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S C I E N T I F I C

Automated analysis of powder quality and defects in additively manufactured components using Perception Software

Metal additive manufacturing has emerged as a promising new technology primarily for aerospace and biomedical component applications.

Technologies such as powder-bed or directed energy deposition require the use of metal powders. The powder chemistry, size distribution, and sphericity play key roles for the beam-induced melting process. While established technologies exist to examine powder size distributions (for example, laser-diffraction-based techniques), in some instances, powders are now used with sub-micrometer sizes that are very difficult to assess with these techniques. Existing technologies to analyze powders cannot distinguish between different elements or chemistries.

Challenge

Despite the novel and advanced nature of additive manufacturing, many of the issues that have long troubled traditional manufacturing techniques, such as defects and porosity, also emerge in additively manufactured parts. Therefore, much focus is currently placed on determining the nature of defects in additively manufactured parts and their link to the processing parameters. Here again, a manual defect-by-defect analysis using electron microscopy cannot yield defect size and shape distributions with the sampling sizes that are required by many manufacturers.
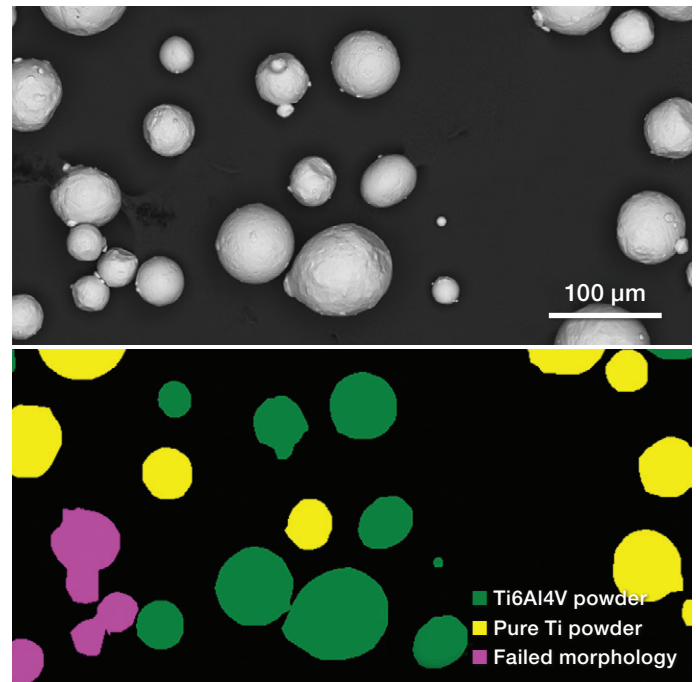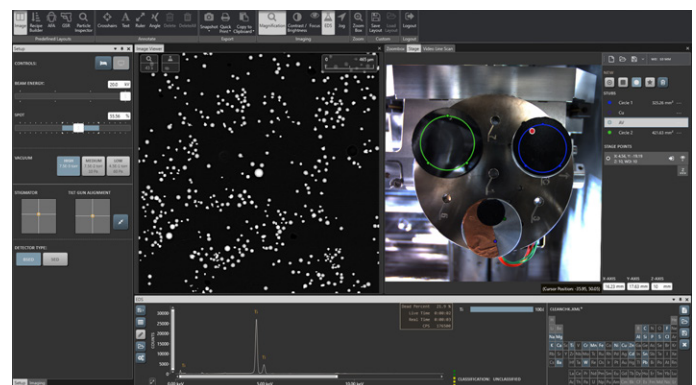

100 µm

■ Ti6Al4V powder
■ Pure Ti powder
■ Failed morphology

Figure 1. Example of chemistry-specific particle analysis for Ti-6Al-4V powder.

