QUANTUM GRAVITY RESEARCH | HOME OF EMERGENCE THEORY

# Synthesis and characterization of Pd and Ni nanoparticles confined in microporous structures for the LENR applications

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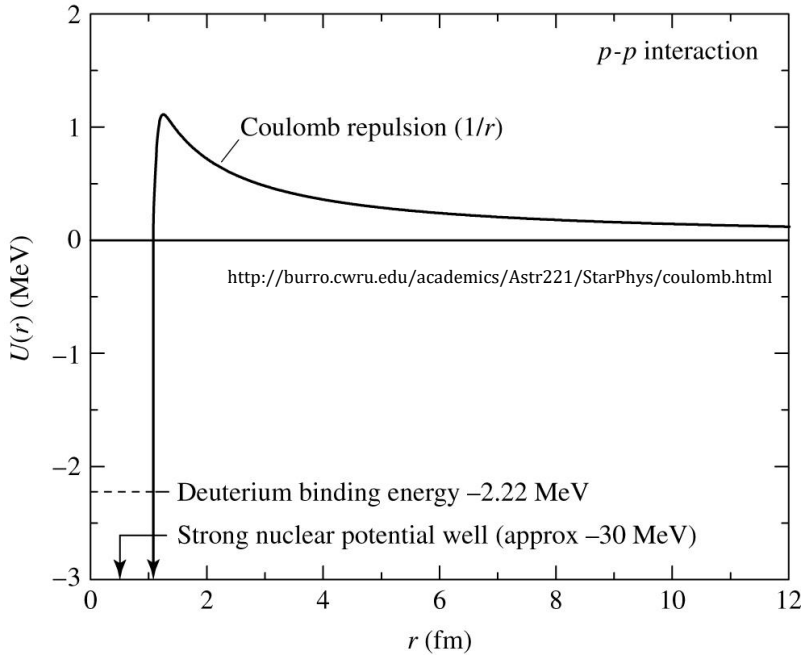
Assisi, 2019

# Overcoming the Coulomb Barrier




Driving of adjacent potential wells occupied by hydrogen ions

*large amplitude anharmonic lattice oscillations (discrete breathers )*

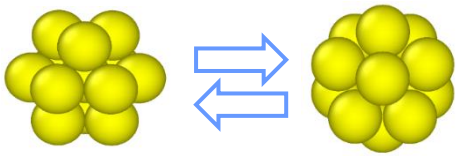
*fast phase transformation of quasicrystals (phasons)*

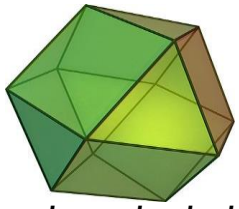
nuclear active sites

*Result of molecular dynamic modelling*

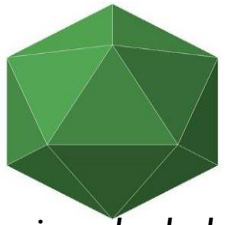
*Reversible phason flips from CUBO to ICO symmetry is possible for Pd or Ni nanoclusters*

