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Particle size determination and shape recognition of particles with the “analysette 22” NanoTec

The FRITSCH “analysette 22” NanoTec Laser-Particle-Sizer is the **first laser measuring device in the world** for the determination of **particle size distributions together with recognition of the particle shape in a single process!** A completely new sensor geometry and revolutionary software are the major innovative features of this **unique instrument**.

1. Particle size determination with the “analysette 22” NanoTec

The physical basis

Laser light falling on particles (powder, suspensions etc.) is diverted from its original direction by scattering. The angular distribution of the scattered light depends principally on the size of the particles, the laser wavelength and the refractive index. For particles that are smaller than about 1 μm , there are also important effects related to the polarisation of the laser: depending whether the plane of observation is perpendicular or parallel to the plane of polarisation, there are significant differences in the intensity distribution. This is shown in the polar diagrams in figure 1.

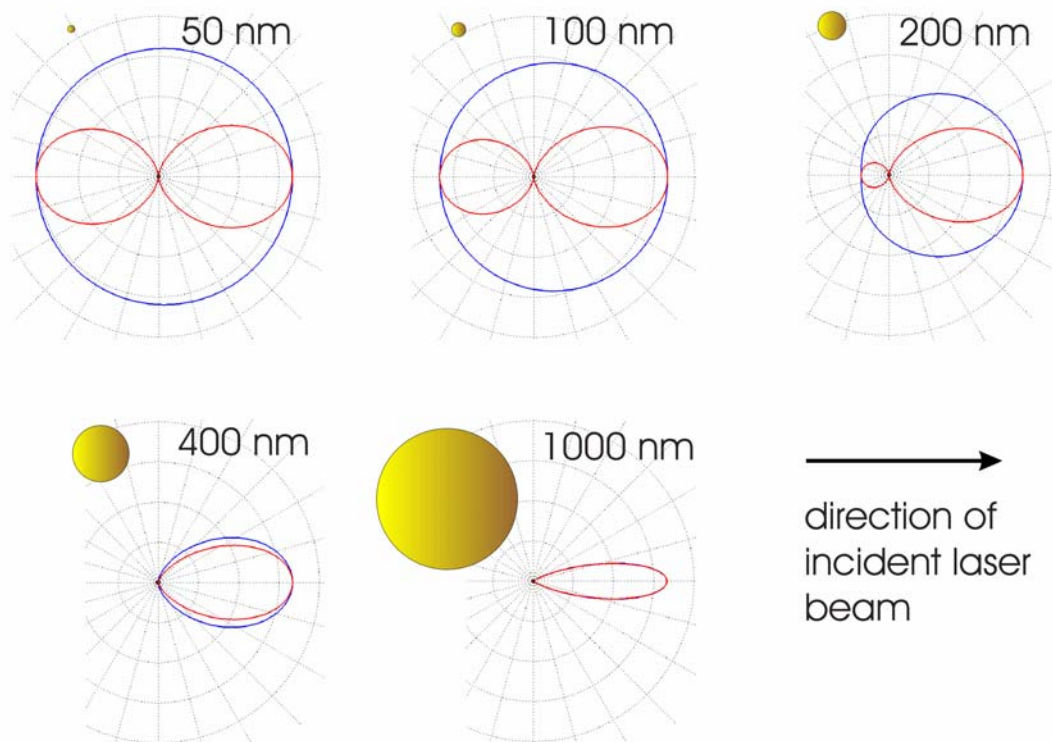


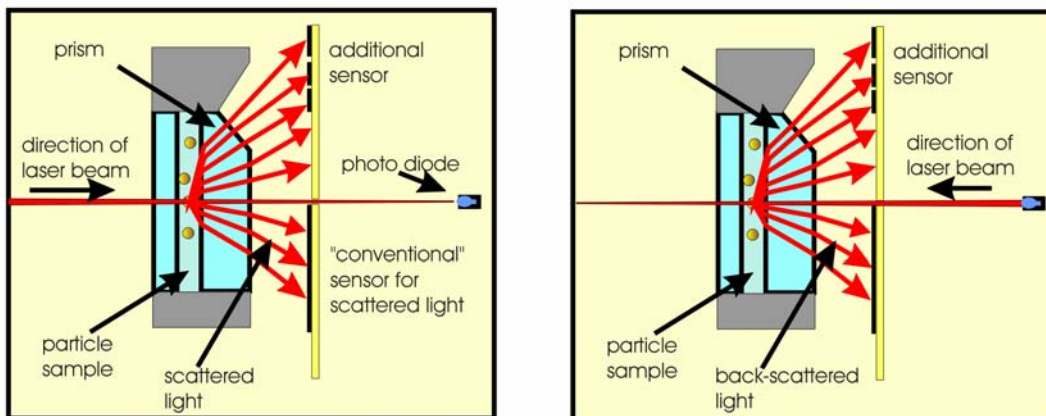
Figure 1. Angular distribution of the light scattered by small particles. The blue curve shows the intensity distribution perpendicular to the plane of polarisation, the red curve is parallel to the plane.

