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THE APPLICATION OF THE SYMPATEC LASER DIFFRACTION SYSTEM IN THE RANGE 0.1 MICRON TO 2.6 MILLIMETRES.

1. INTRODUCTION

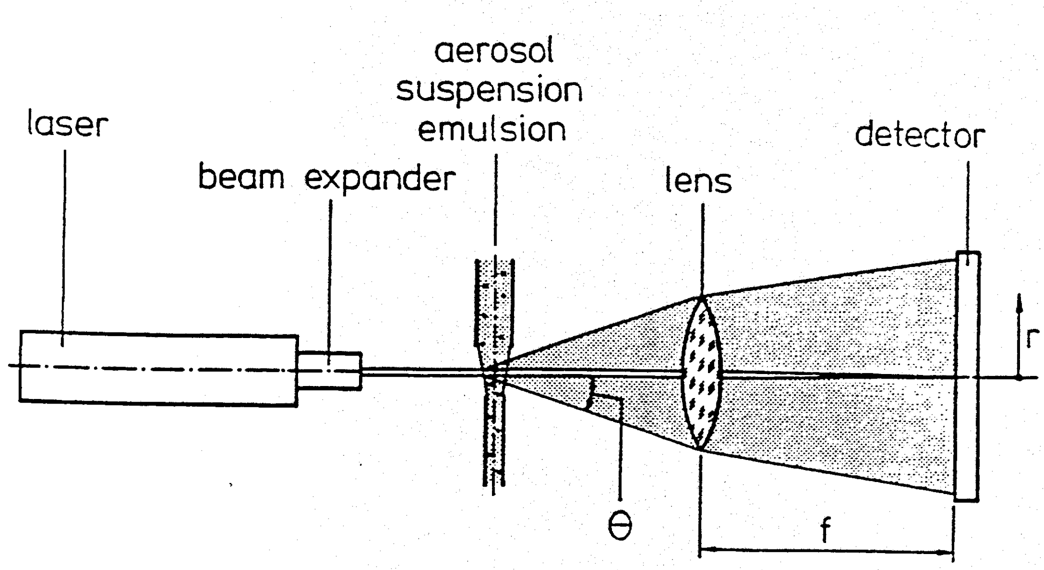
The development of laser diffraction technology over the last two decades has been a story of rapid development. During that time, this modern analytical technique has become the most important method for determining particle size distributions in the field of particle engineering. At present, laser diffraction is on the threshold of becoming the dominant standard method for Off-Line laboratory applications, whilst simultaneously offering, for the first time, the possibility of achieving On-Line particle size analysis.

The Sympatec HELOS/RODOS system was the first system which was capable of measuring particle size distributions using dry collectives of dispersed particles. It thereby offers an extremely broad measuring range, from 0.1 microns up to 2.6 millimetres. Moreover, it is the first laser diffraction system which offers a large selection of dispersion systems, both wet and dry, all of which are modular and therefore interchangeable.

This flexibility enables a change in philosophy to be achieved; that the system is now adaptable to suit the sample not the reverse. This represents a major turn-round in thinking and opens the way for many wider applications.

2.FUNDAMENTALS

As a general rule, diffraction spectrometers employ an optical arrangement such as that illustrated in figure 1.



Optical setup for the measurement of diffraction patterns

fig 1

The light beam, produced by a laser and widened with a beam expander passes the measuring zone where the particles to be analysed are properly dispersed in either a gas or liquid. The diffraction patterns are generated by introducing the particles into the laser beam in the focal plane of the lens, which is situated downstream of the measuring zone. The laser is focussed onto a special multi-element detector. If all the particles were the same size, a radial intensity distribution of high intensity in the centre of the laser beam and of low intensity in the outer elements would be produced (see figure 2).