*Pd-13 and Ni-13 clusters*



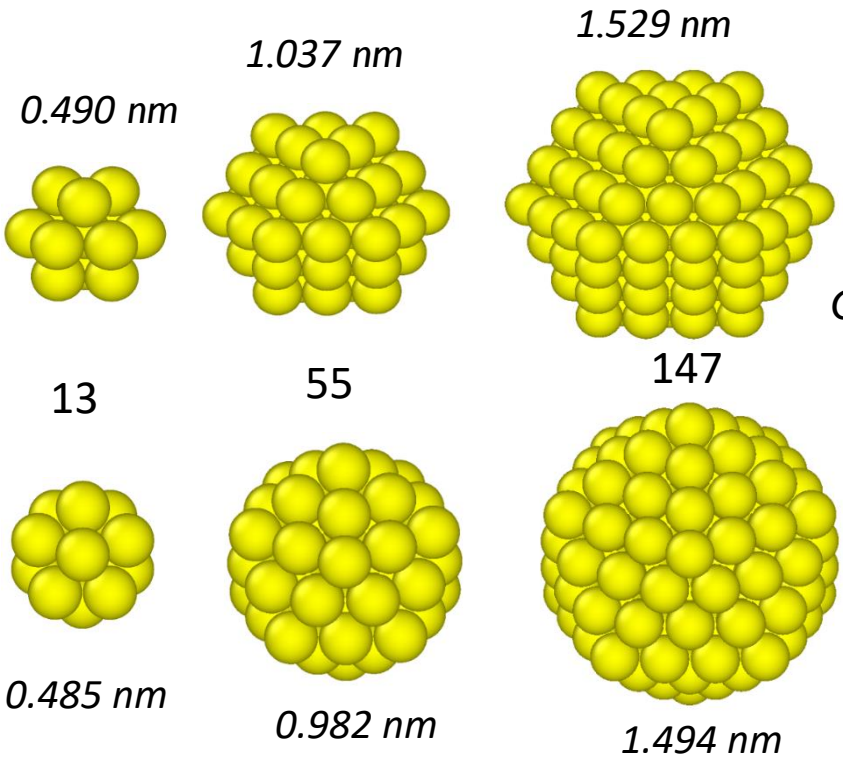


*cuboctahedral*



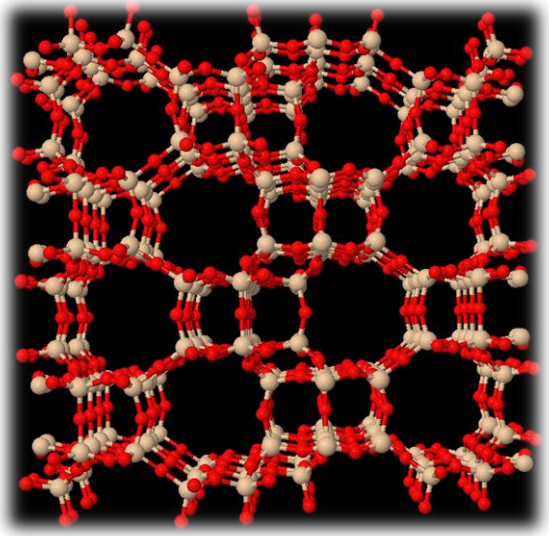
*icosahedral*

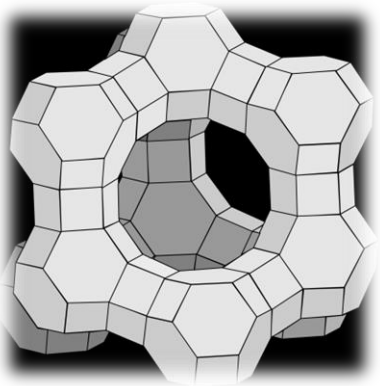
## *Pd and Ni nanoclusters*



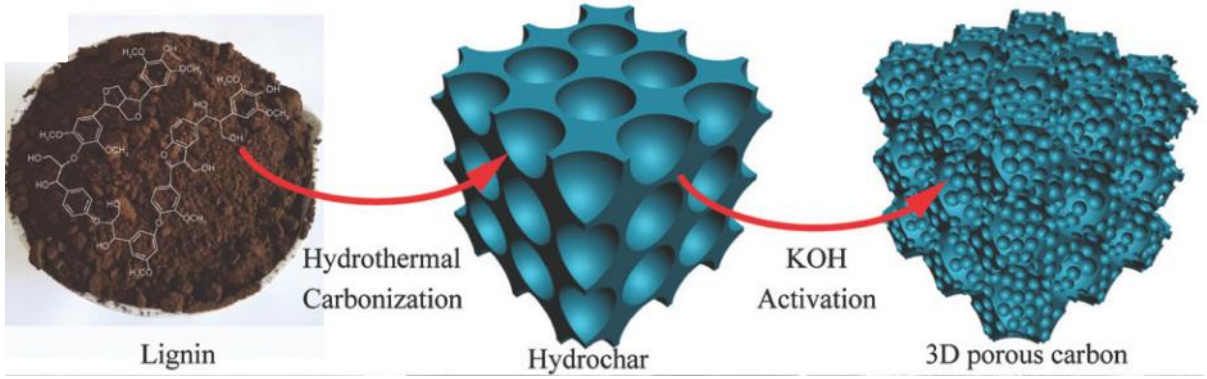

Template synthesis

