Native oxide film reduction on stainless steel for direct growth of carbon nanotubes

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Native oxide film on stainless steel (SS) gives a high resistant to degradation by oxidation or by corrosion processes, which is attributed to the formation of the surface chromium oxide film. This chemical stability allows the use of SS in environments where is critical to avoid undesired reactions. A particular use is as a substrate for carbon nanotubes (CNTs) forests. Hence, it is possible to manufacture electrodes for batteries or supercapacitors that are in contact with liquid electrolytes, which are extremely corrosive environments for other conductive materials like cooper. Several works use the native oxide films as part of the diffusion barrier system to obtain CNTs. However, this film has two drawbacks: low electrical conductivity and shielding of the catalytic activity of metal (iron) nanoparticles. Here, we present our results about the chemical reduction of the native oxide film on SS 304 using hydrogen/argon plasma. Through this process, it is possible to keep the original surface properties such as electrical and thermal conductivity as well as catalytic activity. The samples were heated up to the annealing temperature (AT) in a hydrogen atmosphere. After reaching the AT, the argon gas was introduced. During three minutes the samples were exposed to a RF hydrogen/argon plasma. The growth process of CNTs was carried out directly, without the need of a diffusion barrier and using the alloy elements of SS (Fe, Ni) as catalysts. To evaluate the catalytic activity of the substrate, two processes were used: plasma enhanced chemical vapor deposition (PECVD) and water assisted chemical vapor deposition (WACVD). By this method, a uniform forest of CNTs were obtained on 9 cm² samples at 730°C. The optical emission spectroscopy (OES) spectra, obtained during the reduction time, shows the gradual decrease in intensity of the peaks generated by the interaction between the hydrogen and the oxygen atoms. In addition, scanning electron microscope (SEM) images show the morphology of the CNTs. With

PECVD, the CNTs are short and present a uniform distribution and orientation. But, with WACVD the CNTs are longer, present a uniform distribution and random orientation. In both cases, the density of the CNTs is lower than that of the samples produced in our laboratory on silicon wafers. Finally, using Raman spectroscopy, the quality of CNTs was evaluated. With PECVD, there is a presence of amorphous carbon. The G-band and the D-band peaks are not well defined, and the G'-band peak does not appear. Whereas for the samples obtained by WACVD the G-band, D-band and G'-band peaks are sharper, which indicates that with WACVD the amorphous carbon was removed.

References

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Figures



Figure 1. Raman spectra for directly growth of CNTs at 730 °C on stainless steel 304 by PECVD and WACVD.