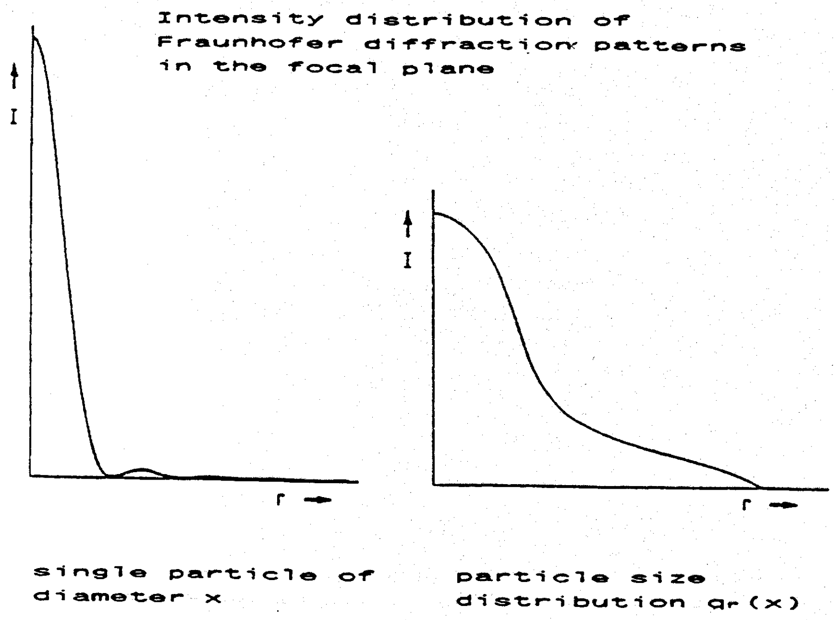


fig 2

The intensity distribution curve passes through numerous changes of high and low energies, which appear as concentric rings of light and dark zones getting weaker with increasing distance from the centre. The diffraction angle θ describes the location of the first minimum of intensity and is a measure of the geometrical dimension of the diffracting particles. A simplified explanation is that that diffraction angles θ_i are inversely proportional to the particle sizes which produce them.

Hence, small particles show large diffraction angles and expand the light to the extreme outer edge of the detector, whereas large particles which produce small diffraction angles are focussed near to the central, straight-through position.

In order to determine the shape of a particle size distribution, there are two factors which must be known:-

i) the equivalent diameter that describes the particle size

ii) the so-called "measure of quantity" eg. the number, length, area, volume or mass can all be used as measures of quantity

The Laser Diffraction principle uses Volume as the measure of quantity. The relative proportions of particles can be derived from the distribution of light intensities in the diffraction pattern. This is a complex derivation as particles of different size are simultaneously projecting diffracted light to every position in the focal plane.

By applying an expanded matrix calculation to this problem, a unique solution of the resulting system of equations can be found. A more detailed explanation of the mathematical solution is given in the the papers by Heuer and Leschonski (ref.1) in 1985 and also by Roethele, Naumann and Heuer (ref.2) in 1989.

The former describes the specific algorithms which Sympatec evaluated to solve the problem, whilst the latter explains the extension of the Fraunhofer diffraction principle to the submicron range. The main advantage of this unique approach over that used by other manufacturers, is that the solution of the equation system is parameter-free; ie. there is no requirement to know refractive indices or absorption coefficients for the materials to be analysed, to achieve the result.

The transformation of the measured light energy patterns into the resulting particle size distribution is achieved by applying a one-to-one mathematical filter where no information is suppressed, modified or added.

Thus multimodal particle size distributions can be measured and the resulting size distributions are not only representative of the multiple modes but also of their relative proportions, as seen in figure 3. This shows the distribution of multicomponent mixtures.