# *Pd and Ni nanoclusters: templates*



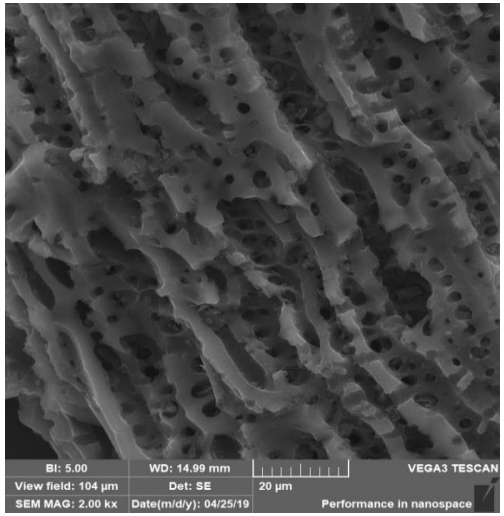


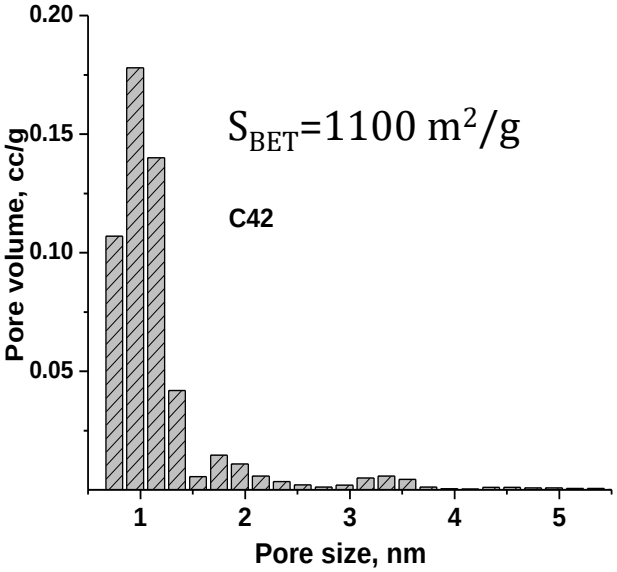
*Zeolite (alumosilicate) structure*
*three-dimensional framework*
*with two types of cages:*
*1.3 nm and 0.74 nm.*
*Uniform pore size distribution*

