MICROTRAC
PARTICLE CHARACTERIZATION

Advantages of Combining Laser Diffraction and Dynamic Image Analysis in a Single Instrument

Instrument: SYNC

Introduction

Microtrac launched the first commercial laser diffraction analyzers for particle size measurement in the early 1970s. Since then, this method has become the de facto standard for quality control in many industries, and only sieve analysis enjoys wider use for routine determination of particle size distribution. Laser diffraction impresses with its ease of use, robustness, versatility, and high sample throughput combined with short analysis times. However, like any method, laser diffraction also has drawbacks. In order to mitigate these drawbacks, adding an additional technique to complement laser diffraction was necessary. This is where imaging methods come in handy, as the weaknesses of one method are the strengths of the other. For this reason, there have been increasing efforts to combine laser diffraction and image analysis in a single measurement device. Microtrac has pioneered a unique hybrid instrument, the SYNC, which measures particle size and shape simultaneously in a singular measuring cell using laser diffraction and dynamic image analysis - for dry materials as well as suspensions and emulsions. (Fig. 1).

This article will expand upon the advantages of combining laser diffraction and dynamic image analysis using several industry-related application examples.

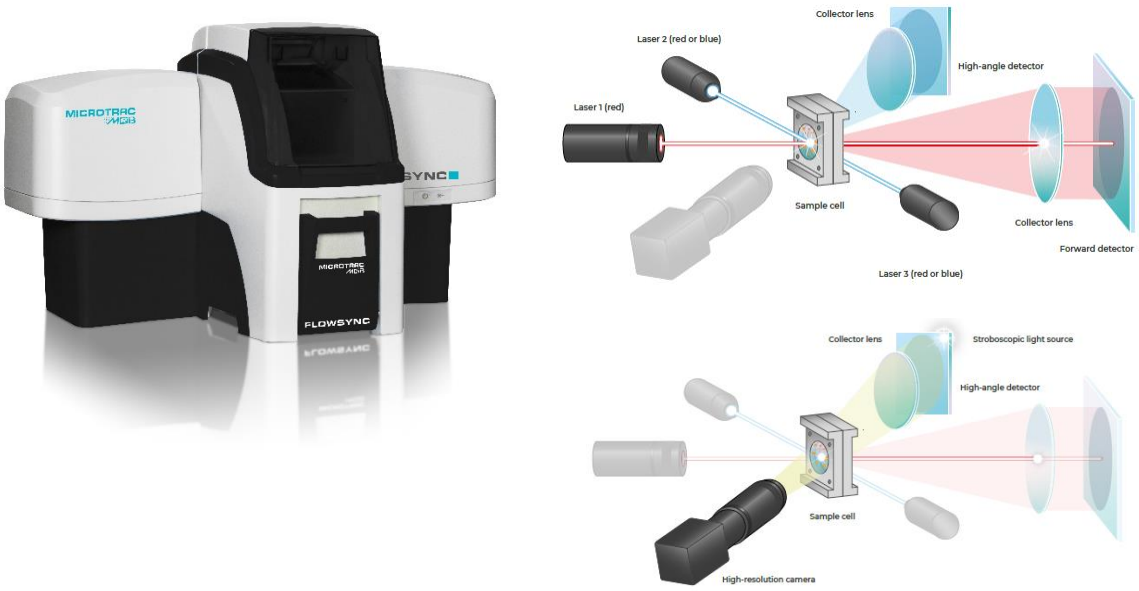
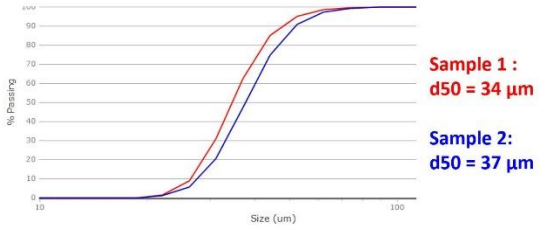


Fig. 1: *The Microtrac SYNC uniquely combines laser diffraction (top right) with dynamic image analysis in the same measurement cell (bottom right).*