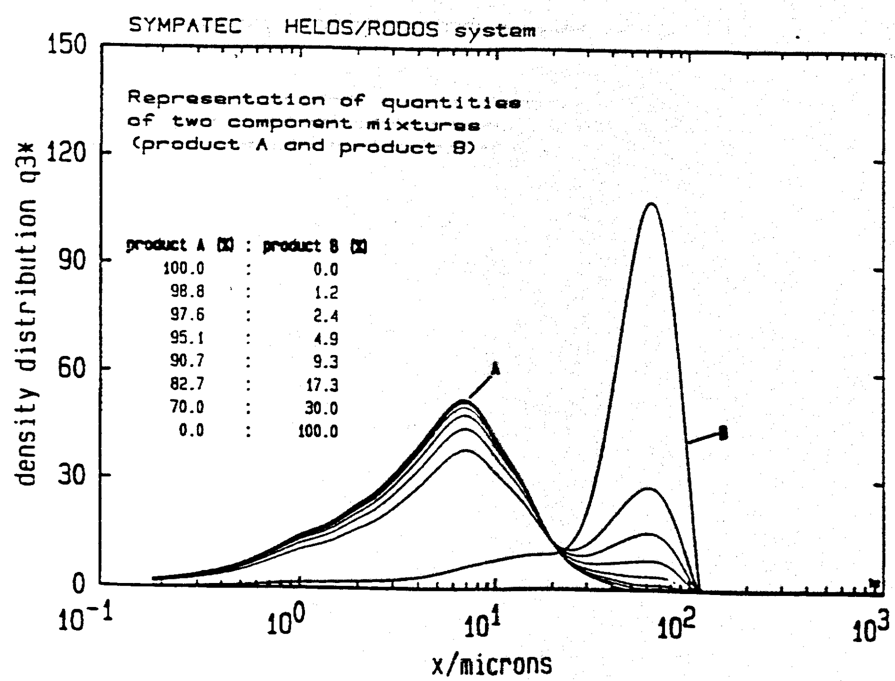
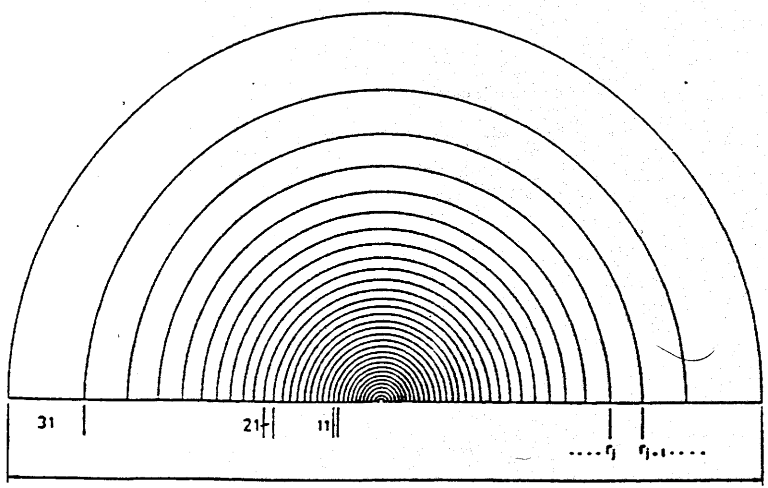


fig 3

Sympatec use a semicircular, multi-element detector with 31 rings as shown in the fig.4. The actual distribution of diffracted light energy is divided into 31 classes. The determination of the particle size distribution is consequently performed by the application of the previously mentioned parameter-free mathematical transformation with a 31 x 31 element matrix, and the results are presented in a tabular or graphical format, again in 31 classes.



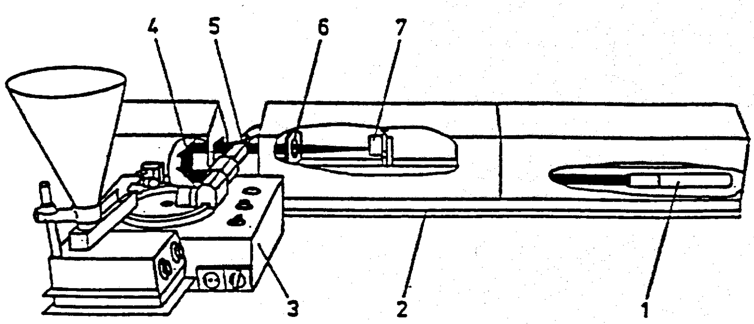
Multi-element detector
with 31 classes

fig 4

3. THE HELOS LASER DIFFRACTION ANALYSER

HELOS is an acronym for HELIUM-NEON LASER for OPTICAL SPECTROMETRY and is the central element on which the Sympatec laser diffraction system is based (see fig.5).

The cabinet of the Helos unit, which houses the optics and electronics, is made entirely of stainless steel. The standard models offer upto six different measuring ranges, determined by six lenses each used at its respective focal distance from the detector. The focal lengths can be from 20mm to 1000mm, giving the user the possibility of measuring particles from 0.1 to 1750 microns. Sympatec also manufacture special long-body models which can go up to 2625 microns.



Prospective view of HELOS/RODOS system

1 He-Ne-Laser with beam expander
2 optical bench system
3 dry dispersing system RODOS
4 deflection mirrors
5 sensing volume
6 collecting lens
7 multi-element-detector with autofocus-system

fig 5

The laser (fig.5, item 1) with the beam expander optics, is integrated in the optical bench (item 2) and is thus protected against shock. It is cooled by an integral fan. The laser beam, folded twice by two deflecting mirrors (item 4), then enters the measuring zone (item 5).