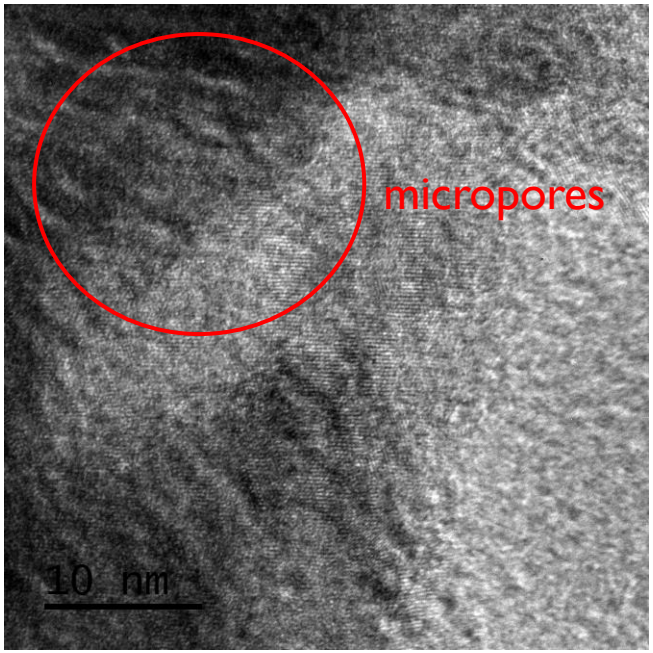


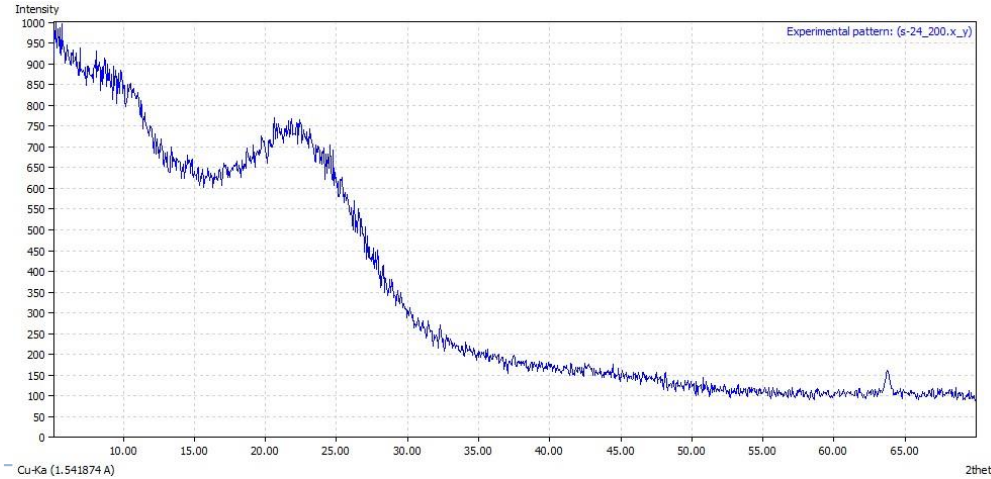

*Porous carbon*
*three-dimensional framework*
*Pore size depends on the carbonization and activation conditions*
*Pore size is controllable*

**DOI:**10.1039/C7GC00506G




*SEM and TEM images, XRD pattern and pore-size distribution of C42 carbon sample used as a template for Pd-particle growth*

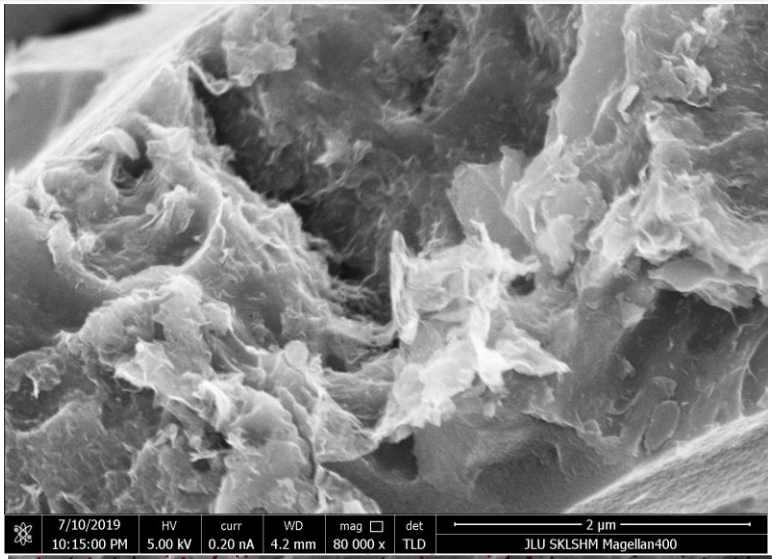
*Syntesis protocol:*

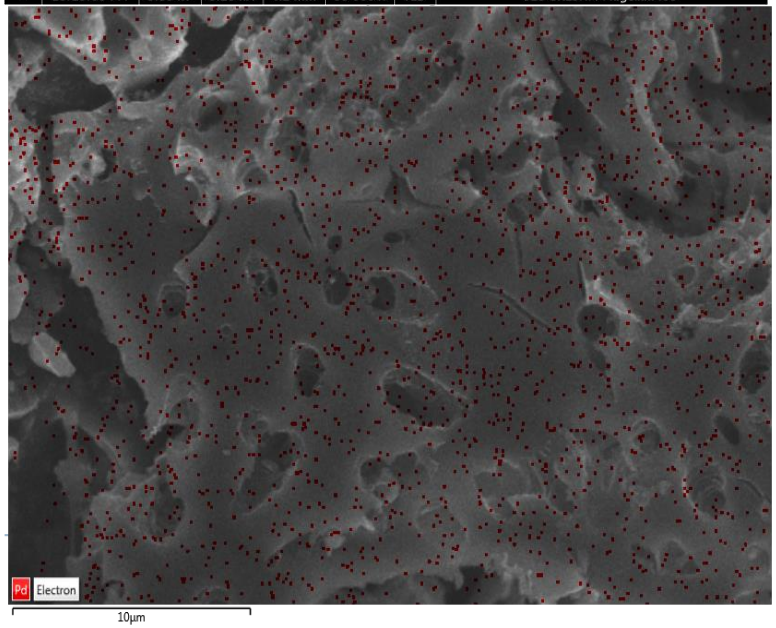
*Pd nanoparticles were obtained using saturation of activated at 250°C microporous carbon with tetrachloropalladous acid - acetone (1:2) solution and reduction procedures in $H_2$/Ar flow at 200°C for 4 hours.*

