MOSSBAUER SPECTROSCOPY OF RESIDUAL IRON IN PURIFIED MULTI-WALLED CARBON NANOTUBE BY THERMAL ANNEALING UNDER VACUUM

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1. Introduction

The best way to remove iron content from multi-walled carbon nanotube (MWCNT) powder is by thermal annealing under vacuum. Beside of efficient iron evaporation, this kind of treatment brings improvement on graphitic ordering of tube wall [1-2]. However, very small quantities of iron (less than 1% in weight of carbon nanotubes) can be detected by Mossbauer spectroscopy. Hence, a brief study of iron phases present in as-grown MWCNT and MWCNT treated at high temperatures will be shown in this work.

2. Experimental

The MWCNT powder was obtained by pyrolysis of camphor mixed with ferrocene (16%), at 850^{o}C under atmospheric pressure. The powder passed by thermal annealing under vacuum at 1800^{o}C during 1h for iron removal. The as-grown and purified MWCNT were analyzed by transmission electron microscopy (TEM), X-ray diffraction, Raman spectroscopy, thermogravimetric analysis and Mossbauer spectroscopy.

3. Results and Discussions

Figure 1 shows TEM images of MWCNT powder (a) before and (b)after annealing. The dark points in images refer to iron nanoparticles used as catalyst of nanotube growth. Notice that in Fig.1a, there is a higher quantity of iron nanoparticles is inside the nanotubes. Although the purity of carbon nanotubes has reached at least 99% , iron can be seen in the nanotube walls in Fig.1b. Mossbauer spectroscopy confirms the iron presence in form of α-Fe (sextet), γ-Fe (singlet) and Fe_3C (sextet), as shown in Fig.2 (a-b). However, α-Fe is absent in Fig. 2b. During thermal annealing, the iron is evaporated, and passes through the graphitic

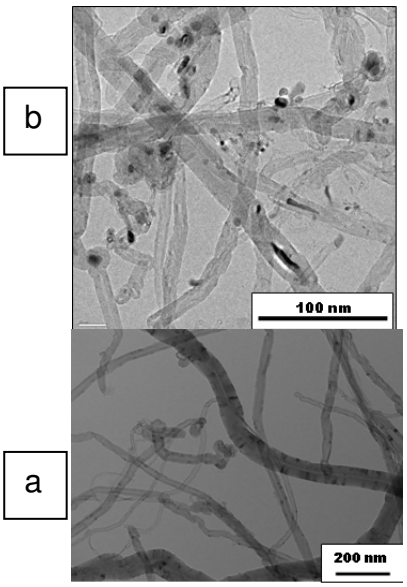

Fig 1. *TEM images of iron nanoparticles inside the tubes and at tube walls, for* **(a)** *as grown and* **(b)** *purified carbon nanotubes, respectively.*

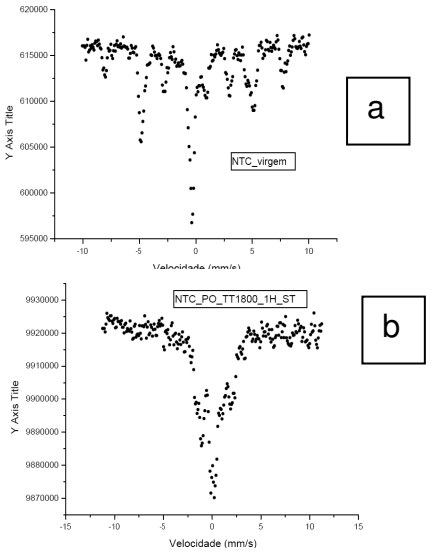

Fig. 2. *Mossbauer spectra of iron content in* **(a)** *as-grown and (b) purified MWCNT. Notice the ausence in peak ~5mm/s from a-Fe for* **(b)**

walls by diffusion mechanisms. If the treatment time is not enough, a part of iron is retained in tube walls in the form of γ-Fe-C [3-4].

4. References

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Acknowledgments

The authors are grateful to Fapesp for financial support and to L.C. Pardini from Instituto de Aeronáutica e Espaço (Brazil) for the high temperature furnace.

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