Perception Software enables automated morphology and chemistry particle analysis.
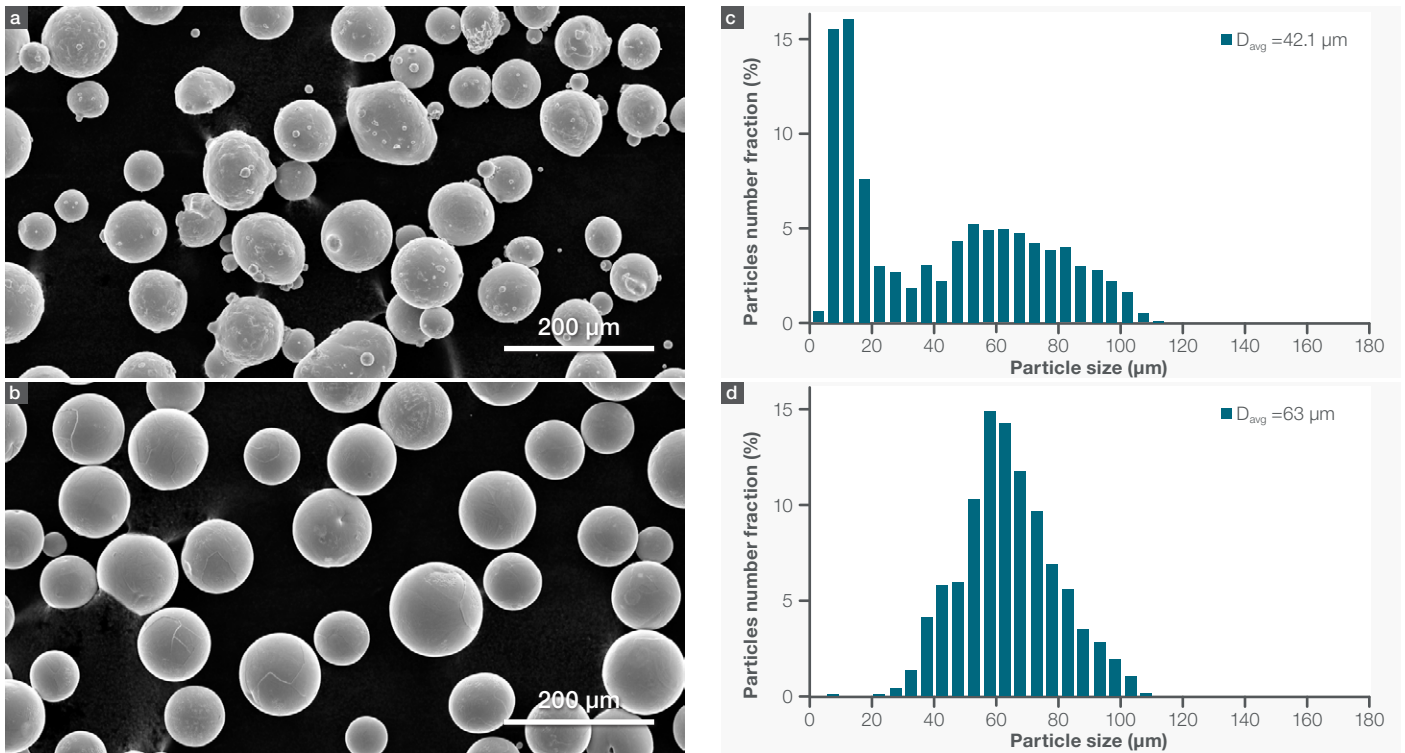
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Figure 2. Secondary electron SEM images and the corresponding particle size distributions measured for Supplier A (a, b) and Supplier B (c,d) Ti-6Al-4V powder.

Method

In this application note, we show how Thermo Scientific™ scanning electron microscopes combined with energy-dispersive X-ray spectroscopy (SEM-EDS) and Thermo Scientific Perception Software can be used to reveal powder particle size distributions and defect distributions. Not only can size and shape distributions be obtained, but the EDS detector can also be used to differentiate between powder particles or microstructural features with different chemistries. For this example, commercial Ti-6Al-4V powders were obtained from two vendors, hereafter referred to as Supplier A and Supplier B.

Perception Software enables automated statistical analyses of microstructures and particles with both morphology and chemistry filters. For example, Figure 1a shows a blend of pure titanium and Ti-6Al-4V powder particles. Both powder particle types are almost perfectly spherical and their Z contrast is similar. The two powder particle types are therefore not distinguishable with backscattered electron (BSE) imaging. However, with Perception Software, a strategy can be devised to analyze either one of the two powder types.

Analysis

In the first step of the analysis, the software separates all particles from the background using an image contrast threshold criterion. Once the scanning electron beam detects features with image pixel intensities above the threshold, the instrument performs a quick EDS measurement of these features as a rough chemistry assessment. The rough chemistry information is then used for a rule set with morphology and chemistry filters.

