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SEM AND TEM CHARACTERISATION OF Ni-Pd-CNTs COMPOSITE FILMS

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Introduction

Recently, carbon nanotubes (CNTs) due to their unique properties are applied as chemical sensors, biosensors and hydrogen storing materials. Developed structure makes CNTs an ideal material for gas adsorption. A lot of researchers groups investigate hydrogen storage and hydrogen sensing on CNTs. To improve hydrogen adsorption of CNTs doping of transition metals like Pd, Ni, Pt is applied. Decoration of CNTs by such objects as Pd, Pt or Ag nanoparticles enables an application of these functionalised materials for biosensors or activators.

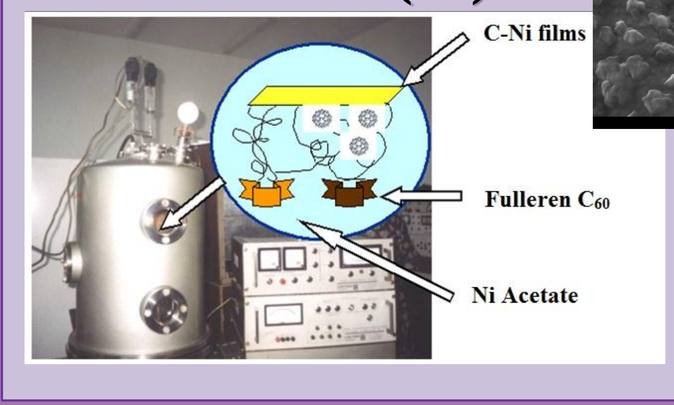
In this work investigations of Ni-Pd-CNTs composite films prepared by elaborated in Tele- and Radio Research Institute, new three steps method are presented. This method consists of PVD (Physical Vapor Deposition) process in which nanocrystalline Ni precipitate was obtained, next CVD (Chemical Vapor Deposition) process in which film composed of CNTs with included Ni nanoparticles were prepared and then again PVD process that allowed for covering of CNTs with a carbonaceous – palladium nanograins with a cap-like shapes. Films were characterized by SEM, TEM and EDX. The hydrogen sensing properties of prepared in such way samples were also studied.

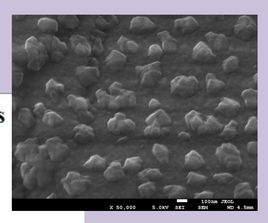
Experimental

Films were deposited on Al_2O_3 (sample S1, S2) and Si (sample S3) substrates.

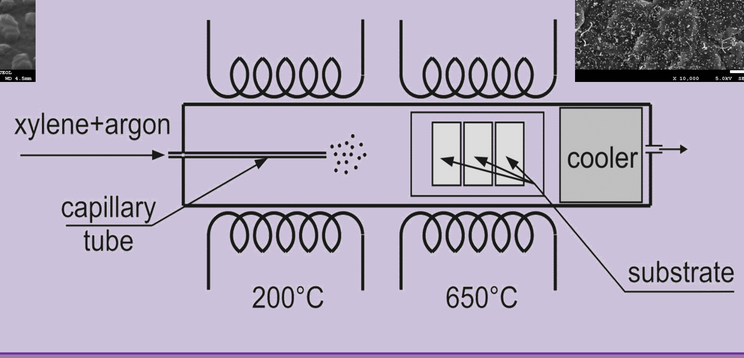
Preparation steps of Ni-Pd-CNTs films

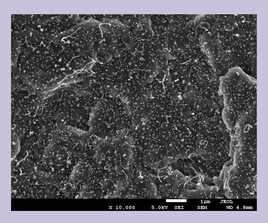
1. PVD (Ni)



