface for convexity.

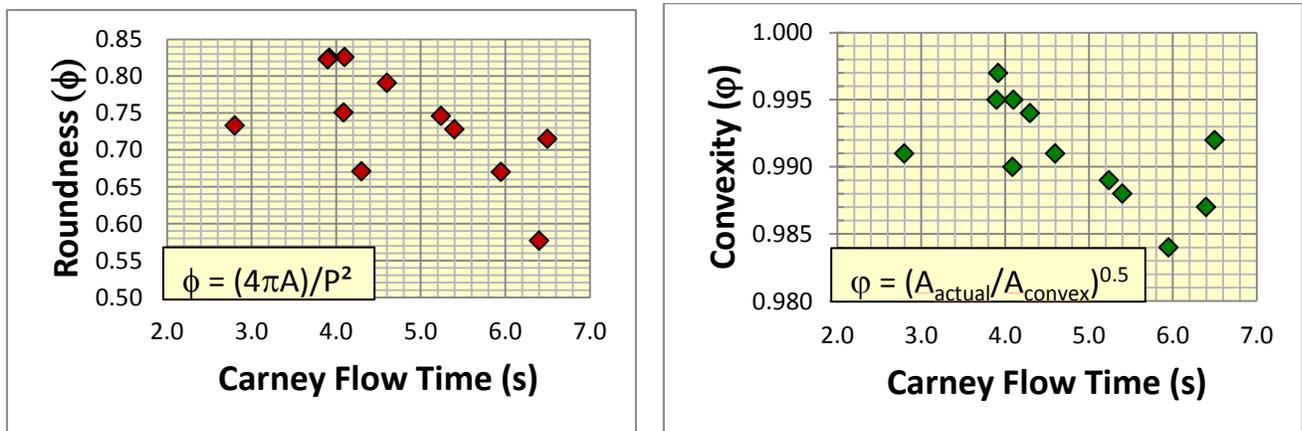


Figure 11. Correlation graphs of roundness and convexity for >10 lots of AM grade alloys.

In both graphs from Figure 11, the trend for the correlation appears to be correct, with a faster flow measured for rounder and smoother particles. However, looking at the data, the roundness range is 0.57 to 0.83, which is a wide spread. The 0.57 value equates to a perimeter length ≈30% longer than an equivalent area circle perimeter length, compared with a ≈10% increase in perimeter for the 0.83. The indications are the particles are substantially different in shape. Further, to achieve the spread in convexity values in the graph of 0.984 to 0.997, the actual area would be 0.97 – 0.995 (97 – 99.5%) of the convex area measurement. It seems the convexity difference is remarkably small compared with the roundness spread of 0.57 - 0.83. This is an example illustrating, while some measurement data may be

accurate, results and techniques must be scrutinized. It should be remembered the small particle size distribution of the AM powders might present difficulties with some of the test techniques.

Static Image Analysis

Light optical microscopy (LOM) is the primary vehicle for performing static image analysis (IA). Ordinarily, a light optical microscope is attached to an automated system that captures and analyzes the digital images, using automation to step through a predetermined stage pattern and focus the sample. The area covered by the stage movement can be customized to fit specific sample shapes and the data can be archived for later use. A major benefit of this system is the ability to change magnification on the microscope, thus increasing the resolution and the accuracy of the measurements. The limitation of this arrangement is the samples must be planar in the form of cross-sections of powder particles or parts, or whole particles fixed to transparent or translucent slides. Both reflected and transmitted light can be used for illumination. The analysis is two dimensional and no volume-related information is generated directly.