*The Pd content (EDX data) was about 11 wt.%.*

| Element | Atomic % | Wt% |
|---|---|---|
| C | 90.9 | 80.0 |
| O | 6.8 | 7.9 |
| Al | 0.4 | 0.8 |
| Si | 0.1 | 0.3 |
| Cl | 0.3 | 0.7 |
| Pd | 1.5 | 11.3 |

*SEM images and EDX data of Pd-doped carbon*



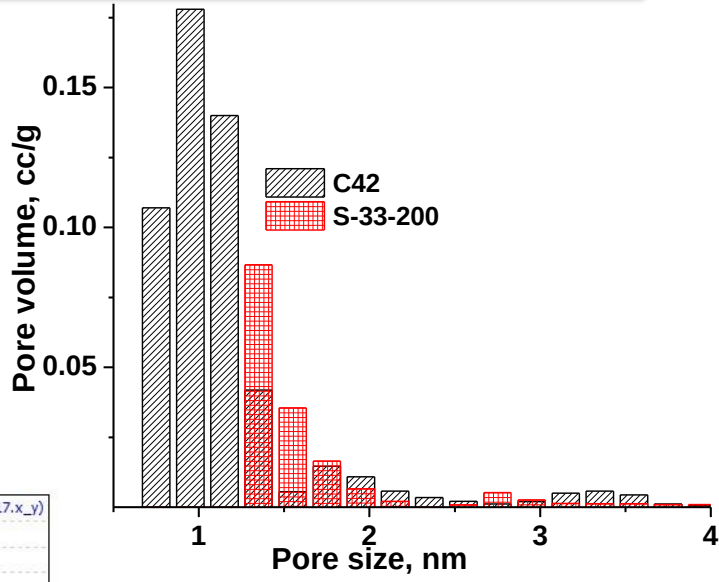
# Pd-clusters in porous carbon matrix

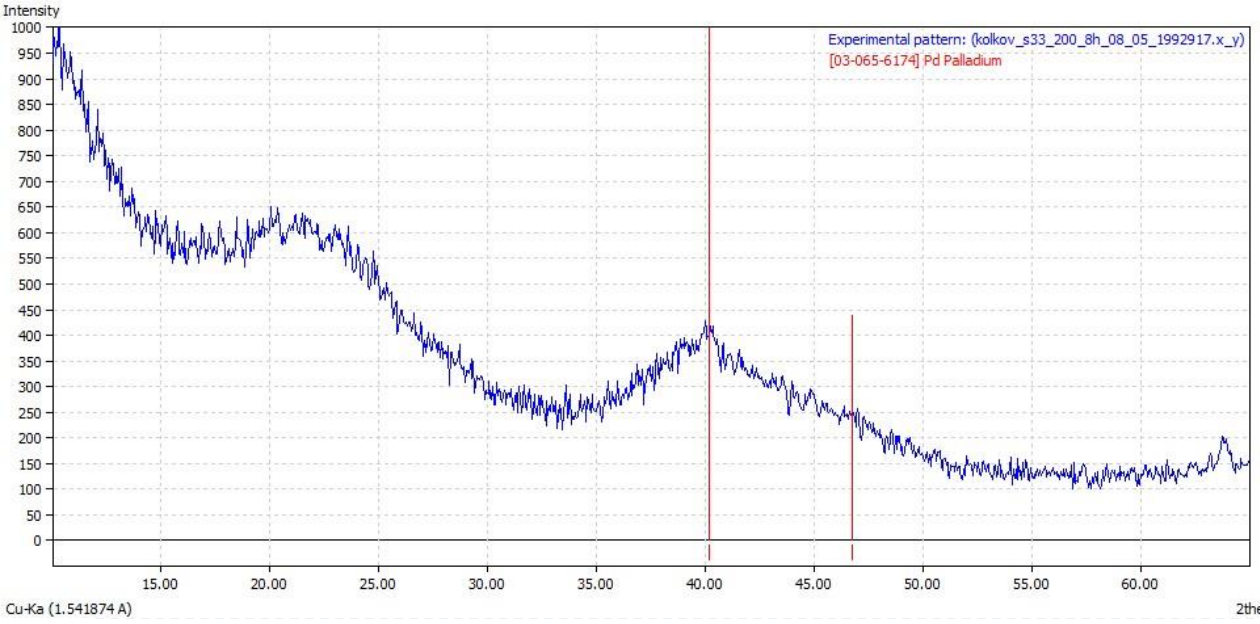
## Syntesis protocol:

