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Particle metrology

- the renaissance of the microscope

We have seen that in diagnostic testing the microscope has some stiff competition – but in the world of particle analysis it is still the daddy according to Graham Rideal

MICROSCOPY is one of the oldest methods of particles sizing, dating back to 1590 when two Dutch spectacle makers, Zacharias Janssen and his father Hans first developed the compound microscope. Microscopes were used initially as an identification tool rather than a method of quantitatively measuring particle sizes.

However, a combination of three very significant developments resulted in a stratospheric leap in the capability of today's microscope: the ability to produce high precision lenses, digital image capture and ultra-high speed computers. The great advantage of a microscope is that you can actually see the objects in question. Therein lies a problem – how do you describe an irregularly shaped object?

For example, if you were asked to describe your physical characteristics to an interested party, the equivalent spherical diameter would not be very helpful, but in particle size analysis, this is often exactly what we do. Height and weight give more information but even more detail is contained in the 'vital statistics', which attempts to describe the contour of the human body. A similar situation

exists in particle metrology.

Most particle sizing instruments measure an equivalent spherical diameter but the microscope is unique in being able to describe particle shape. Powder properties are an amalgam of many different factors, which uniquely combine to determine not only the performance of the constituent particles, but the behaviour of groups of particles when stored, transported or ultimately react in the final purpose for which they were designed. Particle size, particle size distribution and moisture content are well known for their ability to affect powder rheology, but particle shape is being increasingly recognised as an important parameter.

Counting fibres and measuring their length to thickness ratio (the aspect ratio) has been extremely important in the asbestos industry but manually counting and measuring sufficient particles to obtain statistically valid results has taken up to 8 hours to perform. A similar analysis today would take just a few minutes.

Without traceability to an international standard, any particle size analytical technique has little or no value because the results are open to be challenged.



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Microscopy is no exception and images without an associated magnification are meaningless in the field of particle size analysis.

The primary calibration parameter in digital imaging is the pixel size, so the pixel array of the camera must be geometrically certified as in some cases there may be a 10% difference in the X and Y dimensions. This can be done through the National Physical Laboratory particle graticule, which contains a 400 micron square grid subdivided into squares down to 23 microns.

Once the 'squareness' of the field has been established, the pixel calibration can be determined and particle measurement can begin. The NPL graticule also has a root 2 array of circles from 50 microns down to 2.5 microns. This is very useful in ensuring that a given calibration holds over the size range of the particles to be measured. A useful graticule for checking the reproducibility of the shape analysis is one based on a series of 200 different shaped images. This graticule also has a grid to confirm length and 'squareness' of the image captured.

One problem in the transition from manual counting to automatic counting is touching particles. The human eye is very good in identifying clusters of individual particles from a large 'fused' particle; a simple analogy is a string of beads. A necklace could be identified as one long particle or a multiple of the individual component. In automatic counting, touching particle should be measured and stored but overlooked in the final analysis in case they are physically attached in some way. If a slide is suspected of containing more than 20% of touching particles, it should be abandoned and a new one prepared.

In microscopy, sample selection and presentation are of paramount importance because such small quantities are required for analysis, typically a few milligrams. The single biggest source of error in any particle size analysis is taking a representative sample from a larger weight. It is quite common for particle segregation to occur during transit. Larger particles can settle to the bottom of a container, so sampling from the top would give a false answer. There are also cases where larger particles can 'float' to the top. The so called 'Brazil nut' effect is where Brazil nuts will end up on the top of a container when transported with rice.

One method of taking a representative sample is to make a thick paste with glycerol and take a small sample with a micro spatula, which is then further diluted on the microscope slide. Making glycerol pastes is not always possible, especially when analysing glass particles, which have a similar refractive index to glycerol and are therefore rendered 'invisible'. In such cases a dry dispersion technique is to be preferred.

The latest evolution of dry dispersion is in the Malvern image analyser, where a bursting disc principle is used to

impart a high impact, high velocity air current onto the dry powder, which then falls as individual particles onto a macro microscope stage (Figure 1).

Once a representative sample of individual particles has been prepared, the next question is how many particles should be counted? This could be anything from one to 10 million; the number being dependent on the particle size distribution of the powder.

For example, if the label had come off a ball bearing box, one measure would be enough to determine the size of the remainder in the box. But where size distributions of 10:1 or 100:1 need to be measured, much larger numbers of particles would need to be counted, especially if measurements at the extremes of the distribution are required. In practice, a useful guide is to continue counting until there is no further change in the particle size distribution at the percentiles of interest.

Uncertainty of measurement is something often overlooked but is of vital importance if one is to have confidence in the results. If two simultaneous results are 20% apart there is considerable uncertainty as to which one is correct. Being able to count over a million particles on the latest image analysers certainly gives confidence in the results from the particular sample being analysed, but if there is a sample-to-sample variation, the source of error is almost certain to arise from the method of sub-sampling.


The most accurate way of obtaining representative sub samples is to use a spinning riffler. This device can reduce samples from kilograms to milligrams where the sub samples will vary by less than 1% from bottle to bottle.

From being a simple qualitative tool, microscopy has evolved into one of the most powerful instruments in particle metrology and is unique in giving information on both particle size and shape. Provided care is taken on both sample selection and presentation to the microscope, the high computer power now accompanying image analysers ensure excellent reproducibility of the method, which is unlocking some age-old problems in the handling and reaction of powders. **LN**



Figure 1: The powder dispenser on the Malvern Morphologi G3 particle characterisation system.

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Filter testing of a wire woven stainless steel mesh

Suspension challenge test

In this method the filter was challenged with a range of NIST traceable glass calibration microspheres. A 0.1% suspension of the microspheres was ultrasonically dispersed and drawn through a 47mm diameter disc the filter under vacuum at 0.1 bar. The volume of suspension used was 20ml. The change in concentration of the suspension can be used to measure the efficiency of the filter, while the change on the particle size distribution reflects the cut point of the filter. The apparatus is shown in figure 1.

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