HORIBA Scientific

Application Note

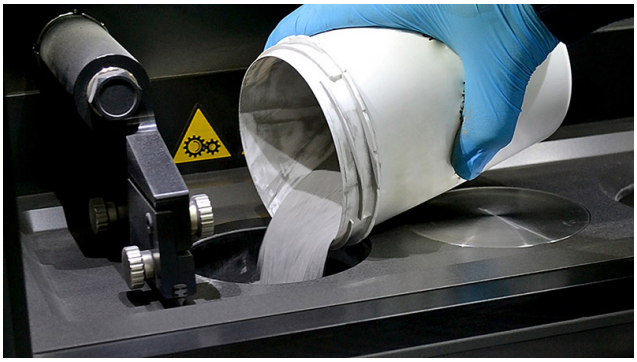
Identifying Trace Large Particle Impurities

AN216

Identifying Trace Large Particle Impurities by Image Analysis: Standard Addition Test

Introduction

Many manufacturing processes, including additive manufacturing, are sensitive to small amount of large particles. For example, these large particles can lead to voids or weak spots in the finished product. Simply determining the average or median particle size is insufficient for predicting manufacturing performance. The volume of particles that are larger than some limiting size must be carefully monitored. One can set a specification requiring that no more than small fraction of particles can be larger than a critical size. For example, one might require that no more than 0.01% of particles by volume are larger than 200 microns, that is 99.99% of particles are in specification.

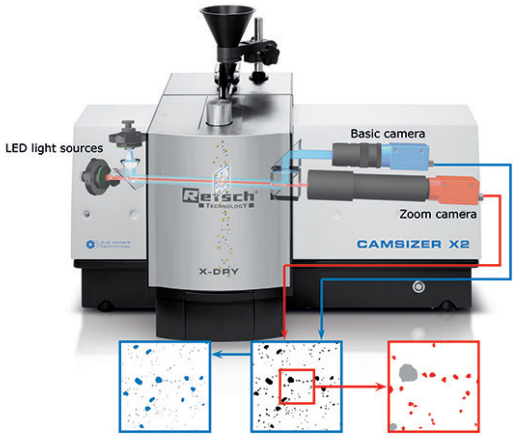


This low percentage presents a significant analytical and manufacturing challenge.

Image analysis is a proven technique for determining particle size and shape. By taking and analyzing photos of particles, many size and shape parameters for each particle can be determined. Image analysis has had three classic limitations: analysis effort, dynamic range, and speed. Recent advances have addressed these limits. Modern software and fast computers have shifted the analysis effort from highly skilled operators in front of a manual microscope to software that computes size and shape parameters from automatically acquired images.

The second problem is more fundamental. Microscopes and cameras only cover a limited size range. The resolution limit of the imaging system limits the smallest particles that can be analyzed. The field of view (which is coupled to the resolution either through camera size or lens distortions) limits the largest particles that can be analyzed. The advent of two camera technology in the CAMSIZER addresses this issue by significantly expanding this range with one camera and lens designed for large particles and a second camera and lens designed for small particles.

Finally, and germane to this discussion is system speed. Accuracy of size distribution results, particularly at the tails, or for trace analysis requires measuring a large number of particles. Dynamic image analysis where the particles flow in front of the high-speed cameras address this issue.



In this note, a series of metal samples with varying amounts of impurities were prepared gravimetrically and the resulting size distributions measured to illustrate how a high speed, dual camera system can be used to find small amounts of large particle impurities.