*Pd nanoparticles were obtained using saturation of previously activated at 250°C microporous carbon with tetrachloropalladous acid - acetone (1:2) and reduction procedures in $H_2$/Ar flow at 200°C for 4 hours.*

*The Pd content (XRF data) was about 9-11 wt.%.*

*Average size of Pd clusters (XRD data) was about 1.5-1.7 nm*



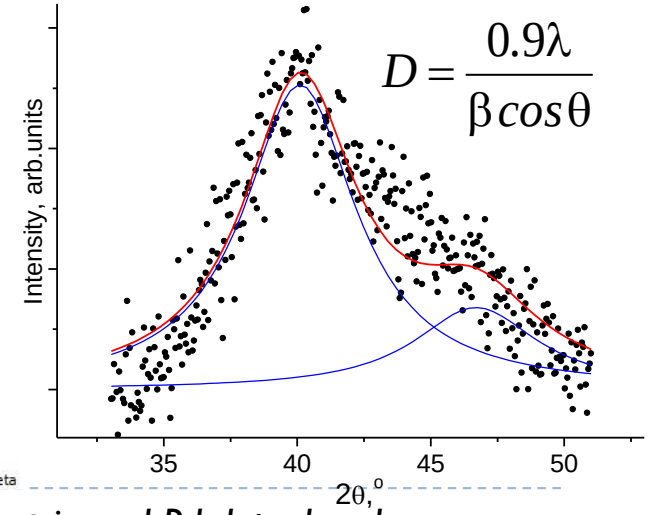
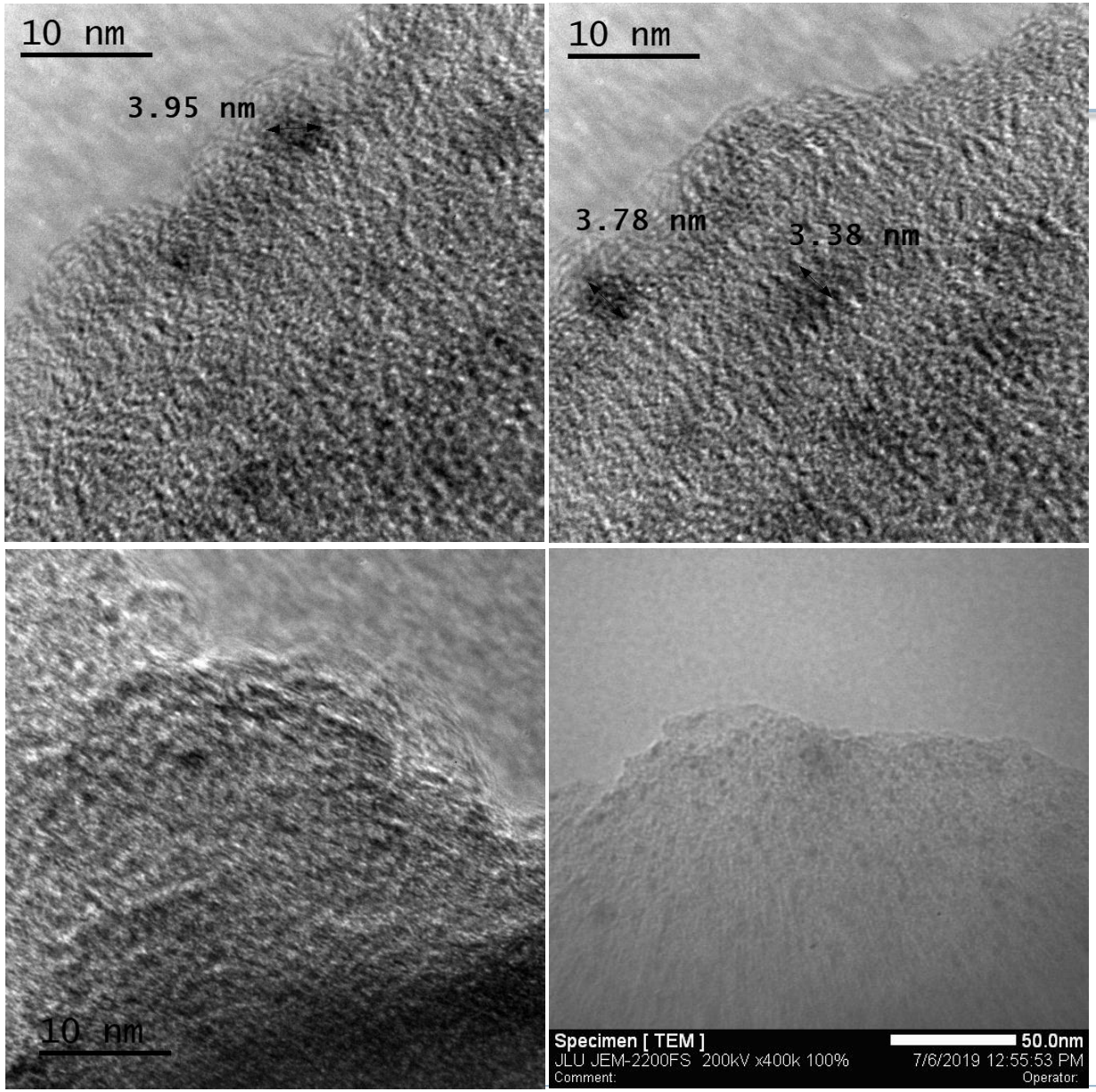



