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Palladium particles situated in SiO_2 nanotubes

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Introduction

Metal nanoparticles including palladium nanoparticles have been intensely investigated because of their unique chemical and physical properties that allow the use of these structures in various fields like optics, electronics, magnetic devices, photocatalysts, biomedical sensors and adsorbents. Pd nanoparticles due to their small size are very attractive in the catalysis process because of their large surface to volume ratio. Morphology of palladium metallic particles is strongly dependent on an interface with surrounding matter.
Here we present palladium nanoparticles placed inside silica nanotubes („glassy" nanotubes).

Applications

This type of SiO_2 nanotubes with Pd nanoparticles included inside them can be applied in chemical sensors, electroluminescence diodes, photodetectors, MOS transistors and other electronic devices. Also they can be use as low-resistance interconnects in high-speed circuits.

Synthesis process of SiO_2 nanotubes with Pd nanoparticles placed inside

The synthesis process is composed of third stages. **In the first stage** the nanocomposite palladium-carbonaceous films are deposited on Si substrates using PVD method. As precursors of PVD films fullerene C_{60} and Pd acetate are applied. Depending on technological parameters of PVD method we can obtain films with the different concentration of Pd. **In the second stage** PVD films are modified by xylene and the temperature in CVD process. As a result of two stages palladium silicide (Pd_2Si) nanowires are formed. **The final stage** (third stage) consist in the annealing of Pd_2Si nanowires in the air at the temperature above 900℃.

PVD process (I stage)

PVD process was performed under dynamic vacuum of 10^{-5} mbar. The current through the fullerene source was 2,1A, and through palladium source was 1,2A. The distance between sources was 54mm and the time was 10min. As a result carbonaceous-palladium films with the thickness of ~300nm was deposited on Si substrate.

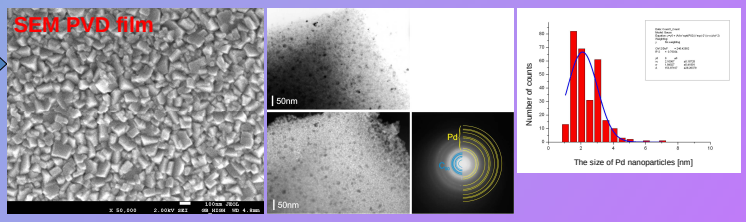
CVD process (II stage)

CVD process was carried out in the quartz reactor using xylene as an additional the carbon source. Xylene was injected with the speed of 0,1 ml/min to the reaction zone (650°C) by argon flowing at the rate of 40l/h. In these conditions Pd nanoparticles from PVD film reacted with Si substrate and palladium silicide in the form of nanowires were formed. Pd_2Si nanowires grow locally in the different spatial directions and their diameters vary along their length.

Annealing of CVD film in the air (III stage)

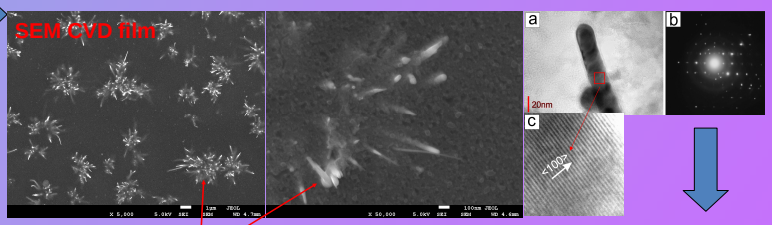
The film after CVD process was annealed in the air at temperature above 900℃. This high temperature caus es the silicon oxidation from Pd_2Si nanowires to SiO_2 and fragmentation of palladium to nanoparticles with the size of 20-50nm. In this way we prepare silica nanotubes with Pd nanoparticles placed inside.

SEM and TEM investigations



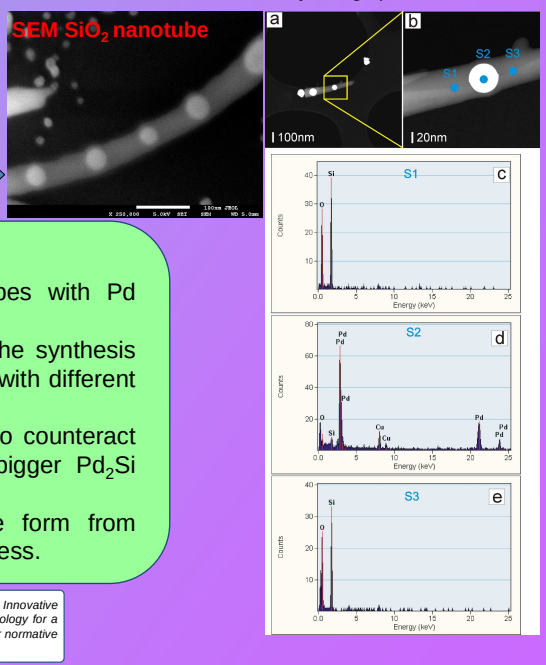
Carbonaceous nanograins with different size (50-300nm) are visible on the filme's surface

TEM investigation shows that carbonaceous grains contains C_{60} nanograins and Pd nanoparticles (with the size of 2-4nm)



Pd_2Si nanowires

(a) TEM image of Pd_2Si nanowires in bright field, (b) electronogram corresponding to this structure, (c) fragment of Pd_2Si nanowire at high magnification with the selected crystallographic directions



EDX collected from the different places S1, S2 and S3 marked on silica nanotubes

Conclusion

- Our research has focused on the synthesis of silica nanotubes with Pd nanoparticles placed inside;
- Depending on the technological parameters of three stages of the synthesis process we can obtain SiO_2 nanotubes containing Pd nanoparticles with different size and different distribution;
- In CVD process the presence of hydrocarbon is recommended to counteract the formation of palladium silicide in the form of the layer or bigger Pd_2Si nanoobjects like islands etc.;
- Silica nanotubes with included Pd nanospheres one can be form from palladium silicide nanowires at the proposed parameters of CVD process.

INNOVATIVE ECONOMY

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