Example 1: Quality of Metal Powders for Additive Manufacturing

Due to production conditions, particles can be fused together in gas atomized metal powders and deviate from the desired spherical shape. In additive manufacturing, such defective particles are problematic because they have a negative effect on the flow and sintering behavior of the powder. Although aggregates of several spherical particles are much larger than single particles and can be removed by sieving, this does not work for elongated, drop-shaped particles or so-called satellites. Satellites are small particles that adhere to larger particles, which cannot be removed by sieving because the diameter of the larger sphere determines the sieve diameter by which the total particle is retained.
Fig. 2 shows the analysis of two spherical metal powders with laser diffraction. The median values are very similar at 34 µm and 37 µm, respectively, as is the distribution width. Thus, it could be assumed that both samples are qualitatively comparable. However, when evaluating the simultaneously acquired images, it very quickly becomes apparent that this is not the case! The images in Fig. 2b show predominantly round particles in sample 1 and many satellites, as well as elongated or deformed grains in sample 2. Shown on the scattergram is the particle size (diameter in µm) and the sphericity, which is a measure of how closely the particle resembles a perfect sphere (sphericity = 1.0). Each point corresponds to a particle projection. For sample 1, the sphericity values are consistently close to 1. Sample 2 shows a wide scatter. Without image analysis, this important information would not have been available!



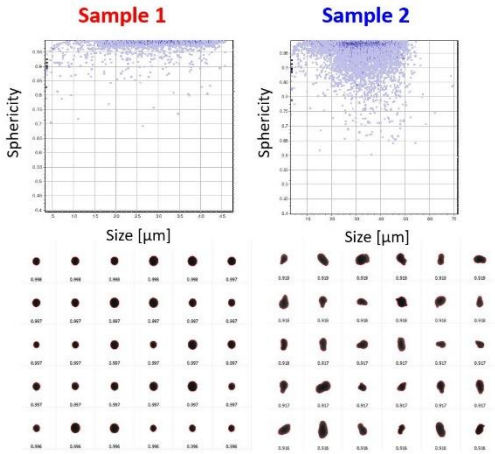


Fig. 2a: *Laser diffraction measurement results of two metal powder samples. The size distribution is almost identical, however, the image evaluation (**Fig. 2b, right**) shows clear differences in the particle shape.*

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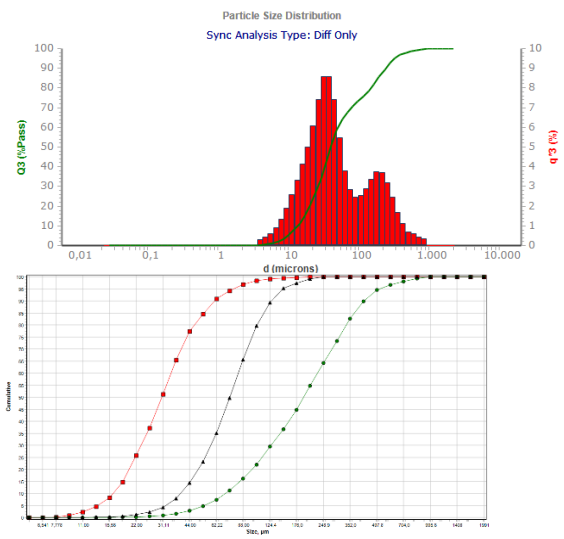
Example 2: Acicular (needle-shaped) Crystals

When evaluating particle size measurement with laser diffraction, spherical particles are always assumed. This means that all diffraction signals are treated as if they were generated by spherical particles - the particle shape cannot be detected. However, this limited evaluation model does not correspond to the geometry of most real samples. In many cases, extremely plate-like (e.g., minerals of the mica group) or needle-shaped crystals occur, as in the case of the mineral wollastonite, a calcium silicate. Many powdered pharmaceutical ingredients are also needle-shaped. Over the course of data acquisition, these particles pass through the laser beam in different orientations. Depending on the orientation of the particle, the diffraction signal can reach the detector from the long side or the short side. In the first case, the evaluation interprets the signal as a "large particle", in the second case as a "small particle". Consequently, a bimodal distribution is output, where the first maximum of the distribution tends to correspond to the width and the second maximum tends to correspond to the length. Without knowledge of the particle shape, the user will interpret this result as a mixture of two differently sized components (Fig. 3, top left). This misinterpretation can be prevented very easily by simultaneous image analysis. Here, the acicular geometry of the particles is obvious and even the aspect ratio (width-to-length) can be determined (Fig. 3 top right). In addition, separate distributions for the length and width of the crystals can be generated (Fig. 3 bottom).