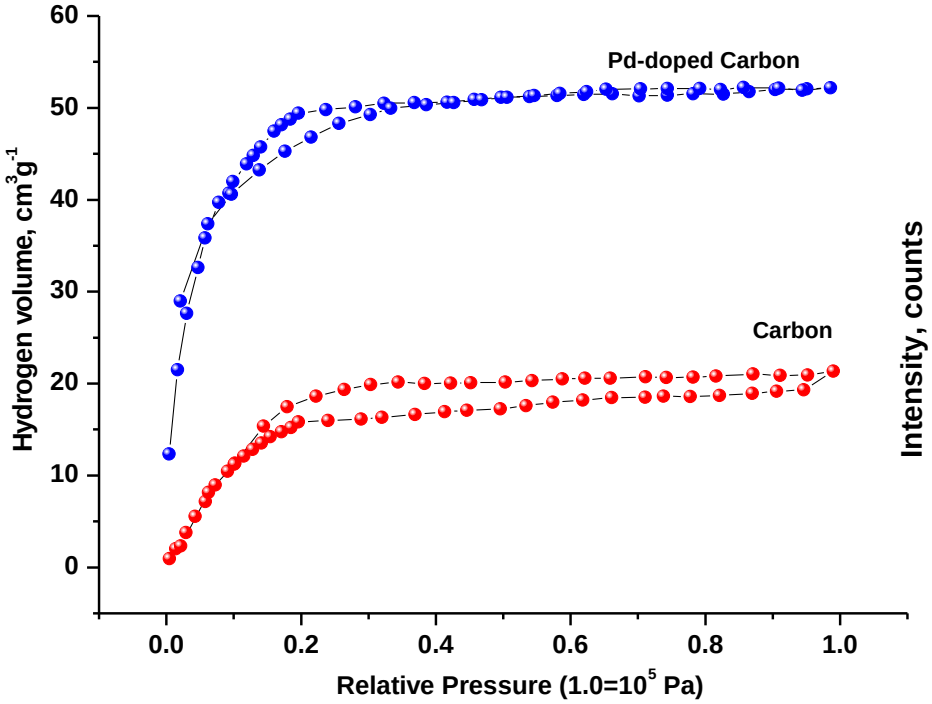

*Fig.1. XRD pattern and pore-size distribution of carbon matrix and Pd-doped carbon*