Laser diffraction instruments measure the angular distribution of the light from a particle sample. Using special algorithms, the particle size distribution can be calculated from the results. From the illustrations above, it can be seen what demands are placed on a system for characterising particles in the nanometre range:

- the measurement must cover as wide an angle as possible,
- the polarisation of the light is important; the scattered light must be measured perpendicular *and* parallel to the plane of polarisation of the laser.

The technical solution

The “analysette 22” NanoTec uses the proven *inverse Fourier construct*, developed and patented by the FRITSCH Company. The measuring cell with the particle sample is placed in the path of a focused laser beam. Displacing the measuring cell, changes the range of particle sizes that can be measured. Measurements in different positions are “assembled” to obtain a maximum dynamic range. For the fine range (particle size from 10 nm to a few microns), the measuring cell is only a few millimetres from the focus. The novel measuring principle is explained in figure 2.



"Normal" measurement using laser 1

Illumination from back side using laser 2

Figure 2: The particle sample is first illuminated with laser 1. The forward scattered light is detected by a conventional scattered light sensor. Light that is scattered at large angles is deflected by a prism onto the additional sensor. In step 2 of the measurement, the sample is illuminated from the opposite direction so that back-scattered light can be measured. Using a second combination of prism and additional sensor (not shown here), the scattered light can be measured both parallel and perpendicular to the plane of polarisation of the laser. In this way, the angular distribution of the scattered light can be fully characterised.

The advantages of the measuring method:

- wide particle-size range using reversed Fourier optics,
- no complex imaging optics to measure the scattered light,
- robust; no re-adjustment/calibration of the additional sensors required.

Even without the second measuring step shown in figure 2, the “analysette 22” NanoTec measures particle size distributions in the range of 100 nm to 1000 µm. With measuring step 2, the range is extended downwards as far as 10 nm. The particle size distribution is calculated using a highly efficient algorithm based on the solution of a Fredholm integral equation of the first kind.

2. Shape recognition of particles with the “analysette 22” NanoTec

The recognition of the shape of particles is possible on the basis of diffraction structures. The diffraction pattern created by the particles in the laser contains information about the shape of the particles. Different shapes of particles create different diffraction patterns in the laser beam. Information on the shape can be extracted from appropriate methods of signal processing.

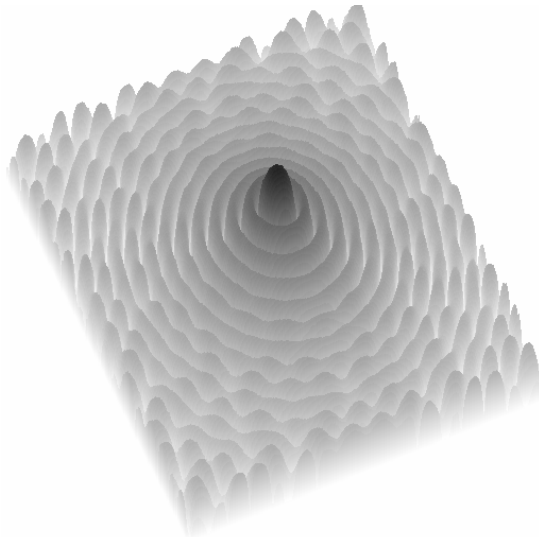


Figure 1: diffraction pattern of a sphere-shaped particle

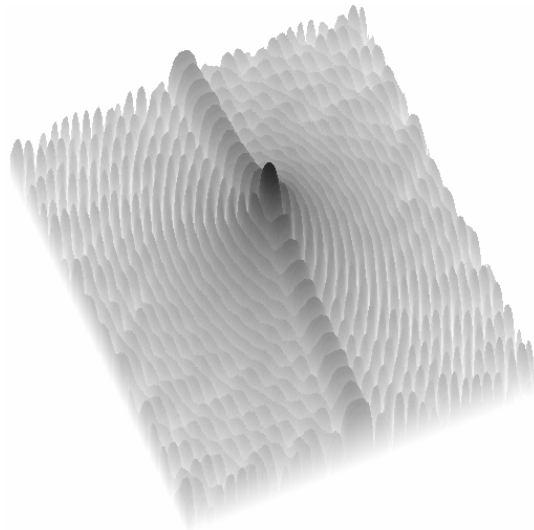


Figure 2: diffraction pattern of an ellipsoid particle

For real measurement, however, there are several particles in the volume to be measured with arbitrary alignment in the room and with varying particle sizes. The diffraction pattern resulting now contains combined information about the particle size, spatial arrangement and particle shape (*figure 3*).