In this example, the result of applying the filters is shown the second image of Figure 1. The yellow color-coded pure titanium powder was rejected by the chemistry composition filters, and the scanning electron beam skipped these features. Precise size and EDS chemistry analysis was then carried out for only the particles that passed chemistry and morphology filters, shown in Figure 1 as green and purple color-coded Ti-6Al-4V powder particles. The particle aspect ratio can be used as an additional filter. A threshold was set for the aspect ratio so that green color-coded spherical or nearly-spherical particles were separated from the purple color-coded irregularly shaped particles. This approach avoids complications from overlapping or otherwise agglomerated particles contributing to the partical size analysis. As a result of the chemistry and morphology rule set, only spherical Ti-6Al-4V powder particles were analyzed in detail.
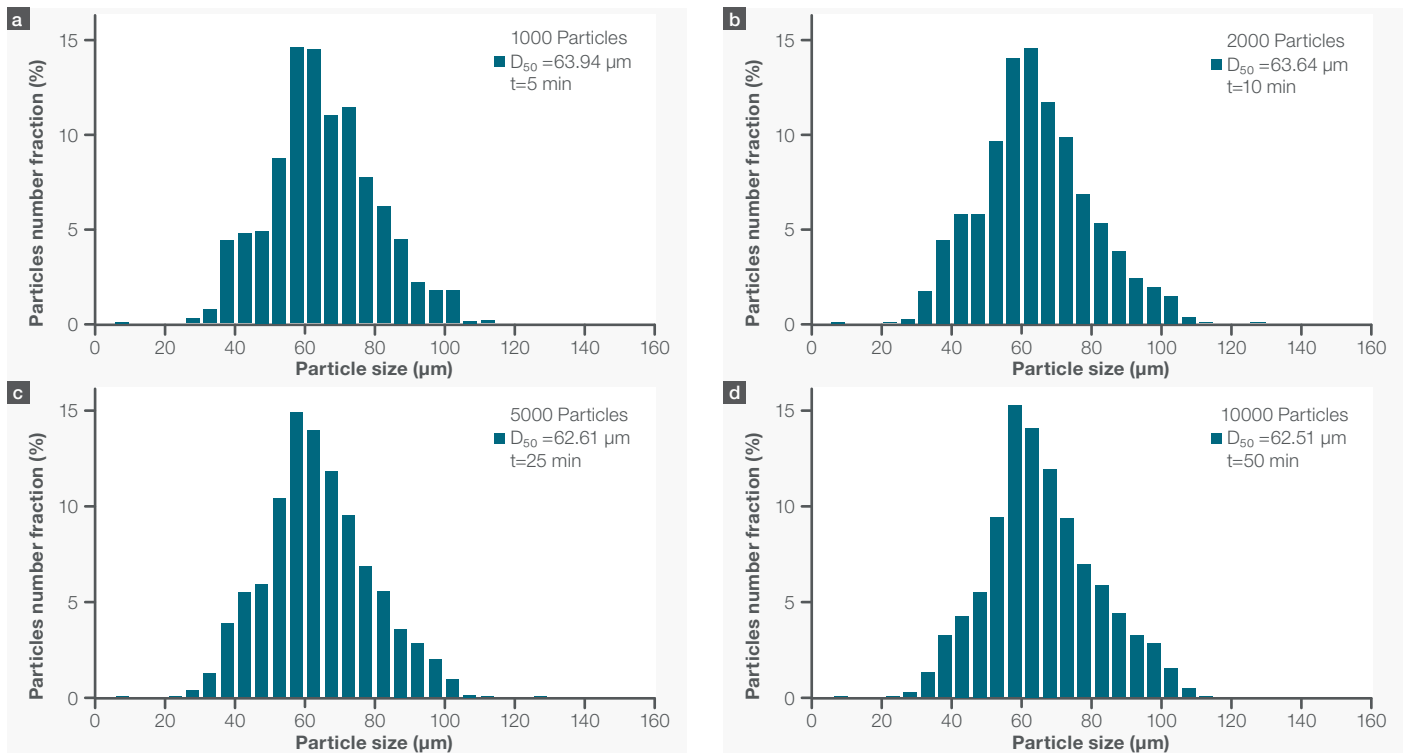
Figure 3. Particle size distributions measured from Supplier B Ti-6Al-4V powder using 1,000 particles (a), 2,000 particles (b), 5,000 particles (c), and 10,000 particles (d).