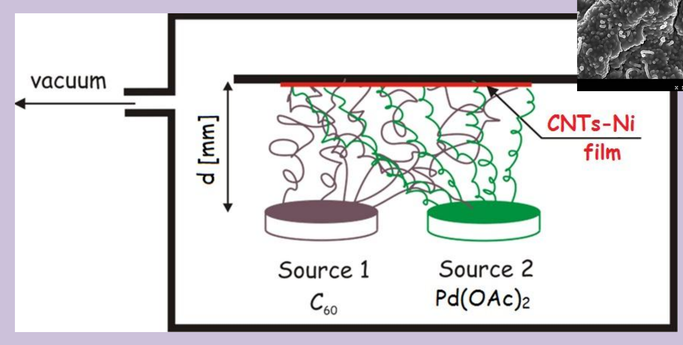


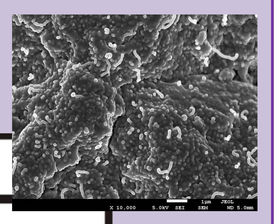
2. CVD





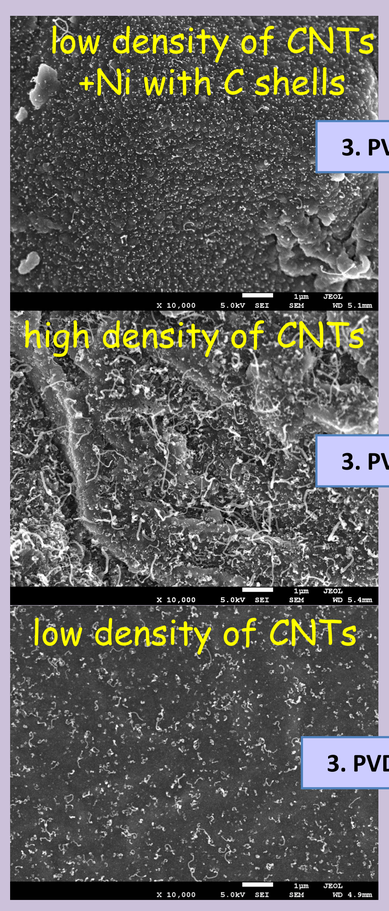
3. PVD (Pd)





SEM, TEM and sensing characterisation

Ni-CNTs films



S1

S2

S3

3. PVD (Pd)

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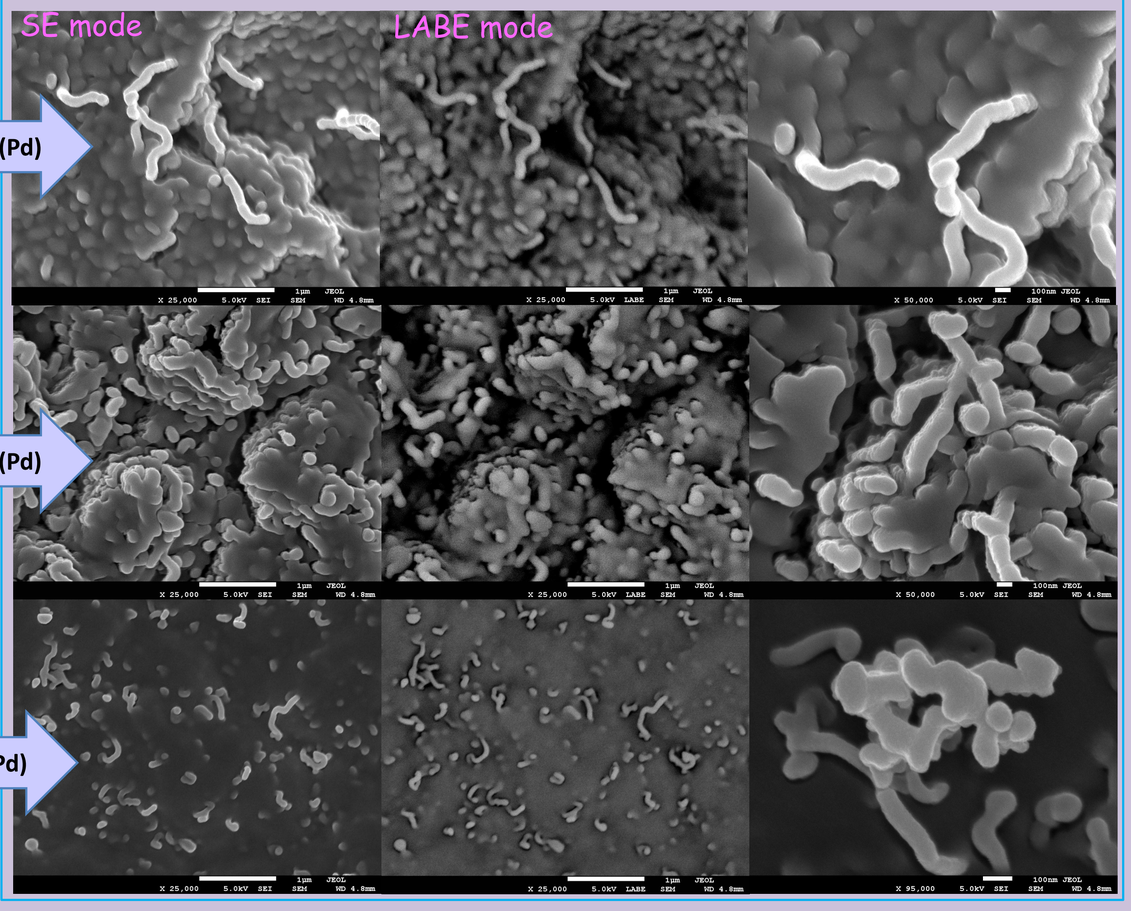
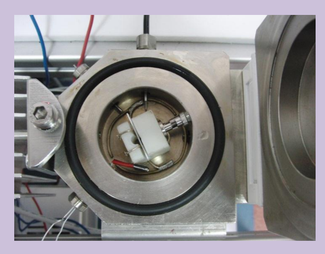
Pd-Ni-CNTs films