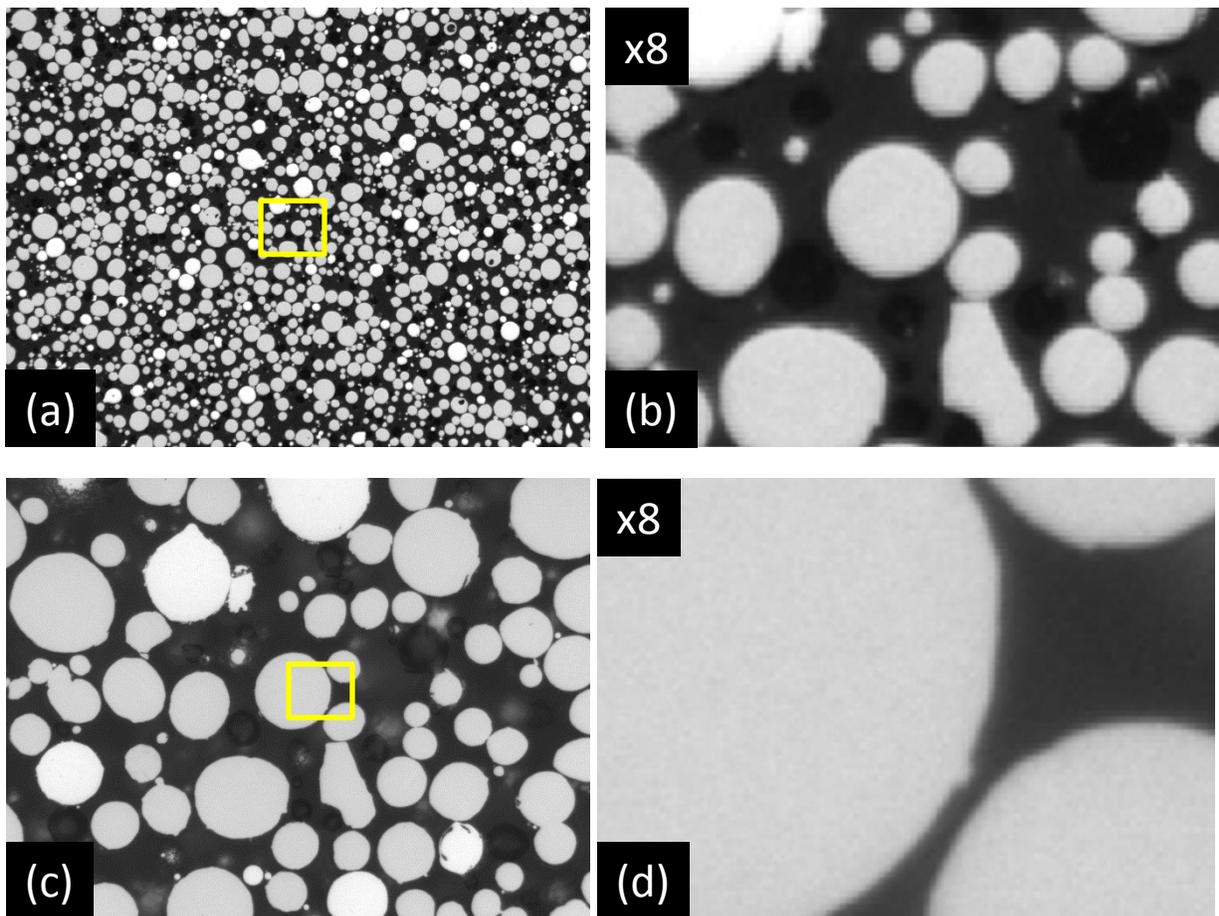


Figure 12. LOM images showing the improvement in resolution attained through increased magnification. The (a) and (c) images are taken directly from the digital camera and the (b) and (d) images are enlarged 8x in an effort to show the square, pixel size. With these resolutions, the pixel size is difficult to see with the enlarged images, although the edges appear slightly fuzzy.

The benefit of increased resolution can be seen in Figure 12, which is cross-sections of powder particles imaged at two magnifications. The (a) and (c) images are unaltered and as acquired from the IA system. Their relative resolutions are $0.69 \mu\text{m}/\text{pixel}$ for (a) and $0.14 \mu\text{m}/\text{pixel}$ for (c). Although difficult to see, the square representations of the pixels, as simulated in Figures 3 and 4, are the same here, only more than 10 times smaller than the simulation. The (b) and (d) images are 8x enlargements of the outlined regions in (a) and (c) images to show, although difficult to see, the square pixels along the particle edges. The magnified size results in an increase in accuracy of the subsequent measurements, with substantially more detail imaged and available to be included in the measurement.

Another advantage of sectioning the samples during preparation is to detect and measure entrapped gas, pores, inside the powder particles. An example can be seen in Figure 13, which shows a grayscale image, (a), on the left with pores detected and shown as red features in the right image, (b). These features are not available to the light scattering and DIA techniques. They cannot view inside the particles.