Examples of automatically measured powder particle size distributions are shown in Figures 2 and 3. For the two size distributions shown in Figure 2, the software analyzed approximately 4,000 particles for each sample, corresponding to an analysis time of 20 minutes per sample. The two size distributions differ in the number of particles with sizes less than about 30 μm: the powder from Supplier A contains a large number of sub-30 μm particles, while the powder from Supplier B reveals a cut-off particle size of approximately 25 μm. The measurement time required to obtain the distributions can be reduced by limiting the particle count. Figure 3 shows the effects of particle count on the size distribution for counts of 1,000 to 10,000 particles from the same sample. The distributions show very little change for counts of more than 2,000 particles. The median particle size, in fact, only changed by approximately 1 micrometer upon extending the measurement time from 10 minutes to 50 minutes (i.e. from 2,000 particles to 10,000 particles). These measurements highlight the potential of Perception Software to analyze powder particles for size distribution and by chemistry with measurement times on the order of 10 minutes.

Defects

After parts are made with additive manufacturing, the focus of the analysis shifts from the powder pedigree to the microstructures and defects in the additively manufactured parts. Some defects, such as unmelted regions, are typically on the order of micrometers to hundreds of micrometers and can thus be identified even with light microscopy.

Smaller defects, such as pores or inclusions, are detectable only with electron microscopy. Perception Software and its automated measurement capabilities are well suited to generate defect maps and size distributions over regions with dimensions of centimeters. Figure 4a is a photograph of an additively manufactured test sample. Figure 4b is an image from a cross-section through the test sample showing an unmelted region next to a spherical inclusion. Within the elongated, unmelted region, individual round powder particles can still be discerned. The cause of these defects is certainly of interest, but equally relevant is their distribution within the sample and their size distribution. Since the spherical inclusion differs in chemistry from the matrix and the unmelted region, the chemistry-based filtering mode of Perception Software allows for an analysis of size distributions only of inclusions.

