

Particle Characterization Helps Deliver Uniform Carbon Nanotube Products to Customers

THE CUSTOMER

Carbon nanotubes (CNTs) are low density, flexible, electrically conductive materials, with individual tubes having relatively high tensile strength. Nanocomp Technologies, Inc. produces carbon nanotubes in the form of sheets, tapes, powders, dispersions, and yarns. Nanocomp's products are used for aerospace, aviation, armor, and flame-resistant applications.

These hollow, tubular cylinders of aromatic carbon atoms exhibit distinct mechanical, electrical, thermal, and chemical properties. Individual tube diameters are ~ 10 nm with lengths of ~ 1 mm. Nanocomp's CNTs have tremendous aspect ratios; thousands of times greater than other commercially available carbon nanotubes.

Part of producing the carbon nanotube powders and slurries involves a proprietary, surfactant-free dispersion process. The result of this process is bundles of CNTs that are ~ 100 μm in width and ~ 1000 μm in length. That's where Dr. Joe Johnson, the Principal Scientist at Nanocomp, comes in. He oversees the dispersion and particle labs, as well as related applications like paints, composites, and polymers. His background includes over 20 years of experience working with particles, including synthesis, modification, product development, applications, and characterization.

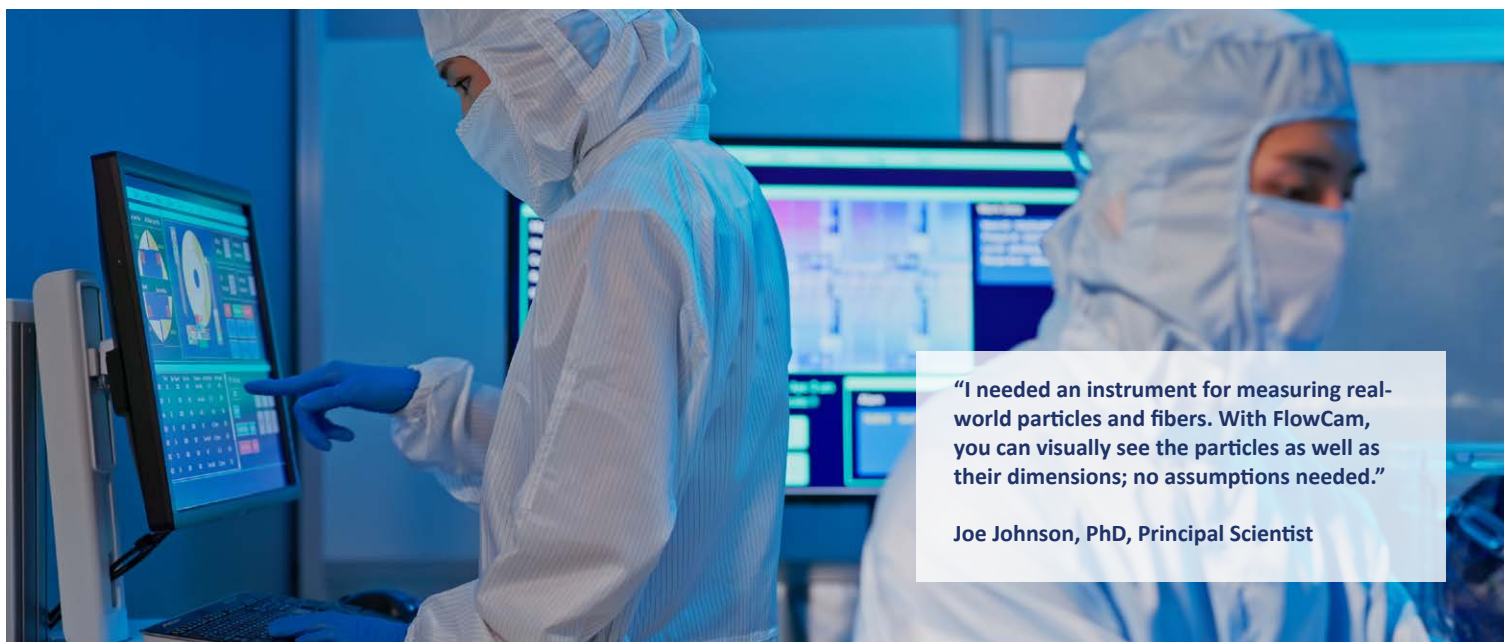
THE CHALLENGE

Dr. Johnson wanted an instrument that would help determine the grinding quantification of carbon nanotubes. Nanocomp's production procedure results in non-woven sheets - the CNTs held together mainly by van der Waal's forces. An analogy, although using vastly different forces and scale, would be many Velcro® strips pushed together to form a sheet.



Joe Johnson, PhD

A grinding device was previously identified to "rip" the nanotubes from each other resulting in fibers, or bundles of CNTs. Previous experiments identified process conditions (e.g. CNT concentration and instrument settings). The time of grind to achieve small, uniform fibers was unknown, so a study was planned that would measure particle size vs. grind time. Dr. Johnson needed an instrument that could quantify the width, length, aspect ratio, ESD (equivalent spherical diameter), and their distributions for dispersed carbon nanotubes.



"I needed an instrument for measuring real-world particles and fibers. With FlowCam, you can visually see the particles as well as their dimensions; no assumptions needed."