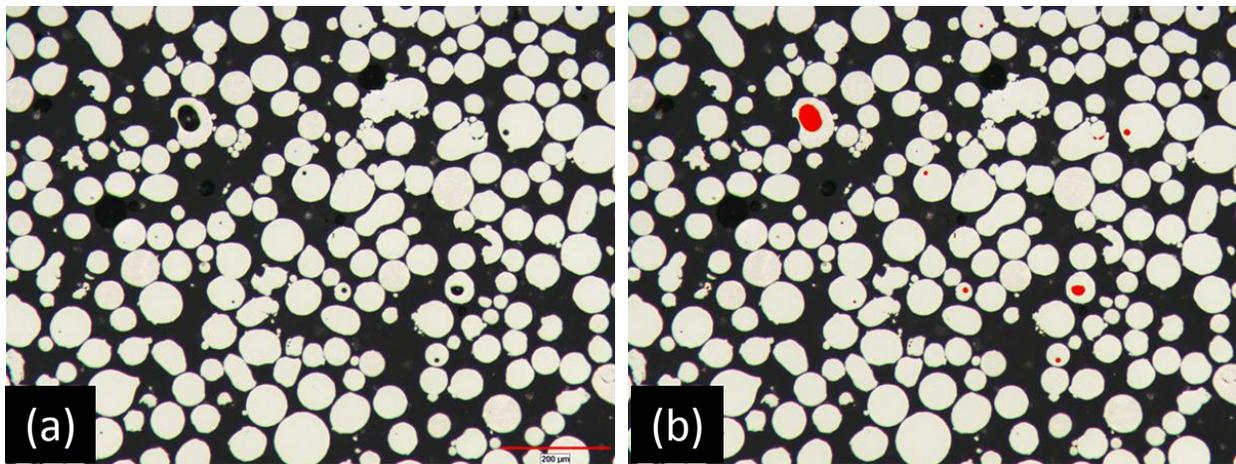


Figure 13. A grayscale image showing particle cross-sections (a) and the internal pores displayed as the red features in the (b) image.

Information accessible using the AIA systems are linear and areal data in both field and feature-related measurements, in addition to defect size distributions and locations, microstructural quantification, phase transformation, diffusion behavior, spatial relationships, in addition to virtually all characteristics that can be distinguished by gray, color, size, shape, etc.

Computed Tomography

As mentioned earlier, CT scans use high energy X-rays as the energy source. The X-rays are passed through the sample in multiple passes, accumulating planar images at various indexed locations and at different times inside the sample volume. These images are similar to the 2D images acquired using other digital imaging methods. They are assembled into a three dimensional representation of the sample volume using volume elements known as voxels, which are analogous to the pixels in a 2D image. Resolution limitations with voxels are similar to what is experienced with other digital techniques. Feature distinction with a CT scan is made by local density in the sample and not by visual appearance or chemical composition.

The CT scanners are normally lower in resolution compared with either of the microscopy techniques, e.g., LOM and SEM. This results in difficulties in ‘seeing’ and distinguishing small features and feature edges in a sample. To improve this situation, techniques have been developed to electronically enhance a CT image in an effort to improve image appearance, artificially improve resolution, and make feature detection easier and less prone to errors by an operator. This helps, but separating features of interest is still difficult and not as straightforward and repeatable as with some other digital imaging methods. An example of the electronic enhancement of an acquired planar section is shown in Figure 14, where a pore was detected in an AlSi casting, outlined, and measured. With the similarity in the appearance of the detected feature with other locations in the two images, it is difficult to determine the identity of the components characterized by the various gray levels.

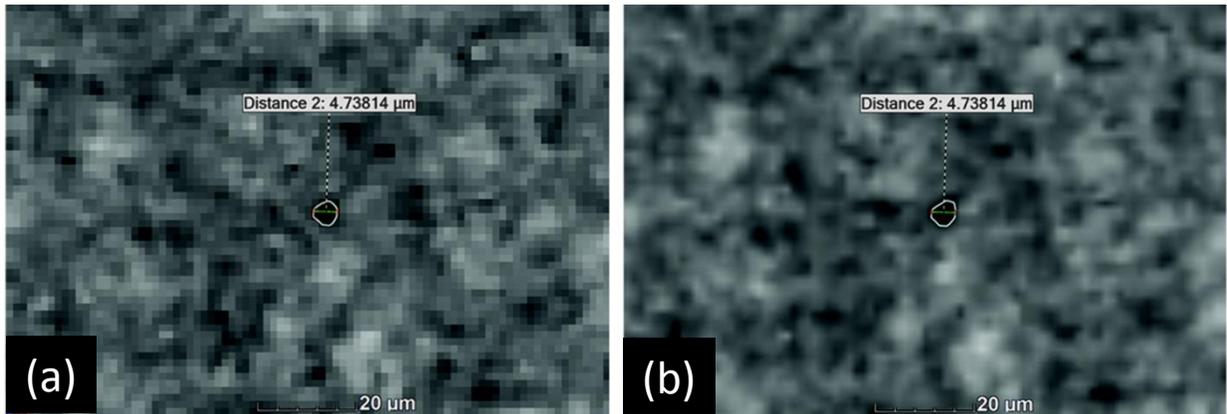


Figure 14. CT scan images with (a) as-acquired and (b) electronically manipulated to alter pixelation.¹¹

