APPLICATION NOTE

Titanium Dioxide – Documentation of the grinding progress in a Retsch PM200 with laser diffraction analysis

Instrument: SYNC

Titanium Dioxide (TiO_2)

Titanium dioxide is one of the most important industrial minerals and is mainly used as a white pigment. It is also used as an additive in the food industry (E171) or as a UV blocker in sunscreens. The refractive index of titanium dioxide is very high at 2.65 and shows strong dispersion (wavelength dependence). The starting material for the production of titanium dioxide is usually the mineral ilmenite ($FeTiO_3$). In nature, titanium dioxide occurs in various modifications with different crystal structures. The most common modification is rutile, others are anatase and brookite, as well as riesite (rare, only in meteorite craters).

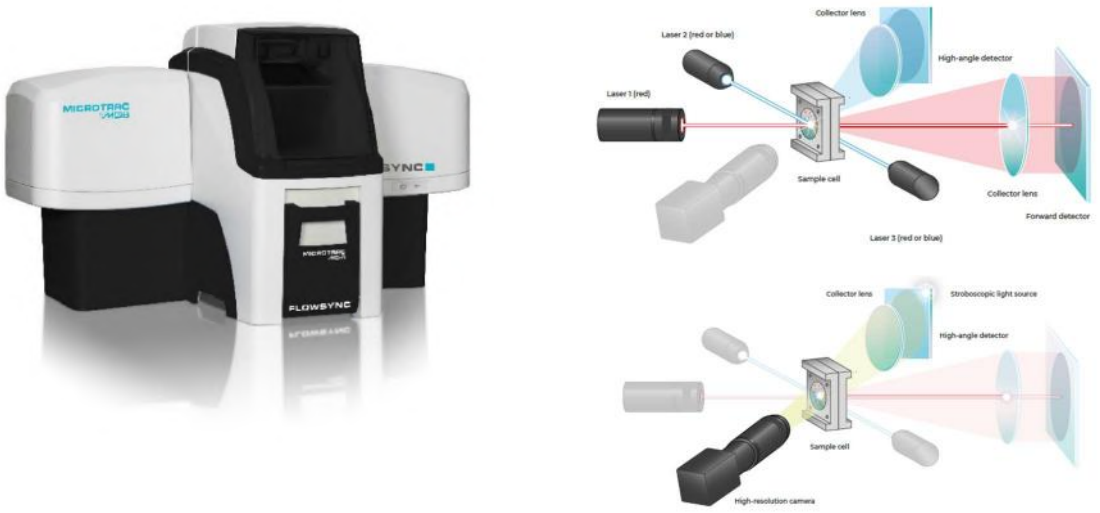
In this example, a titanium dioxide sample was ground using a Retsch planetary ball mill PM200. Wet grinding was performed in a sodium phosphate solution. The grinding progress was documented at defined time intervals via laser diffraction analysis with the Microtrac SYNC. The initial particle size was a d50 of 380 nm and a d90 of 650 nm. The goal was to achieve d90 smaller than 250 nm. Grinding can improve the product properties of titanium dioxide, and a particle size between 200 nm and 300 nm is ideal for use as a pigment.

Laser Diffraction Analyses with SYNC

With the SYNC analyzer, Microtrac MRB extends its established Tri-Laser technology with a powerful image analysis that offers users a new, unique measuring experience. The patented, synchronous measurement technology allows both methods to be performed simultaneously on one sample and in the same measurement cell:

- One sample
- One optical system
- One dispersion system
- One measuring cell
- One analysis

This makes the SYNC ideal for routine quality control applications. At the same time, it provides valuable information in research for the development of new materials and processes. The powerful software provides detailed size distribution and a wide range of morphological parameters. The patented BLEND function enables analyses over a wide measuring range from 0.01 to 4000 µm.



Microtrac's SYNC uniquely combines laser diffraction (top right) with dynamic image analysis (bottom right).