	Diffraction	Width	Da	Length
D10 [µm]	14.8	15.5	39.1	69,5
D50 [µm]	37.5	29.8	73.5	193.8
D90 [µm]	225.0	59.9	125.6	418.8

Fig. 3: *Laser diffraction analysis of wollastonite shows a bimodal curve (top left). Particle images of wollastonite crystals (top right). The image analysis (bottom left): width distribution (red), length distribution (green), and distribution based on circle-equivalent diameter of particle projections Da (black). The numerical values of the percentiles d10/d50/d90 are shown in the table (bottom, right).*

Example 3: Detection of Oversize Particles

Oversize is defined as a small amount of particles that are larger than the rest of the sample and often outside the specification of a product. In many areas, the absence of oversize particles is an important quality criterion, e.g. for abrasives, where excessively large particles would leave scratches on the machined surface.
Classical laser diffraction is only of limited suitability for determining oversize particles. This is because an angle-dependent diffraction signal is evaluated, which is generated by all particles simultaneously (ensemble measurement method). Large particles scatter light at smaller angles, but a certain minimum amount of these particles must be present for the signal-to-noise ratio at the corresponding detector elements to be large enough to be considered for the result. Modern laser diffraction analyzers can reliably detect oversize particles above a few percent. However, this is not sufficient for many applications. Since image analysis detects and evaluates individual particles, i.e. each particle projection generates a recorded measurement signal, the sensitivity for oversize particle detection is significantly increased. During evaluation, the result of image analysis can be combined with that of laser diffraction to produce a particle size distribution. This is shown in the example in Fig. 4.

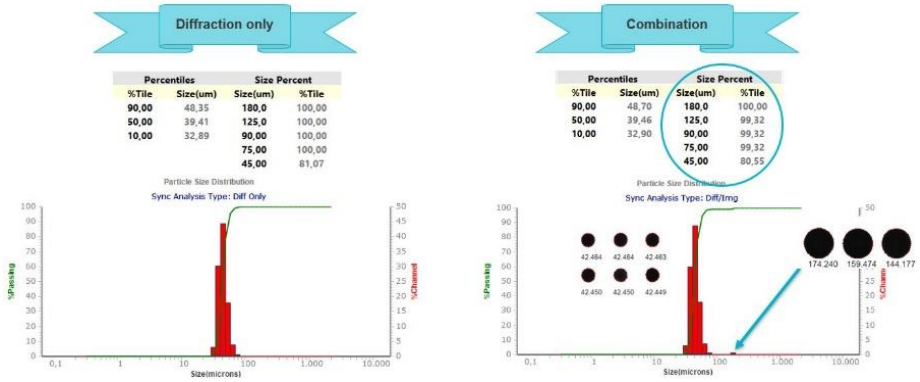


Fig. 4: *0.6% oversize particles were added to a sample of glass spheres. This is not detected in the diffraction analysis (diagram on the left). Only by combination with the image analysis the oversize material can be detected (right). In addition, the large particles can be detected on the images.*

Another example in Fig. 5 shows the particle size distribution of a polymer suspension with an average particle size of 170 nm. With such small particles, one has to rely on laser diffraction. By combining this with dynamic image analysis, it is not only possible to reliably detect the oversize particles, but it is also very easy to macroscopically identify whether these are sample particles, contaminants, air bubbles, or entrained materials. The result in Fig. 5 was determined by diffraction analysis and shows approx. 3 % oversize particles with a reported size of approx. 10 µm. These particles can be detected by the images and evaluated separately if required. In addition, a very small

proportion of particles > 40 µm is observed (bottom line of the particle images), which the diffraction analysis does not detect.