As with other analysis techniques, CT scanning has major benefits. It is the only imaging method to provide three dimensional, volume-related information in a single image. This can be seen in Figure 15, which shows the locations of defects in the same AlSi casting that was mentioned above in Figure 14. The individual colors represent the volumetric sizes of the defects, in this case, as blue and red.

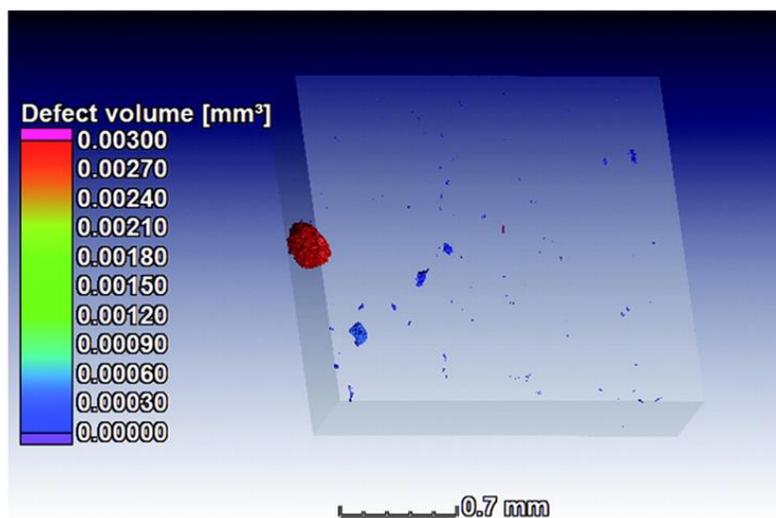


Figure 15. CT scan volume showing the location of pores and defects as colored features in the volume.¹¹

CT scanning is also effective in comparing an AM built part with the CAD design of the part for dimensional accuracy. An example is shown in Figure 16 as a comparison of two parts of the same design, manufactured by two methods. The upper part labeled AM1 was manufactured by fused deposition modeling (FDM) and the AM2 part by the more dimensionally accurate, stereolithography. It can be seen in the comparison of the two parts that spatial distortions have taken place in several locations in the FDM part especially, with the horizontal arms like the region within the oval.

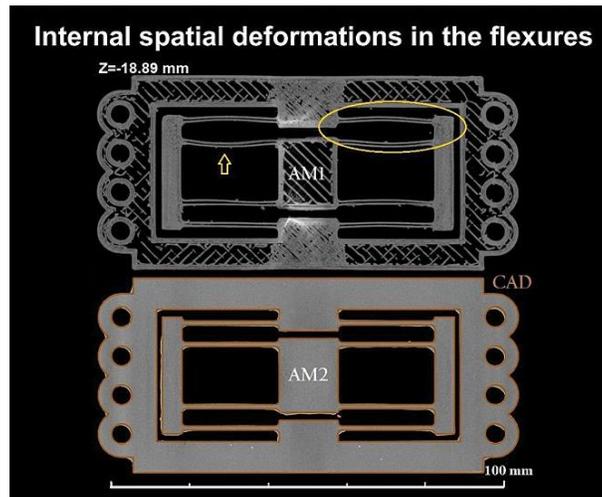


Figure 16. Comparison of the dimensional accuracy of a fused deposition modeled part (AM1) with the same part manufactured using higher resolution stereolithography. Samples were scanned to compare dimensions.¹²

A few of the difficulties with CT scanning are:

- High density materials may present more difficulties because more power is needed to penetrate the part.
- Parts made from multiple materials can be a problem if the densities of the materials are not similar. Multiple scans could be required to produce acceptable images.
- Irradiation of different alloys may present the problem of noisy images or poor quality scans.
- Part size may be an issue, especially if a high resolution scan is needed.
- Image resolution is lower compared with the microscopy techniques, LOM and SEM.