Methods

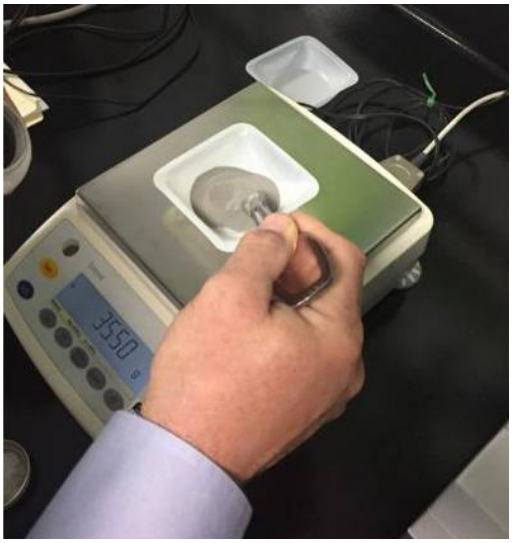


Figure 1: Weighing pure metal powder sample.

Figure 2: Weighing boat with impurity particles before mixing.

Metal Powder was sieved with an analytical sieve to ensure removal of trace large particle impurities. This powder was then weighed, and a small amount of large particle impurities was separately weighed before mixing with the pure metal powder. This gave a series of samples with known trace impurity mass concentrations. Spike concentrations were 0.005%, 0.01%, 0.02%, 0.05%, 0.1%, 0.2%, and 1% by mass. Sample masses were approximately 15 - 30 grams.

Samples were then analyzed with the CAMSIZER X2 dynamic image analysis system. This system features two high-speed cameras for a wide dynamic size range, air dispersion to ensure single particles are analyzed, and fully accounts for all variations in measurement due to particle velocity. To confirm analysis, the system was configured to save images containing large particle impurities as an option.

Particle feed rate was automatically adjusted so that 0.1% of the image was covered with particles. This low percentage eliminates the chance of particles touching during analysis and automated feed control reduces issues with operator error. Air dispersion with a pressure of 20 kPa was used for these metal powders to ensure loose agglomerates were separated and individual particles were analyzed.

Particle images were analyzed automatically to determine the particle width $x_{c\ min}$. This parameter was chosen since it is ideal for correlating result data with sieves. In addition, particles were analyzed for x_{area} and $x_{Fe\ max}$. However, these values were not used in this discussion.

Results and Discussion

Figure 3 shows a typical image containing a large particle from the analyzer. Note that while most particles are the same size, there is one large particle, the target of this test. The vast majority of images did not contain the large particles.

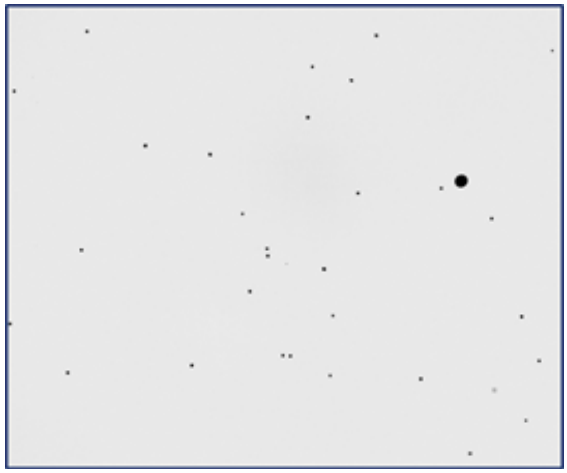


Figure 3: Typical image including a large particle impurity captured with the CAMSIZER X2.

The measured size distribution for the 1% and spike samples is shown in Figure 4a. Note that the 1% of impurities are difficult to see at full scale and the detail in Figure 4b shows the details of the oversize particle population. Figure 5a shows the measurement of 0.005% particles. Again, due to the small amount of impurities, the 0.005% spike cannot be seen in Figure 5a. Figure 5b shows the same data at higher magnification. Here, it is clear that the small amount of impurities has been resolved.