Joe Johnson, PhD, Principal Scientist

THE SOLUTION

Dr. Johnson did some research to learn more about particle size analyzers, and after comparing the available instruments he knew that the FlowCam flow imaging microscope met his requirements. With its VisualSpreadsheet® software, FlowCam can record over 40 different measurements per particle and can capture particle images at up to 100 frames per second which would enable Dr. Johnson to quickly get the answers he needed. “I have used many types of particle sizing instruments in the past and almost every one makes assumptions which are not valid in many respects,” explains Dr. Johnson. “They assume that the particle is spherical, and that’s not always the case, especially for high aspect ratio materials. Additionally, laser or light scattering devices ignore light absorption and scattering effects in their analysis. Acoustic devices have many assumptions including no particle-solvent interactions, uniform particle size and shape. I needed an instrument for measuring real-world particles and fibers. With FlowCam, you can visually see the particles as well as their dimensions; no assumptions needed.”

THE RESULTS

Dr. Johnson ran over a dozen batches of CNTs using the same process and conditions. “After dispersion we used FlowCam to characterize the carbon nanotube’s ESD [Equivalent Spherical Diameter],” he explains. “And we used ESD to determine how the product size decreased with dispersion energy and time.” The quantification involved measuring 3,000 CNT fibers three times, taking only a few minutes per test. That’s 9,000 particles per run condition.

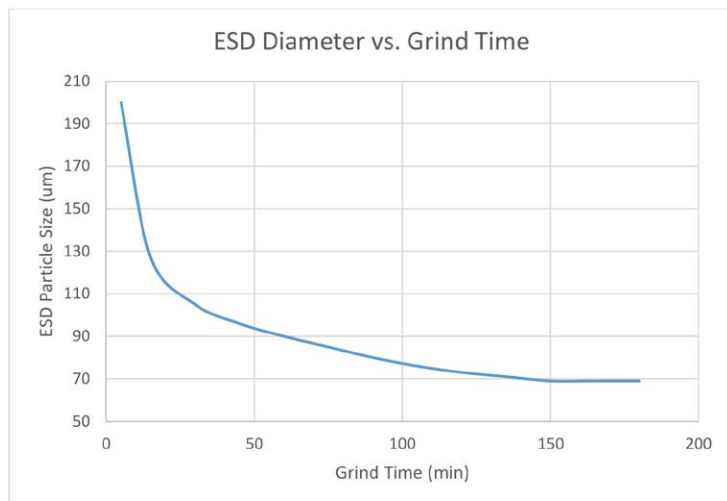


Figure 1. Equivalent Spherical Diameter of carbon nanotubes compared to Grind Time

The testing involved dispersing CNTs of a given concentration and set dispersing conditions. Small aliquots of dispersed CNTs were removed from the disperser over set times and analyzed using FlowCam. As a result, particle size was plotted versus time of dispersion with a typical graph shown in Figure 1. Note that for the first few points (under 30 minutes), estimates were averaged due to large fluctuations. Other studies showed no decrease in size after 200 minutes.

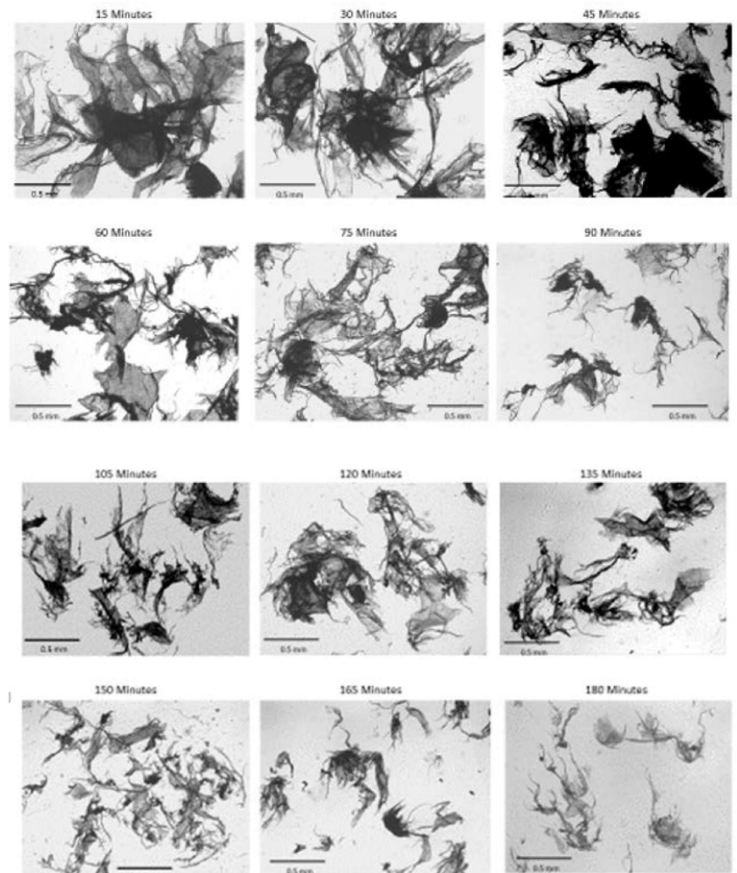


Figure 2. FlowCam images of carbon nanotubes

Figure 2 shows typical FlowCam images depicting diminishing carbon nanotube size with increased dispersion time.

For these studies, Dr. Johnson and his team expanded their work to include many standard and experimental sheets, different CNT concentrations, and differing process conditions. Particle characterization with FlowCam helps the Nanocomp team understand their products’ properties, and deliver uniform products to their customers.



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