# Carbon Nanotubes:

## *Analysis of CNTs using the CPS Disc Centrifuge 'UHR'*

***By Dr Heather Murray, Analytik***

Carbon nanotubes (CNTs) are exceptionally strong, low density nano-scale structures with high thermal and electrical conductivity. To utilise these properties in CNT-based composite production the powdered CNT starting material must be non-destructively dispersed in the required suspending medium to preserve fibre-length. During the dispersion process agglomerated CNTs should be broken down into exfoliated particles, however, the inherent insolubility of CNTs in common organic solvents and water causes problems in dispersion production. Functionalisation (chemical treatment) of the CNT can lead to only partial exfoliation, with dispersion of bundles rather than fully isolated particles. Other suitable dispersion methods can include ultrasonication for dispersing low viscosity liquids and calendering between rollers for higher viscosity.

An evaluation of quality (of both the dispersal method and the dispersion itself) can be made by assessing the ratio of exfoliated particles to agglomerates. This degree of exfoliation can be obtained using particle size distribution analysis by differential centrifugal sedimentation (DCS); a technique employed by the CPS Disc Centrifuge 'UHR'.

**Analysis of CNTs**

A typical particle size distribution obtained using the CPS Disc Centrifuge 'UHR' for agglomerated CNTs (figure 1) shows two peaks.

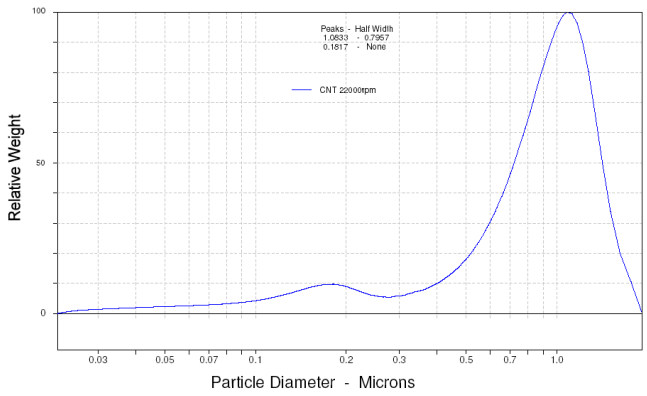


Figure 1: typical CNT analysis

It is known that incomplete CNT dispersion can have bimodal distributions for particle size density function caused by abrasion of larger agglomerates or particles as represented below (figure 2). The shift in the peak of the agglomerated fraction with longer dispersion (red to blue to black) shows the continued breakdown to exfoliated CNTs.




Tel: +44 870 991 4044 Email: info@analytik.co.uk **www.analytik.co.uk**

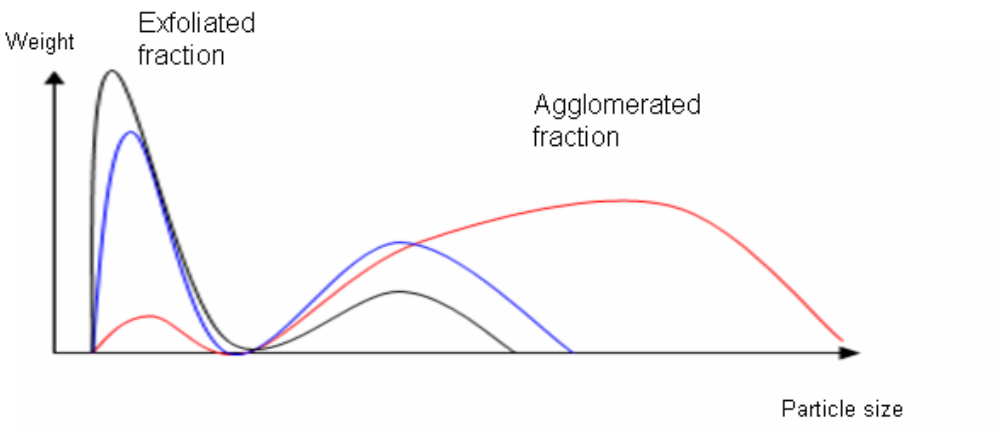


Figure 2: Representation of CNTs analysed by DCS
Dispersion: red low, blue medium, black high

**Comparison of single and multi-walled CNTs**

Agglomerated CNTs can be assumed to have spherical-like dimensions whereas an individual CNT is of a cylindrical shape. DCS techniques require particles to be spherical in order to make a direct measurement of diameter, however *a clear relationship between the real tube diameter and the DCS-measured equivalent diameter can be found*. Typical particle size density function results from the CPS Disc Centrifuge 'UHR' are represented below (figure 3) for dispersed single walled (SW) and multi-walled (MW) CNTs, and it is simple to differentiate between different diameter CNT types using the Disc Centrifuge. Corresponding microscopy studies show that measurement by the DCS is sensitive to CNTs of varying diameter rather than length.