Milling Experiment

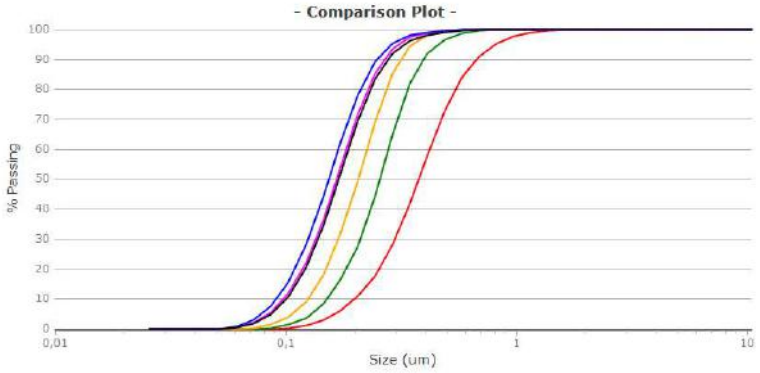
The grinding was carried out with the following settings:

- Retsch PM200 planetary ball mill (picture on the right).
- 125 ml grinding jar ZrO_2, 270 g grinding balls ZrO_2 0.1 mm
- 650 rpm
- Wet grinding in 30 ml sodium pyrophosphate solution (1%)
- 25 g sample
- Documentation of grinding progress after 5, 10, 30, 60, 120 minutes

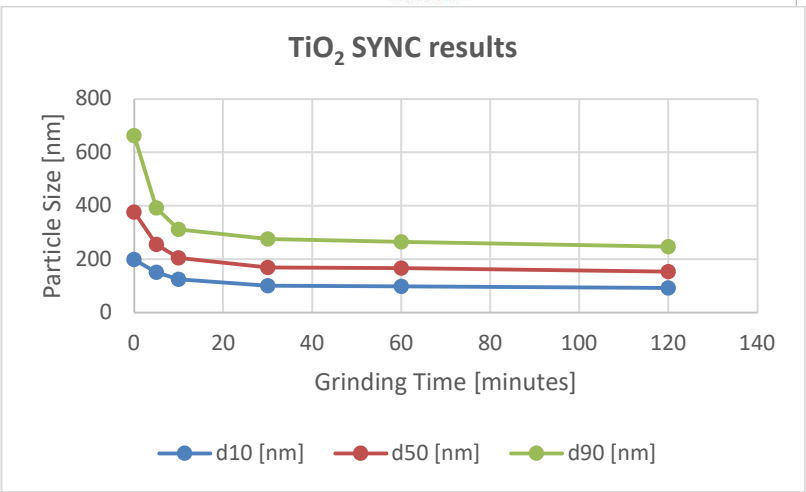
Measurement conditions laser diffraction:

- Instrument configuration 3R (three red lasers, 780 nm wavelength)
- Wet measurement in 0.2 % Sodium Phosphate solution. Phosphate solution prevents re-agglomeration of particles.
- Flow Rate: 45%
- 1 Minute ultrasound treatment
- Measurement time: 30 seconds
- Result calculation according to Mie-Theory, evaluation model for irregular particles
- Refractive index: 2.65

Laser Diffraction Results



Comparison of particle size distributions for different grinding time. The cumulative distribution Q3 is shown. Starting material (red), 5 minutes grinding time (green), 10 minutes grinding time (yellow). 30 minutes grinding time (black), 60 minutes grinding time (purple) and 120 minutes grinding time (blue).



Change of percentile values with increasing grinding time: d10 (blue), d50 (red) and d90 (green).

Grinding Time [min]	0	5	10	30	60	120
d10 [nm]	198	150	124	100	98	92
d50 [nm]	376	255	205	169	166	153
d90 [nm]	663	391	311	275	264	247

Table: Milling progress of the titanium dioxide sample. Shown are the percentile values d10., d50 and d90.

Summary

Laser diffraction is the method of choice for the documentation of the grinding progress of titanium dioxide. The method convinces by easy handling, high sample throughput and a wide measuring range. This makes the SYNC also suitable for characterization of other grinding processes. Thanks to the patented Tri-Laser geometry, highest accuracy is still guaranteed even for very small particles.
Alternatively, dry powders can be analyzed in addition to liquid samples. Through the simultaneous use of a camera, even the smallest amounts of coarse particles can still be reliably detected.

For further information please contact us at:

www.microtrac.com