The diffraction patterns fall on the 31 channels of the detector system, each of which is simultaneously scanned at a rate of approximately 400 Hz. The Analogue-to-Digital conversion is of a very high resolution and once digitised, the data is transferred to the microcomputer via a fibre-optic link, where the transformation and calculation of the particle size distribution takes place.

Solid state mechanics and optics combine with the latest electronics and Sympatec's unique mathematical solution to give a new dimension in reliability and reproducibility when using the HELOS for particle size analysis.

4.DISPERSING SYSTEMS

In addition to being able to obtain a fast, repeatible, parameter-free result the true versatility of such a measuring system is governed by its ability to adapt to the variability of different samples and their associated problems. It is at this point that the Sympatec philosophy has developed. There are two main stratagems:-

i) that the measuring technique applied has to be as closely adapted as possible to match the product properties and the production process by which it is made.

ii) the successful application of the laser diffraction principle, in both laboratory and production situations, demands that dispersing systems must be available for both wet and dry media, as well as for systems in motion and at rest.

The combination of these two statements results in the matrix of dispersing systems - see fig.6. Here, the four dispersing systems are shown, all of which can be utilised with the Helos analyser.

Dispersing medium	in the measuring cross-section in motion	in the measuring cross-section at rest
dry	**RODOS**	**GRADIS**
wet	**SUCELL**	**CUVETTE**

Matrix of dispersing systems

fig 6

The RODOS is Sympatec's patented dosing and dispersing system and is the first such system to produce a truly deagglomerated particle jetstream of dry bulk material in air (or other inert gas), which works over the whole laser diffraction range ie. from below 1 micron to greater than 2 millimetres. It was the development of the RODOS which opened the way for applying the diffraction principle to fine dry powders.

GRADIS is a falling shaft disperser, again for dry materials. Using the influence of gravity, the particles are carefully dispersed under their own mass forces and transported through the measuring zone at low velocities. It is intended for less stable granular materials, and for coarser particles in the millimetre range, where dispersing forces are neither desired or necessary.

The SUCELL is the standard suspension dispersion system where all the functions, such as ultrasonics, stirrers to homogenise the sample and circulation by peristaltic pump are all included in one compact module.

The CUVETTE is a small volume, stirred cell manufactured of high quality optical glass, for the analysis of very small or toxic samples.

The modular concept and design of the HELOS system, all of the different dispersing systems are quickly and easily interchangeable thus giving the user the ability to adapt the system to suit his products now, and in the future.

5.RODOS DRY DISPERSION SYSTEM

Because of its uniqueness, it is worth taking a closer look at the RODOS dry dispersing system (see fig.7). It is a patented system for the dispersion of dry powders down to less than 1 micron.