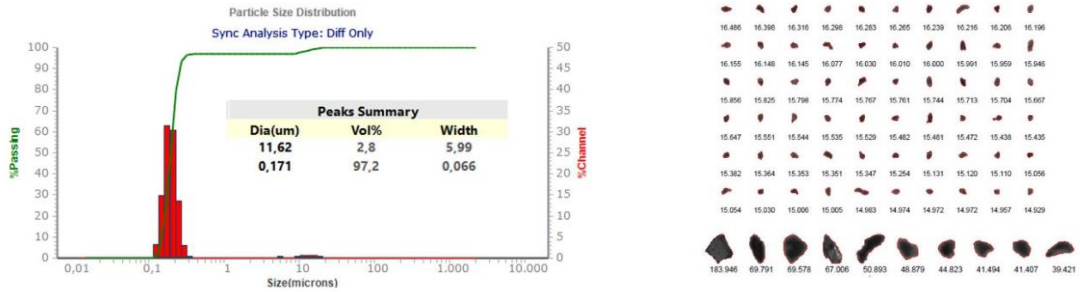


Fig. 5: *Laser diffraction result of a polymer suspension with oversize content (left). Particle images of oversize material (right).*

Example 4: Mixtures and Component Ratio Determination

Since laser diffraction evaluates a signal generated by all particles simultaneously and scattering signals at each detector come from particles of different sizes, it is often challenging to analyze mixtures. This is especially apparent when the sizes are close together. Due to the high resolution of the camera and the analysis of single particles, digital image analysis is clearly superior here. The example in Fig. 6 shows a result where mixture analysis with laser diffraction still works well. From a sand sample with a size distribution of 2 µm - 500 µm, the fraction 56 µm - 90 µm was removed by sieving. Coarse and fine fractions were mixed in a 1:1 ratio and analyzed with the SYNC. Note that the evaluation is based on laser diffraction alone. In the result, the two components are clearly separated and the ratio is correctly reproduced with 48.6 % / 51.4 %.

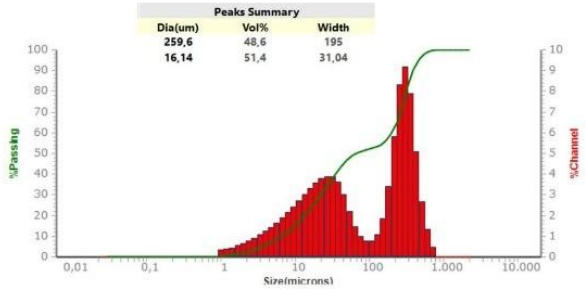


Fig. 6: *The mixing ratio for a 1:1 mixture of fine and coarse sand is determined with great accuracy.*

Another measurement example (Fig. 7) shows results of a trimodal mixture of glass beads (3 components with clearly separated particle size). The sizes of the individual components produced by sieving are < 45 µm, 75 µm-90 µm, and > 125 µm. When evaluated by laser diffraction, only a bimodal distribution is output. By the combined evaluation of laser data and image information, the trimodality is already indicated. In the analysis of the pure imaging data, the components are cleanly separated according to size. In addition, there is no overlap, as would be expected from the sample preparation. Nevertheless, the relevant parameters often specified in particle size measurement technology, the percentiles d10, d50, and d90, are still highly comparable for all three evaluations (only the d50 of the diffraction analysis is approx. 20 µm bigger).

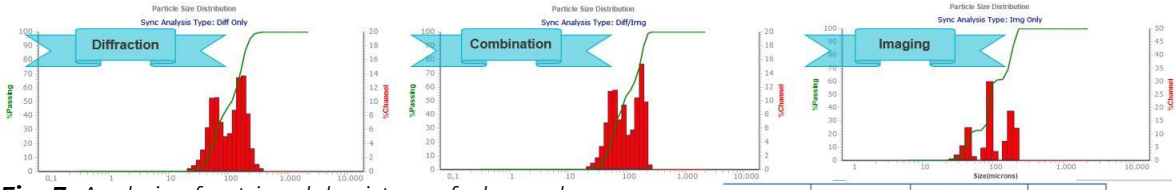


Fig. 7: *Analysis of a trimodal mixture of glass spheres. Analysis with laser diffraction (left), image analysis (right) and a combination of both (center). The table shows the characteristic percentiles d10, d50, and d90.*

	Diffraction	Combination	Imaging
D10 [µm]	41	41	38
D50 [µm]	101	83	83
D90 [µm]	185	177	180