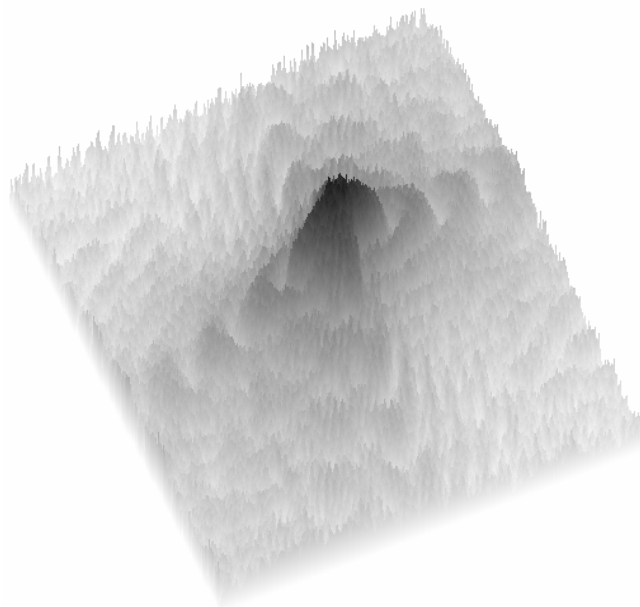
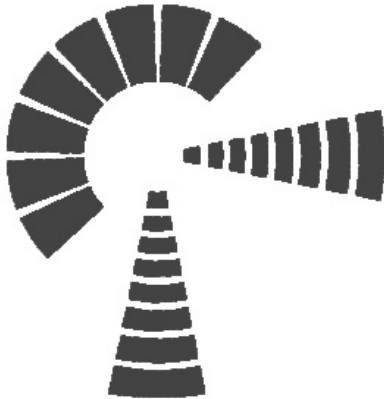


Figure 3: diffraction pattern of a particle collective

Using the new type of sensor (figure 4) it is possible to record the areas of the diffraction pattern in which the information about the shape of the particles is contained.



By use of neuronal network technologies, the sensor data are prepared in such a manner that a statistical evaluation is possible. The result of the measurement is the average elongation calculated from the axis relation of an ellipsoid approximating the particles.

Figure 4: drawing of the sensor arrangement

To evaluate the sensor data, a simulated neuronal back propagation network was trained and used. With this a calibration of shape is received which is to a large degree independent of the material which is displayed in the diagram (figure 5). Investigations have shown that the values measured, averaged over a certain number of measurements agree to a large extent with the desired values.

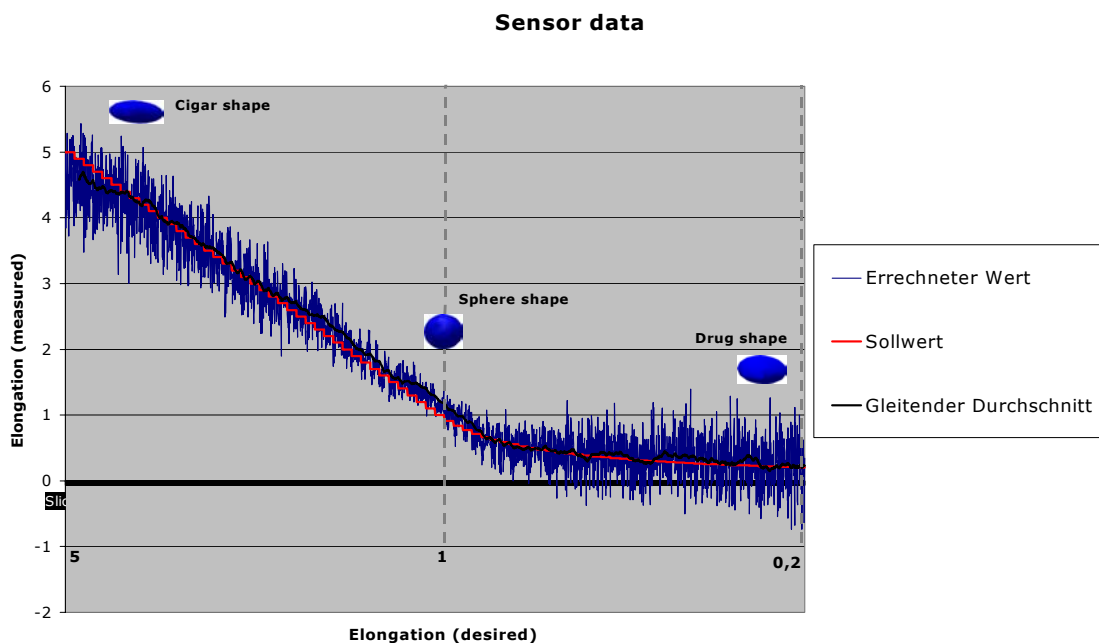


Figure 5: Comparison of prescribed particle elongation with sensor data measured