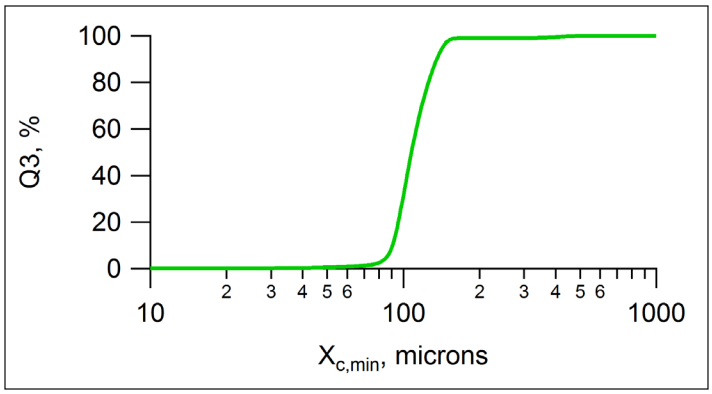


Figure 4a: Measured size distribution of 1% spike sample.

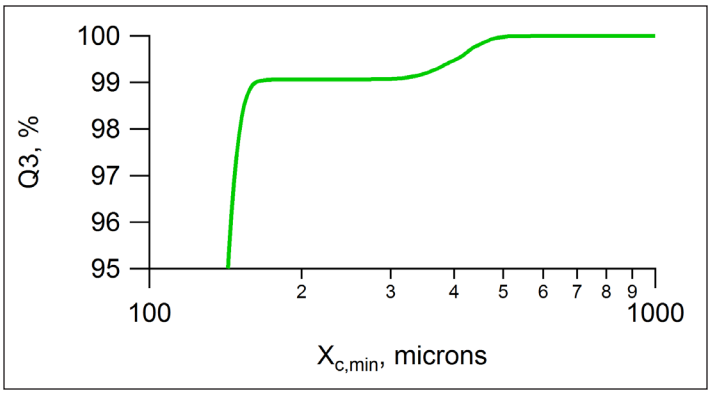


Figure 4b: Detail of size distribution showing fraction of large particle impurities.

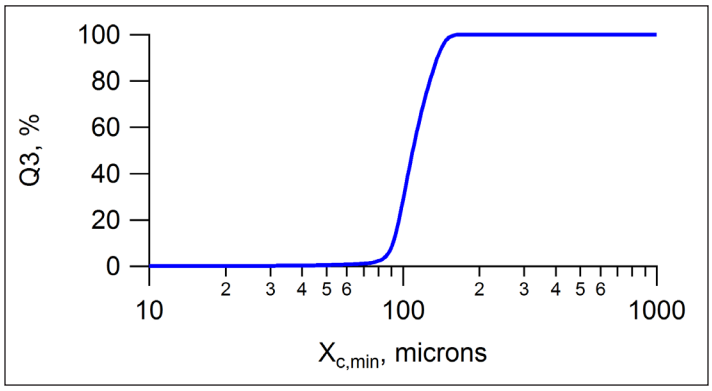


Figure 5a: Measured size distribution of 0.005% spike sample.

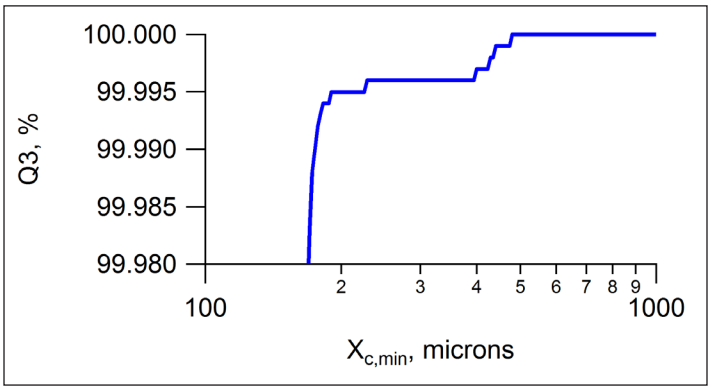


Figure 5b: Detail of size distribution showing fraction of large particle impurities.