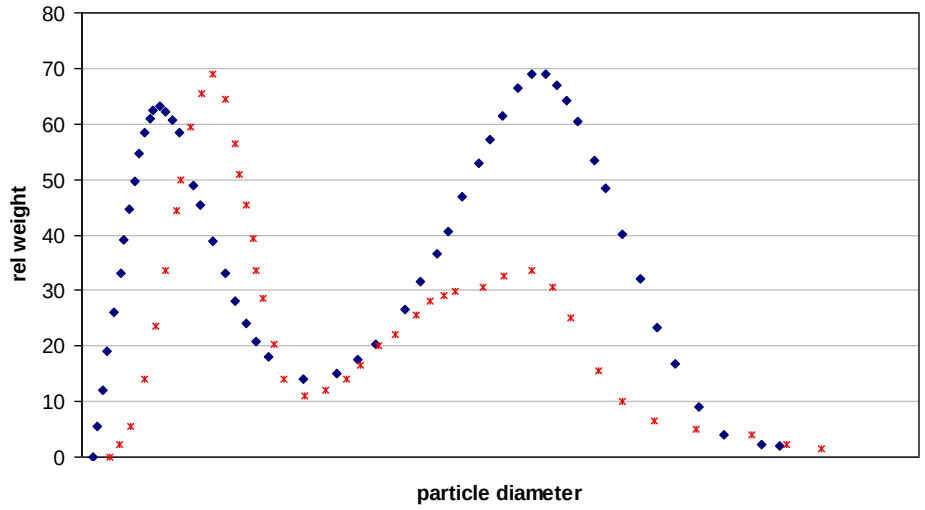


Figure 3: Single walled (blue) and multi walled (red) CNT analysis




Tel: +44 870 991 4044 Email: info@analytik.co.uk **www.analytik.co.uk**

**Real time analysis of ultrasonicated and calendered CNTs**

As stated above, it is known that CNT dispersions can have bimodal distributions for particle size density function caused by abrasion of larger agglomerates or particles. The Disc Centrifuge can be used to measure this bimodal distribution showing exfoliated and agglomerated fractions as a function of sonication time till a single fraction of exfoliated CNTs is found to show optimum processing time. Similarly, by the DCS analysis of CNT fractions during calendering using roll mills it is possible to determine the break up and dispersal of agglomerated CNTs directly in epoxy. Optimum sonication time (figure 4) and number of calendering cycles (figure 5) can be determined directly by calculating the ratio of agglomerated/exfoliated particles.

