The increasing interest in nanostructured materials in recent years has provided incentive to develop new kind of composites containing carbon nanotubes (CNTs). In particular, Copper-multiwall carbon nanotube nanocomposites (Cu-MWCNT) have been considered as a promising material for applications in electronic materials, heat exchangers and structural elements [1,2]. It has been reported that the addition CNTs in a Cu matrix can increase or decrease the mechanical and transport properties of the matrix depending on the Cu-CNT interface, CNT integrity as well as on the CNT uniform distribution, which are directly correlated with the nanocomposite synthesis and processing(sintering). The main objective of this work is to characterize structurally, morphologically and analytically a Copper -5 wt% MWCNT nanocomposite produced by chemical synthesis and thermo mechanical processing (Spark plasma sintering), by means of transmission electron microscopy (TEM).

Nanocomposite powders were produced by dissociation of a homogeneous suspension containing Cu(NO3)2.3H2O-MWCNT, previously functionalized in tetrahydrofuran solution, followed by H2 reduction of the obtained CuO-MWCNT precursor. Bulk nano-composite pellets were obtained using a Doctor Sinter Lab Machine (SPS 1050) applying 70 MPa pressure, at 600 °C for 5 minutes.

TEM samples were prepared from the powder synthesis material and from the obtained final pellets. The former sample was prepared using a dispersion of few milligrams of powder in isopropyl alcohol, followed by ultrasonic agitation. One drop was placed on a nickel TEM grid. The later was made using a FEI Nova FIB-SEM instrument. A TEM -FEI Titan operating at 300kV equipped with EDS, and EELS were used as main characterization tool.

After chemical synthesis Cu powder particles with spherical and faceted morphologies decorating the MWCNTs were observed. The particles were in the 5-100nm range (Fig.1a), showing good adherence at the interface (Fig.1b). After consolidation into pellet and sintering, good consolidation and heterogeneous grain growth (50nm–2μm range) were observed in the Cu matrix. Remaining porosity and annealing twins are present at the lamella. TEM-BF images show regions with high dislocations density as well as the presence of CNTs at the Cu grain boundaries and its transformation into amorphous carbon, nanoribbons and graphitization (Fig 2).

Elemental mapping using STEM-EELS allowed us to identify the presence of carbon, copper oxide and metallic copper at the interface Cu-MWCNT (Fig. 3). Notwithstanding, possible re-oxidation after sample preparation can be considered.

References

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Fig. 1: MWCNTs decorated by copper nanoparticles and good adherence at the interface are observed in the TEM bright field images (a and b).

Fig. 2: (a and b). TEM-BF images show regions with high dislocations density and typical strain fields as well as big damage of CNTs. HRTEM (Fig 2 c) shows a carbon nanostructure like ribbon as product of MWCNT transformation during the sintering processes.

Fig. 3: Elemental mapping using STEM-EELS allowed us to identify the presence of carbon, copper oxide and copper at the interface Cu-MWCNT.