Inspection of Figure 5b shows that the distribution has discontinuities and a flat spot rather than smoothly changing like in Figure 4b. These steps in the distribution arise because at this low impurity level, the number of measured particles is quite small, 47 particles. The internal data is carried in over 3000 size classes and the steps are due to missing particles, not due to the limited size classes in the analyzer. To illustrate this point, Figures 6 shows a further magnification of the data with points indicating the size classes exported for preparation of these graphs.

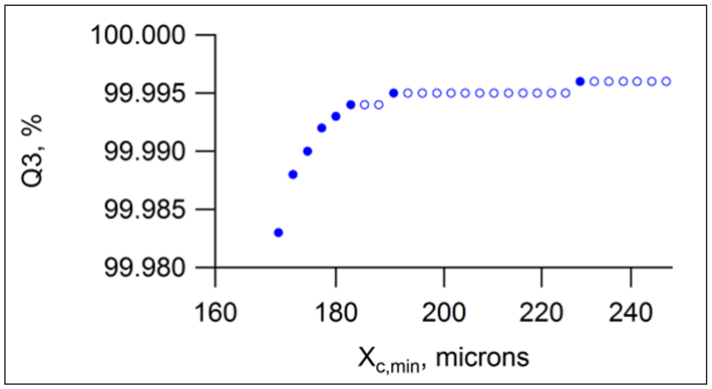


Figure 6: Detail showing data points for exported size classes for the 0.005% spike sample. Hollow points indicate size classes with no particle events. Filled points indicate classes with analyzed particles.

Figure 7 shows an example result table prepared with limited size classes for easy review by an analyst, in this case for the 0.005% spiked sample. Here, the rows were chosen so it is easy to identify the fraction of oversize material.

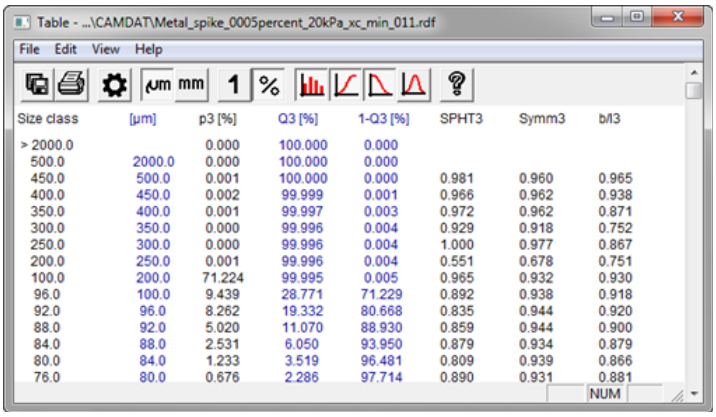


Figure 7: CAMSIZER X2 result table.

The data from this output was used to prepare Table 1, comparison of the fraction oversized particles determined by weighing before mixing with results from image analysis with the CAMSIZER. Here, it is clear that even with only 0.005% impurities, the impurity level can be readily discerned. Furthermore, the amount of identified oversize particles matches the gravimetric measurements of the amount of oversize particles added.

Table 1: Comparison of mass fraction oversize particles determined gravimetrically, and mass fraction oversize particles determined with the CAMSIZER X2.

% oversize prepared by weighing (over 200 micron)	% oversize determined by CAMSIZER X2 (over 200 micron)	difference
0.005 %	0.004 %	0.001 %
0.010 %	0.013 %	0.003 %
0.020 %	0.019 %	0.001 %
0.050 %	0.054 %	0.004 %
0.100 %	0.107 %	0.007 %
0.200 %	0.201 %	0.001 %
1.000 %	0.936 %	0.064 %

It is clear from the table that the impurity levels were measured reliably and with high precision.

Conclusions

The high speed and air dispersion of the CAMSIZER X2 can be used to identify trace amounts of large particle impurities with mass concentrations as low as 0.005%. This technique allows very tight specifications of raw material and corresponding improvement in manufacturing quality and part reliability.