RECOMMENDATIONS FOR LETTING DOWN NANOTUBE MASTERBATCHES

As-made nanotubes intertwine into agglomerates that are difficult to disperse. In order to insure consistent, high quality performance, Hyperion performs the initial dispersion of the nanotubes into a wide range of polymers. In addition to ready-to-use compounds, Hyperion offers a series of masterbatches (concentrates) of nanotubes dispersed into polymers. These masterbatches are typically of 15 to 20% concentration by weight and must be diluted to the final concentration.

It is important that the masterbatch dilution (letdown) be done carefully and thoroughly. A poor letdown gives a non-homogeneous mixture of the high viscosity masterbatch resin mixed with the lower viscosity let-down resin. This two-phase mixture can be visualized as small islands of masterbatch in the larger sea of the letdown resin. These small islands of masterbatch will act as a macro-sized, spherical conductive additive instead of the nano-sized, high aspect ratio nanotubes. The sample will perform as if the nanotubes were poorly dispersed, when, in fact, the dispersion is good, but the mixing is the issue.

Several papers have been published that can guide the letdown of Hyperion's masterbatches to make high quality compounds.

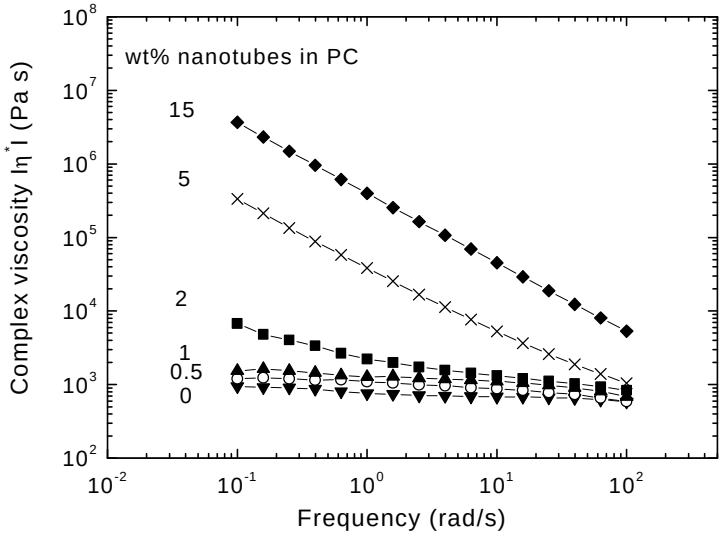
1] Rheological behavior of multiwalled carbon nanotube/polycarbonate composites; Petra Potschke, T.D. Fornes and D.R. Paul, Polymer 43 (2002) 3247-3255

2] Nylon 6 nanocomposites by melt compounding; J.W Cho and D.R.Paul, Polymer 2001; 42:1083 -- This paper has the screw design that was used in the first paper.

In the study a twin screw extruder was used to let down Hyperion's 15% polycarbonate masterbatch to make compounds containing nanotubes at 0.5%, 1%, 2% and 5% by weight. The extruder was a Haake co-rotating, intermeshing twin-screw extruder with a screw diameter of 30mm, having a centerline spacing of 26 mm and a length of 305 mm. Processing conditions were: barrel temperature of 240^{o}C, screw speed of 180 rpm and a feed rate of 920 gram/hour. The screw configuration contained two kneading disc blocks located at 35 and 147 mm, respectively, from the hopper. Both kneading disc blocks consist of one right-handed medium-pitched (L/D = 1.0), one left-handed medium-pitched (L/D = 1.0) kneading disc elements and one mixing ring.

The complex viscosities of the nanotube masterbatch, the pure polycarbonate (PC) and the diluted composites are shown in Fig. 1.

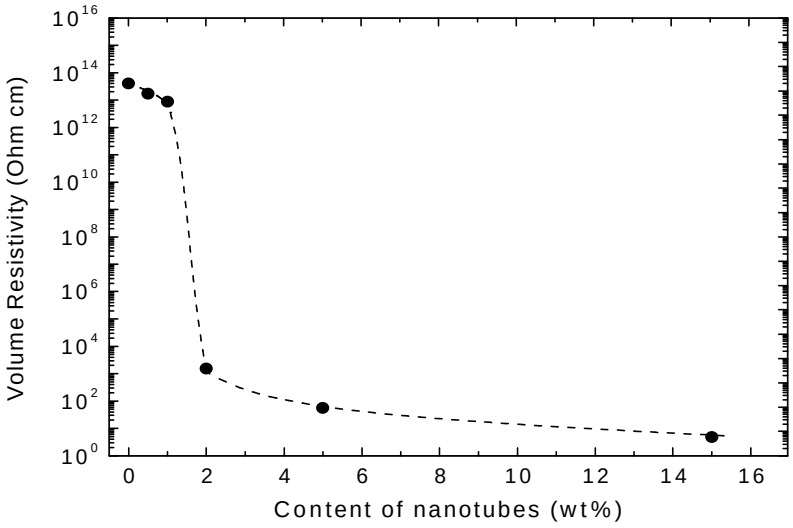
Fig. 1. Viscosity of Masterbatch, PC and Let-down Compounds at Different Frequencies (Shear Rates).



The masterbatch is orders of magnitude more viscous than the pure PC even at high shear rates and exhibits a very strong shear thinning effect. In contrast, the neat letdown PC shows only a small shear dependence. The complex viscosity of the different let-downs increases with the nanotube content. The effect of the nanotubes is most pronounced at low shear and the relative effect diminishes with increasing shear due to shear thinning.

The volume resistivity of the various let-down compounds were measured to give an accurate percolation curve for nanotubes in polycarbonate, Fig.2. It can be seen that the percolation threshold is between 1% and 2% in these well-mixed samples. At 2% loading, the conductivity is well within the range needed for most electrostatic dissipative (ESD) applications.

Fig. 2. Percolation Curve for Nanotubes in Polycarbonate



Recently Dr. Potschke, who is now at the Leibniz Institute for Polymer Research in Dresden Germany, has developed new data using our polycarbonate, polypropylene and nylon 6 masterbatches. The data indicates that it is better to use a low viscosity resin for the letdown resin rather then a high viscosity resin. This is counter-intuitive - we have always used high viscosity resins for letdown on the theory that it better matches the viscosity of the MB. But the data shows otherwise. The rationalization is that the low viscosity resin infuses into what is essentially an Interpenetrating Network (IPN) of nanotubes and resin in the MB. She has also shown that it is better to run the letdown extruder on the high side of the recommended temperature range for the particular polymer as this lowers the resin viscosity and improves dilution.

The data will be published in a communication early in 2005 and Hyperion will make it available as soon as it is out.