Example 5: Comparability to Sieving

In sieve analysis, particles are retained by meshes corresponding to their smallest projection area. For non-spherical particles, this tends to correlate with their width. Thus, sieve analysis always measures particles in a preferred orientation. Since laser diffraction cannot distinguish between the length and width of the particle, the result will account for all orientations of the particles, which tends to push the coarse end of the distribution to larger sizes compared with sieve analysis. Especially samples that have been "screened" at a certain size still show larger particles in the laser result than expected based on the chosen screen, which can lead to confusion for many users. Image analysis helps to interpret the laser diffraction result correctly. Firstly, it is possible to output a distribution that is based on width measurement and is thus closer to the sieve analysis. Secondly, by evaluating the particle geometry and displaying the particle projections, the laser result becomes understandable and interpretable.

Fig. 8 shows the result of a laser diffraction measurement of a polymer powder. The sample was sieved at 100 µm, so no larger particles should be present. In the distribution, however, 10 % are larger than 100 µm, and even particles up to 500 µm are detected. The scattergram shows that there are many particles longer than 100 µm (up to 500 µm), but hardly any are wider than 100 µm, and if so, then only by max. 30 µm. The non-spherical particles of this sample orient themselves on the sieve in such a way that they pass through the meshes in width order and are thus characterized as < 100 µm.

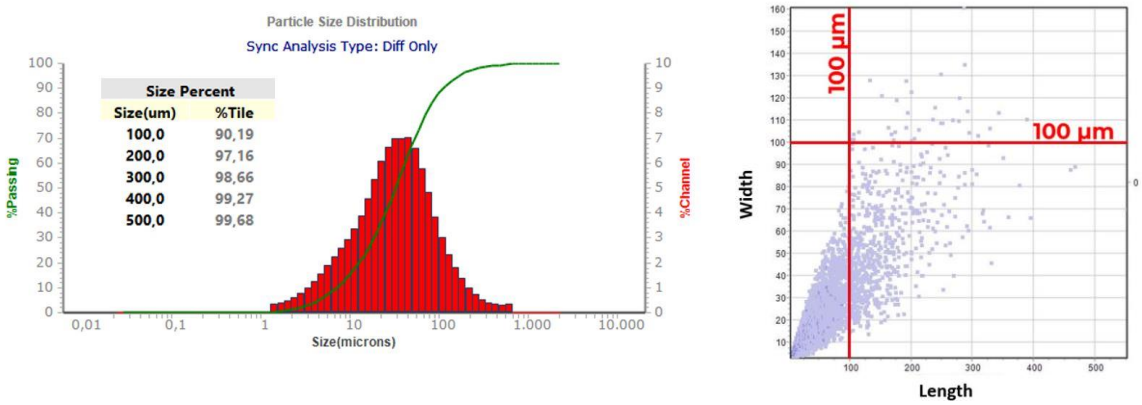


Fig. 8: *Laser diffraction result of a polymer powder sample. Although sieving was performed at 100 µm, larger particles are present in the distribution. The scattergram shows that this is due to the length of the particles being significantly larger than 100 µm.*

Summary: Laser Diffraction and Dynamic Image Analysis complement each other

Dynamic image analysis is perfectly suited to compensate for the known limitations of laser diffraction and to obtain more meaningful measurement results. As a hybrid instrument, the Microtrac SYNC particle analyzer uniquely combines both methods, with both analyses taking place simultaneously in the same measuring cell. The modular dispersion design of the instrument allows measurement of dry materials as well as suspensions and emulsions.

The displayed result can be based on classical laser diffraction, image analysis or a combination of both. Scientists benefit from the advantages of both techniques: the versatility, robustness, and wide measurement range of laser diffraction as well as shape analysis, sensitivity, and resolution of image evaluation.

	Laser Diffraction	Image Analysis
Shape Analysis	**No**	**Yes**
Measurement < 1 µm	**Yes**	**No**
Mixtures and Oversize	**With limitations**	**Very good**
Versatility	**High**	**Limited to larger particles**

Simultaneous laser diffraction and dynamic image anaysis with the Microtrac Sync: Unbeatable

For further information please contact us at: info@microtrac.com

More about the SYNC

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