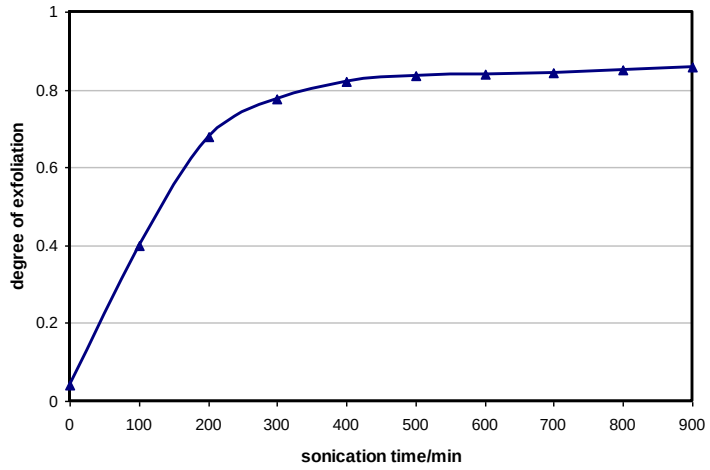


Figure 4: Degree of exfoliation versus sonication time

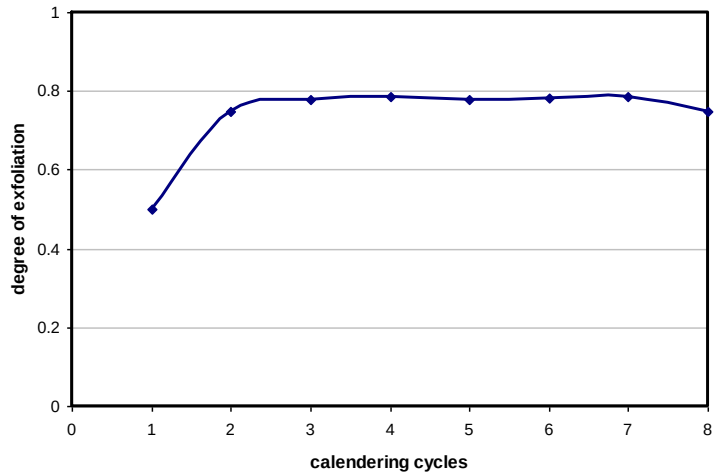


Figure 5: Degree of exfoliation versus calendering cycle

**Conclusion**

Large scale analysis with the CPS Disc Centrifuge 'UHR' can be used to monitor the dispersion of CNTs in real time, avoiding over-processing and maintaining their outstanding mechanical characteristics for composite manufacture.




Tel: +44 870 991 4044 Email: info@analytik.co.uk **www.analytik.co.uk**

**Introducing the CPS Disc Centrifuge 'UHR'**

The CPS Disc Centrifuge 'UHR' (shown in figure 6) separates particles by size using differential centrifugal sedimentation (DCS) in a liquid medium based on Stokes' law (which predicts the settling velocity of particles exposed to acceleration in fluids as a function of their diameter). A sample is injected into a hollow, optically clear disc (figure 7) containing a compatible density gradient, driven by a variable speed motor. The density gradient (typically a sugar/water mixture) is required to prevent the sample suspension simply settling as a bulk. The sample strikes the back face of the disc and forms a thin film which spreads as it accelerates radially toward the gradient liquid. On reaching the fluid surface sedimentation of individual particles begins, and as particles approach the outside edge of the rotating disc, they block/scatter the detector light beam that passes through the disc; the change in light intensity is continuously recorded, and converted into a particle size distribution. While DCS has been used to characterise particles in fluids for many years, recent technological advancements in the CPS Disc Centrifuge 'UHR' now allow for routine analysis of nano particle distributions down to 2nm and varying in diameter by as little as 2%. Typical analysis times are of the order of 3-15 minutes, with up to 40 samples analysed in a single run.


Figure 6: CPS Disc Centrifuge 'UHR'

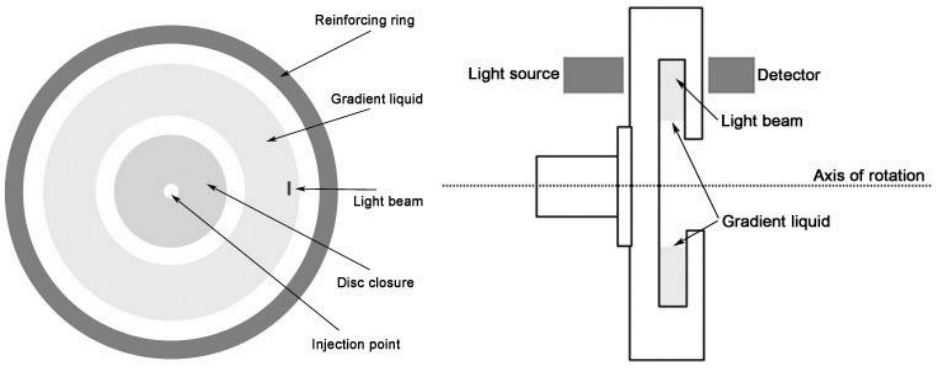


Figure 7: Schematic and cross section of disc

To learn more about high-resolution particle size characterisation using the CPS Disc Centrifuge 'UHR' please visit www.analytik.co.uk (UK and Ireland) or alternatively visit www.cpsinstruments.eu.




Tel: +44 870 991 4044 Email: info@analytik.co.uk **www.analytik.co.uk**