An example of the software's capability to sample macroscopic regions for defects is shown in Figure 5. This is an analysis of large-scale cross-sectional areas for the additively manufactured test sample shown in Figure 4a. The 1 mm × 1 mm square area (colored pink in Figure 5a) resulted in pore and inclusion maps that are shown in Figure 5b as individual and combined maps. The software allows macroscopic regions to be defined for analysis, and the instrument then divides these regions into smaller frames for measurement purposes.
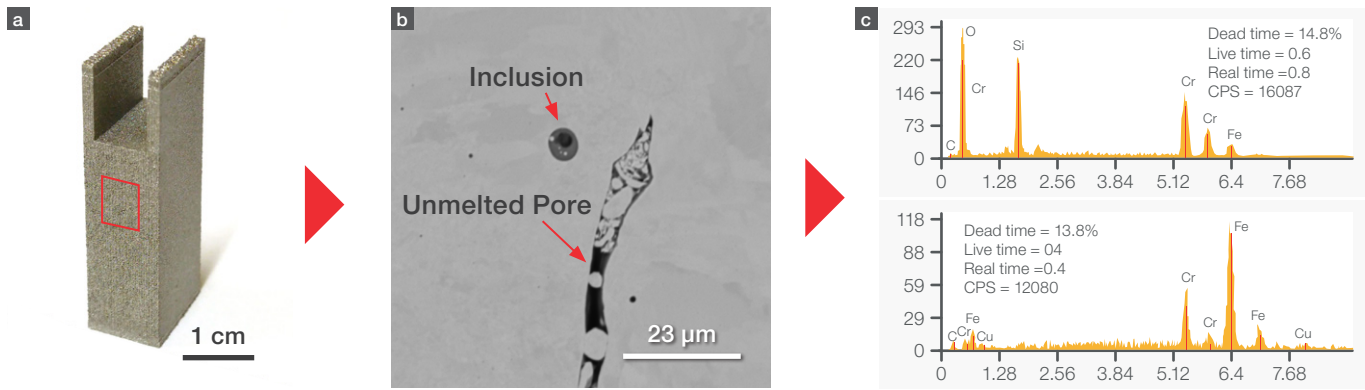
Figure 4. (a) Photograph of the AM as-built test part. (b) Backscattered electron image of a typical unmelted pore and an inclusion in the AM as-built test part. (c) Examples of EDS spectra collected on a silicon oxide inclusion (top) and unmelted pore (bottom) during automatic feature analysis.
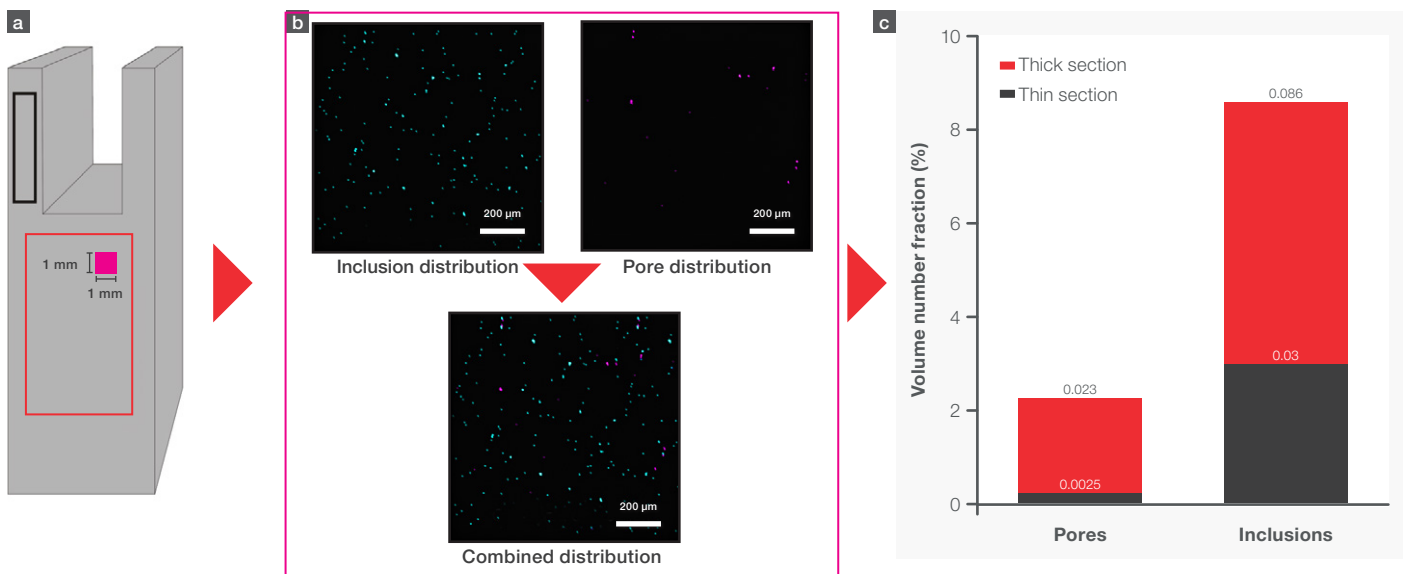

Figure 5. (a) Schematic illustration of an additively manufactured test sample with analysis regions (pink box, red and black rectangles). (b) Measurement results for inclusion and pore distribution and combined image for the 1 mm × 1 mm area highlighted in pink in (a). (c) Measured over all volume fractions of pores and inclusions in the thick and thin sections of the AM as-built part (shown with red and black rectangles, respectively, in (a)).

For example, an area of the main portion of the additively manufactured sample, shown with a black rectangle in Figure 5a, was analyzed for pore and inclusion volume fractions. The same analysis was applied to the thin section on top of the sample. The results of these measurements are shown in Figure 5c. For this particular sample, the pore volume fraction (taken as being equal to the area fraction, based on standard quantitative stereology methods) is 2.3% for the thick section and 0.25% for the thin section, while the fraction of inclusions is 8.6% for the thick section and 3% for the thin section.

Results

Perception Software's automated SEM-EDS particle analysis allows for quick analysis of powder particle size distributions and defect size distributions in approximately 10 minutes. Additionally, the software can analyze and differentiate based on chemical composition and morphologies. For additive manufacturing, key applications include the analysis of defects in microstructures of additively manufactured parts and powders.

Acknowledgments

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