Scanning Electron Microscopy

The SEM is capable of looking at prepared planar or topographic surfaces, but it is not able to look into the sample volume. Resolution limits are higher than other characterization techniques due to the ability to examine features at high magnifications. Details on surfaces, such as powder particles or fracture surfaces, are revealed using secondary electrons and variation in atomic number is visualized through the emission and detection of backscattered electrons. The microscope equipped with an EDS system raises overall capabilities to include the ability to perform chemical analysis on microscopic areas.

Examples of SEI and BEI are seen in Figure 17. The (a) image is a CP Ti AM powder sample taken using secondary electrons. At this magnification, the surface details are clear as is the appearance of

small satellites and protrusions on the particle surfaces. A BEI of a Ti-6Al-4V in the (b) image has a small percentage of 316L contamination, which can be seen as the brighter particles that have been highlighted using the green circles. The 316L particles are the lighter gray due to the higher atomic number and highlighted inside the green circles. If desired, magnifications can be increased to improve the resolution and raise the likelihood of seeing the particles more clearly.

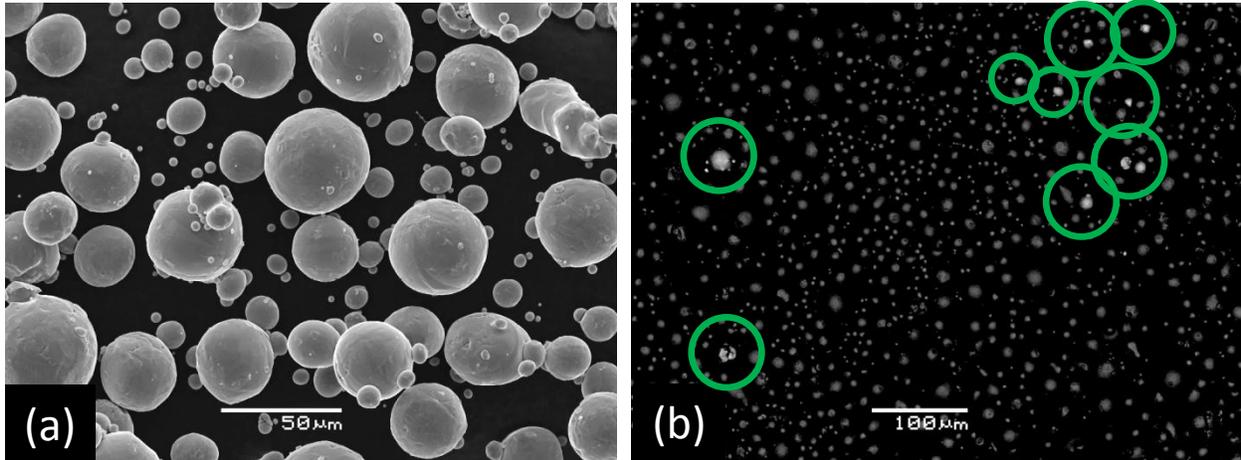


Figure 17. Examples of SEM imaging methods. (a) SEI of AM particles and (b), BEI of Ti alloy with higher density 316L particles within the green circles.

An example of having the ability to determine chemical composition is seen in Figure 18, where a sample of CP Ti with a higher-than-normal Fe content was examined for possible contamination by another AM alloy. In the procedure, a sample of loose particles was distributed onto electrically conductive double-sided adhesive tape that had been attached to a conductive substrate. The sample was imaged and elemental maps made, locating the chemical elements present in the microscope fields. By looking at the SEI overlaid with the Ti and Fe Ka maps, small areas were shown as green, which was the indicator color for Fe. This was thought to be the possible source of the higher Fe content.