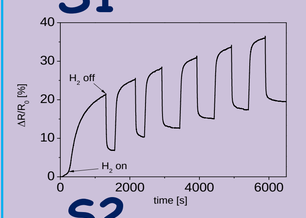


The changes of electrical resistance of Pd-Ni-CNTs films were studied in 1%H_2/N_2 atmosphere. The measurements were performed at the room conditions. After absorption of hydrogen, the air was introduced to measurement chamber (presented below) to remove H_2 gas.

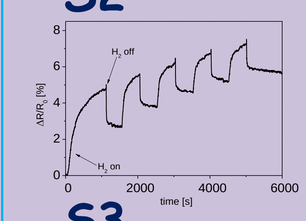


measurement chamber

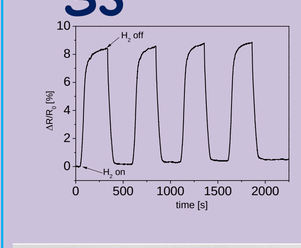
S1



S2

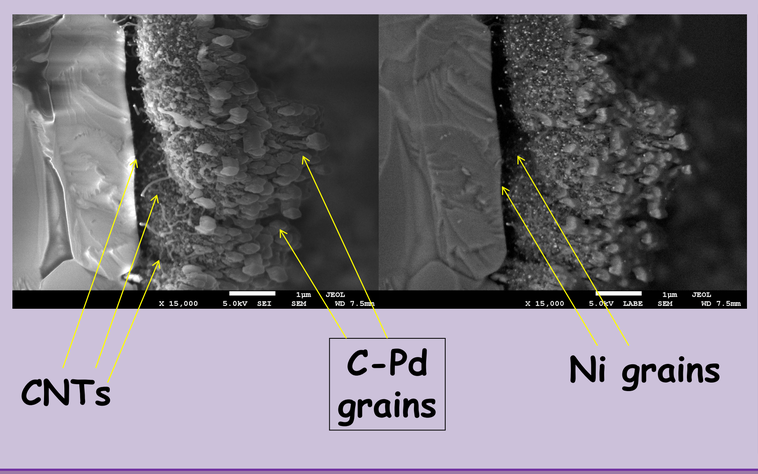


S3

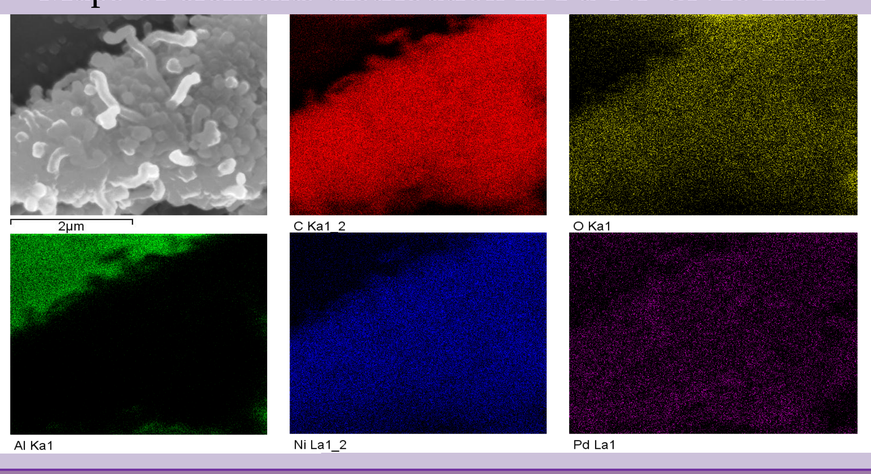


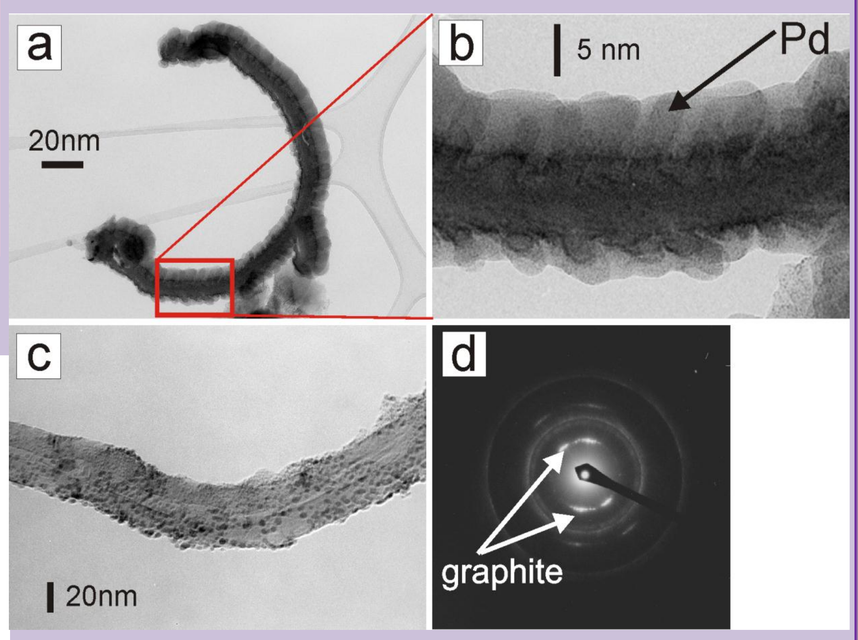
$$\frac{\Delta R}{R_0} = \frac{R_t - R_0}{R_0} \cdot 100\%$$

Cross section of Pd-Ni-CNTs film



Maps of elements distribution in Pd-Ni-CNTs film





a), b), c): TEM images of decorateed with Pd nanoparticles CNT's; d) diffraction pattern for this objects

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Conclusions

We obtained carbon nanotubes decorated with Pd nanoparticles and nanograins. The diameter of initially prepared by pyrolitic process CNTs changes from 20 to 70 nm and their length is from 100 nm to 1µm. Palladium nanoobjects have various sizes and shapes as well as they coat CNTs enlarging their initial size. We distinguish two kinds of Pd nanoobjects: sphere-like and elongated.

The influence of shape, size and type of coating of Pd nanograins should additionally studied in further works.