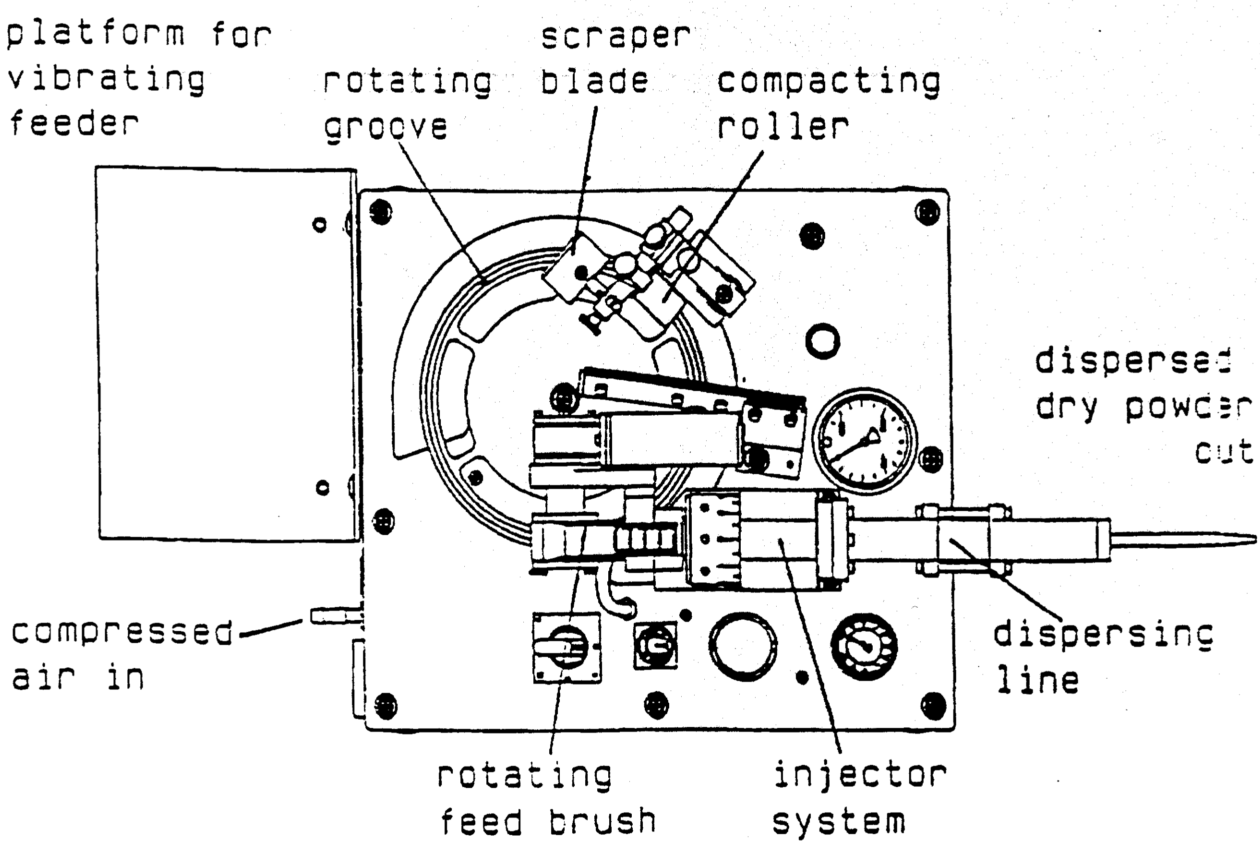


Figure 7: Feeding and dispersing unit RODOS

Its development resulted from the fundamental premise that dry powders should be analysed in their dry state. This approach has been adapted for two main reasons:-

i) it offers the possibility of very fast analysis

ii) it corresponds to the manufacturing process concerned and thus furnishes information on the primary particle size

The central unit supplying the energy for the dispersion mechanism is a gas-solid injector. By accelerating compressed air, or gas, to a high, but subsonic, velocity, a depression is produced at the rear of the injector. Agglomerated particles are transported into the high velocity zone.

The mechanisms which disperse the particles so effectively are frictional shear-forces between the gas and the particles, inter-particle collisions and specially designed wall-particle interactions.

In order to ensure a uniform and optimised dispersion, the injector must be fed with a constant mass flow-rate, with any fluctuations being minimised.

A standard, commercially available feeder is employed to feed an excessive amount of the bulk material to be analysed into the machined groove of the rotary table. The groove is rotated whilst being filled and any excess material is removed by the scraper blade. The next step is to compact the remaining material using the roller so that the groove is uniformly filled.

The rotating feed-brush transfers the solid material into the depression zone, which we described earlier, where it is aspirated and subsequently dispersed.

The free gas-solid aerosol passes through the measuring zone and the laser beam, where it is collected into the vacuum system. Here, the powder is separated out using a high performance gas cyclone and can be recovered if necessary. Only the very fine powder is collected in the bag of the vacuum cleaner.

Once the measurement is complete, the analysis takes only a few seconds, and the system can output the information in a large variety of user-selectable formats. In addition to this, Sympatec have developed complimentary software packages to perform analytical, statistical and graphical evaluation of data already measured, and to correlate data with other standardised methods of particle size analysis and standards. It is also possible to present data in linear, logarithmic, Rosin Rammler or standard distribution formats. Other features include the evaluation of grinding processes, the determination of corrected classifier efficiency curves, and the transfer of data to other microcomputers.

6. SUMMARY

The development of laser diffraction technology over the last 20 years, has ensured it's position as the dominant method for fast and precise determination of particle size distributions.

It's superiority has resulted directly from it's notable advantages:_

i) as a rapid measurement technique with a resolution previously unknown to industrial users.

ii) the measuring range over which diffraction can be employed is enormous (0.1 um to nearly 3mm) using one measuring technique.

iii) the ability to work in all size ranges without the need to know the optical parameters

The success of the Sympatec Helos systems can be attributed to the basic premise of which we spoke earlier; namely the the system should be adapted to suit the sample. This means that powders manufactured dry should be analysed dry, and powders manufactured wet should be measured in suspension. This is the key to the next important stage - the application of Fraunhofer diffraction to Automatic and On-Line systems.

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Title The Application of the Sympatec Laser Diffraction System

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