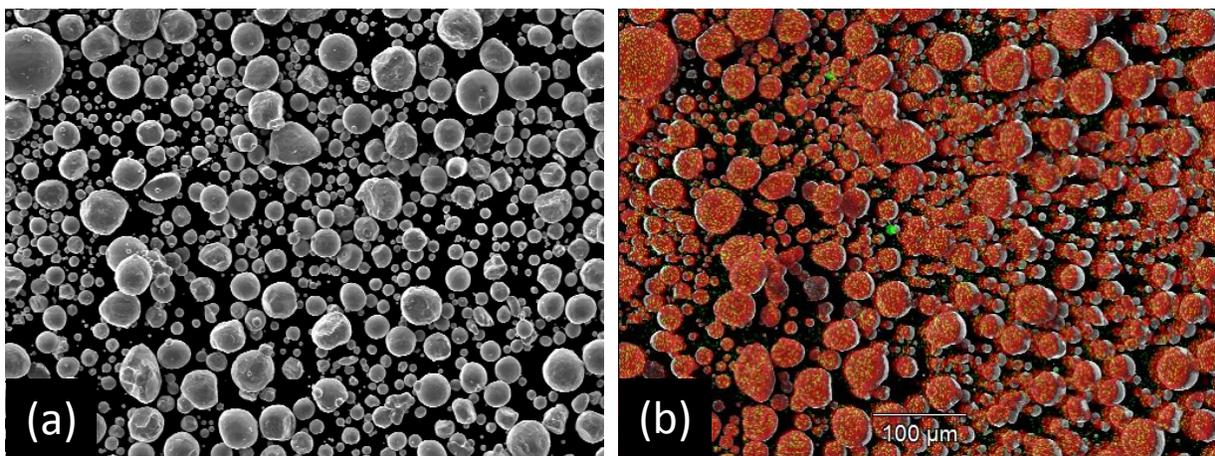


Figure 18. Visual representation of element distribution in a CP Ti alloy. (a) is a SEI of the sample field and (b) is the appearance of the SEI with overlays of Ti (red) and Fe (green) elemental maps.

The magnification was then increased to isolate one of the green colored particles for a more positive identification of the chemical composition signature. The higher magnification image, Figure 19, was taken and the suspect particle analyzed. It was found to be a stainless steel, specifically 316L, which confirmed the contamination source responsible for the higher than normal Fe content.

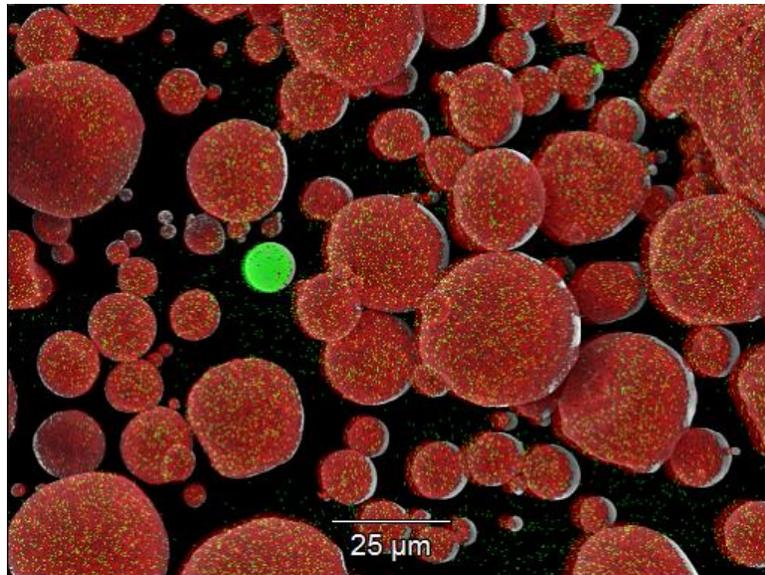


Figure 19. Detail of the Fe-rich particle located near the center of the field from Figure 18.

CONCLUSIONS

Multiple characterization techniques are available to provide the data required to determine the quality and consistency of the AM powders and parts. The following brief synopsis of the available methods provides information on some of the qualities, both positive and negative, in each.

The light scattering systems are limited to measuring particle size distributions and can operate into the nanometer scale range. They assume particle shape to be a sphere, which in some cases may result in the generation of erroneous data depending on the shape of the particles being tested.

The image analysis methods are separated into two categories, i.e., dynamic and static. Both use digital imaging, although the resolution of the dynamic system is substantially lower in comparison. The methods are limited to visual information from the sample. The dynamic system acquires images while the particles are in-flight, while the static system uses stationary samples for imaging. Static image analysis is the more traditional automated image analysis and has the benefit of adjusting magnification on a light microscope to conform to the needs of the sample. Any of the digital-based measurement techniques can be used with the image analysis methods.

Computed tomography has the ability to generate volume-related images by combining planar images into representations of the scanned sample volume. It can show defects and features locations, with their detection determined by variations in density. The resolution is lower than the microscopy methods because the amount of magnification is low and limited. Whole parts can be scanned if this is needed.

Scanning electron microscopy has the capability to provide planar images of samples in a large magnification range, hundreds of times the range of other techniques. The images are projections of either prepared planar or topographic surfaces. SEM is capable of imaging to reveal local differences in atomic number and, with the addition of an energy dispersive spectrometer, determine the chemical composition of magnified features.

It is recommended to combine test methods to generate the most relevant and valuable data on the samples being analyzed. No single test method will answer all of the questions or provide all the information.

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