

Production and Structural-Chemical Characterization of Cu-MWCNT Nanocomposites

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The objective of this study is to produce a bulk nanocomposite constituted by Copper reinforced with multi walled carbon nanotubes (MWCNTs) and evaluate the resulting mechanical and transport properties. Such motivation relies on the well-established superior mechanical and transport properties of CNTs. A resulting metal-matrix bulk nanocomposites with improved mechanical and transport properties is to be expected [1,2,3,4]. The present investigation reports the synthesis and processing procedure followed by the structural and chemical characterization of a Cu - 5 wt (%) MWCNT nanocomposite developed in our laboratory. Purified MWCNT provided by UFMG Brazil were used, with diameters between 10-60 nm. MWCNTs were functionalized using a mixture of H₂SO₄: HNO₃ (3:1).

Nanocomposite powders were produced by dissociation of a homogeneous suspension containing Cu(NO₃)₂ · 3H₂O – MWCNT previously functionalized; followed by H₂ reduction of the obtained CuO-MWCNT precursor. Bulk nano-composite pellets were obtained by a pre-compaction under uniaxial pressure followed by isostatic pressure. Sintering of the compacted material was carrying out at 950°C under Argon atmosphere by 30 min.

A FEI Titan operating at 300KV, a Jeol 2010 operating at 200KV and a SEM-FEG Magellan have been used as main characterization tools. Sample preparation used a FIB-Nanonova instrument. Conventional transmission electron microscopy using diffraction contrast showed well-attached copper nanoparticles (10 -20 nm range) on carbon nanotubes (Fig 1a and Fig 1b). After consolidation into pellet and sintering, the Cu matrix presented heterogeneous grain growth (50nm–300µm range), as observed in Fig 1c. Sample preparation using FIB, allowed the observation of a large electron beam transparent region. Ion beam image confirm the heterogeneous grain growth along the sample thickness as product of the cold compaction (fig 2a). SEM-STEM observations under 5 kV have confirmed the presence of CNTs, taking in advantage the difference in Z contrast between carbon and copper. Oxygen is actually detected in the metal matrix, suggesting it arises from a post-oxidation stage, rather than due to an incomplete CuO particle reduction, Figs 2b and 2c. The interface of CNT's incorporated through the processing of the nanocomposite in the copper matrix has been analyzed using high resolution transmission electron microscopy (Fig 3a). Decreasing of CNTs diameter suggests a possible graphitization of CNTs along the thermo-mechanical processing. Analysis of Fourier transform shows the presence of Cu₃O₄ in the matrix as product of re-oxidation as confirms by EDS (Fig. 3b and Fig 3c) .

References

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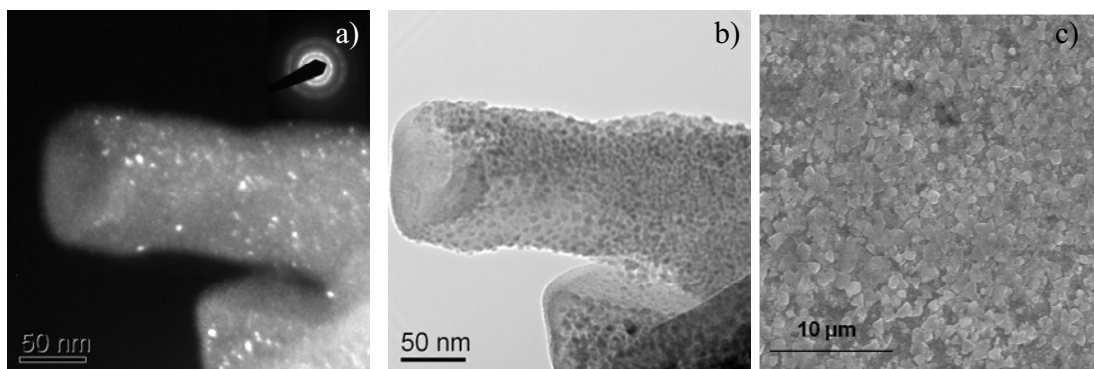


Fig 1. a) and b). TEM center dark field and bright field images of CNT well decorated by copper nanoparticles. The grain size of a pellet, in the 300 nm - 3 μm range as observed in SEM image (1c).

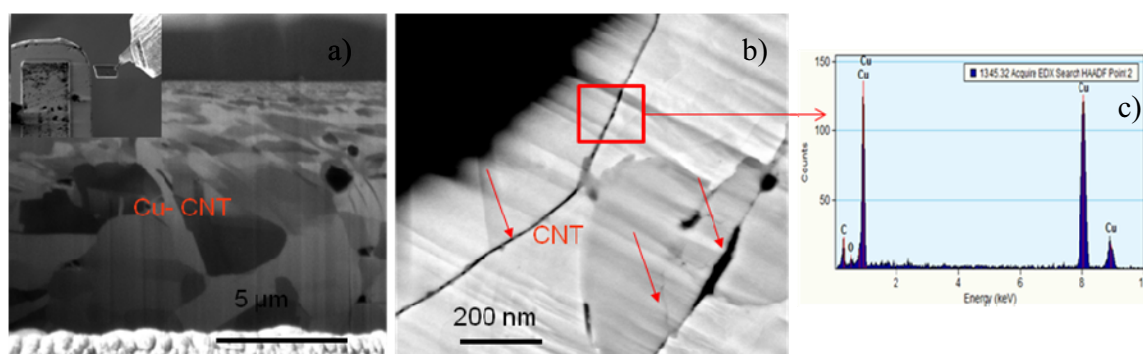


Fig 2. Low voltage SEM-STEM- HAADF images. a) Heterogeneous grain growth as product of consolidation and sintering process; b) shows the presence of nanotubes into the copper matrix; c) EDS confirms the presence of carbon and suggests the re-oxidation of the sample at room conditions.

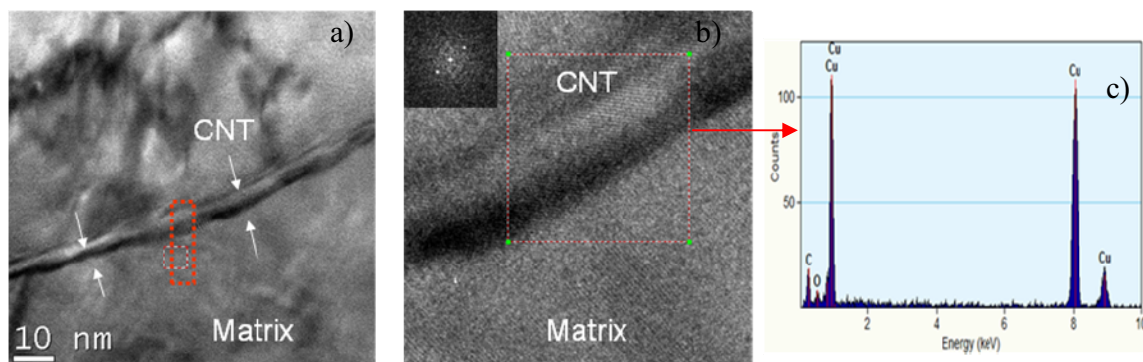


Fig 3a. High resolution transmission electron microscopy image show a CNT imbedded in the Cu metal matrix. Analysis of Fourier transform reveals the presence of Cu_3O_4 in the matrix fig 3b). EDS confirms again the presence of carbon, copper and oxygen.