Sol-gel method and high-pressure technique combined together to obtain carbon nanotubes/silica nanocomposites

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Carbon nanotubes (CNTs) has been used as reinforcement in nanocomposites [1] to prevent crack propagation, increasing fracture toughness and also hardness. However, to improve these mechanical properties a good dispersion of CNTs must be achieved and this is still a challenge. In this work we studied: (i) the dispersion of MWCNTs (multiwalled CNTs) in water using a long chain surfactant (sodium stearate – NaS), (ii) the incorporation of these MWCNTs into silica matrix by sol-gel method and (iii) the obtaining of compacts by high pressure (HP) technique. Firstly, it was prepared an aqueous solution of NaS of 0.01g/mL that was separated in two halves, solution B with $[CNT]_B = 4.15 \times 10^{-5}$ g.mL^{-1} and solution C with $[CNTs]_C = 6.3 \times 10^{-5}$ g.mL^{-1}. Silica powders were prepared by sol-gel method with TEOS as silica precursor, in acidic medium and with 1.6 mL of CNTs/NaS solutions B and C. This sol was left to gelify and to evaporate, in air, at room temperature for 30 days, up to attain a constant weight resulting in the monoliths MB and MC, respectively. They were comminuted in an agate mortar and half of powder obtaining from MB was calcined at 500 ºC for 3 hours in air to eliminate the organic residues. To obtain a compact, these powders were precompacted in a piston–cylinder type apparatus to approximately 0.1 GPa, and then placed inside a lead container, which acts as quasi-hydrostatic pressure transmitting medium. This container is set up in a toroidal-type HP chamber and the compaction is accomplished at 6.0 GPa, at room temperature obtaining samples 1 (from MC), 2 (from MB) and 3 (from MB calcined). The samples were characterized by X-ray diffraction, Vickers microhardness, density measurements (picnometry) and SEM. Figure 1 shows the MWCNTs dispersion in water (A) and in the aqueous solution with NaS (B and C solutions). Figure 2 shows sample 3, a transparent compact. Figure 3 shows density measurements for the three samples. Our preliminary results indicate that samples 2 and 3 have higher density and we attributed that to a better dispersion of MWCNTs into the silica matrix.

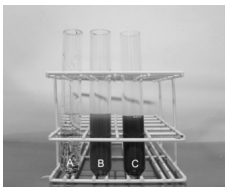

Figure 1: MWCNTs dispersed in water (A); solution (B) and solution (C).

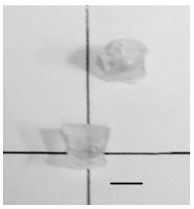

Figure 2: transparent compact obtained at 6 GPa (Bar = 5mm).

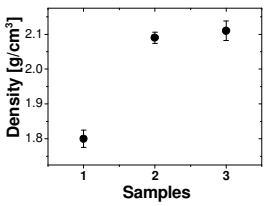

Figure 3: density measurements for samples 1, 2 and 3

Keywords: sol-gel method, silica, carbon nanotube dispersion
Work supported by CNPq, CME-UFRGS (Brazil); AFOSR (FA9550-08-1-0426) (USA); Bayer (for the MWCNTs-Baytubes).

[1] M J de Andrade, M D Lima, C P Bergmann, G O Ramminger, N M Balzaretti, T M H Costa, and M R Gallas. Nanotechnology **19,** 265607 (2008). *marcia@if.ufrgs.br