*Pd-clusters in porous carbon matrix*

*Fig.1.TEM images of Pd-doped carbon*

## *Pd-clusters in porous carbon matrix*



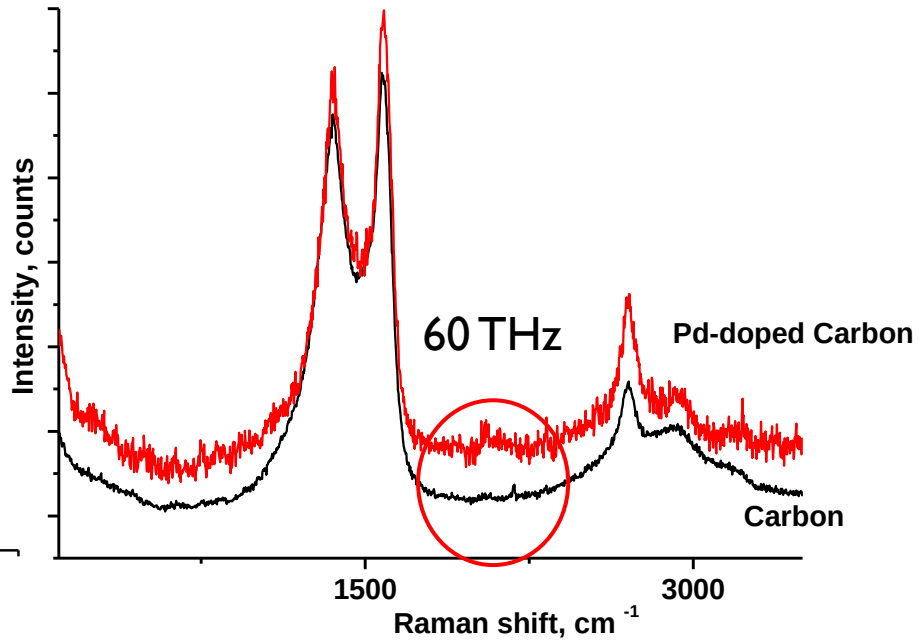
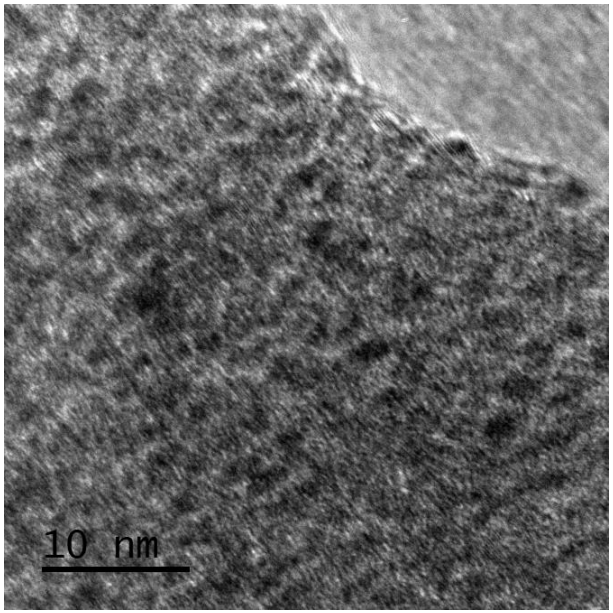



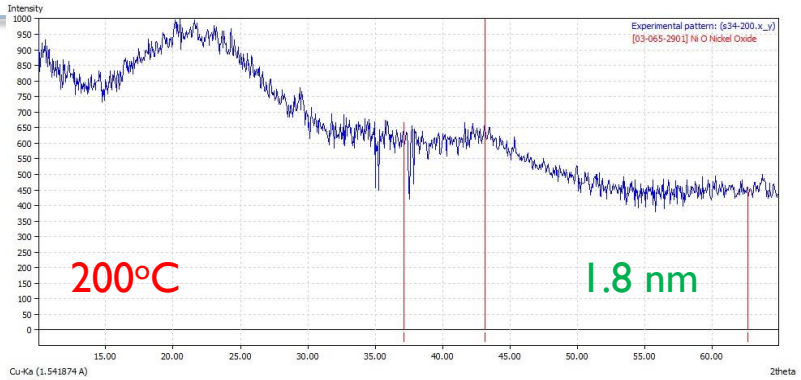

*Fig. 1. Adsorption-desorption isotherm and Raman spectra of Pd-doped carbon*

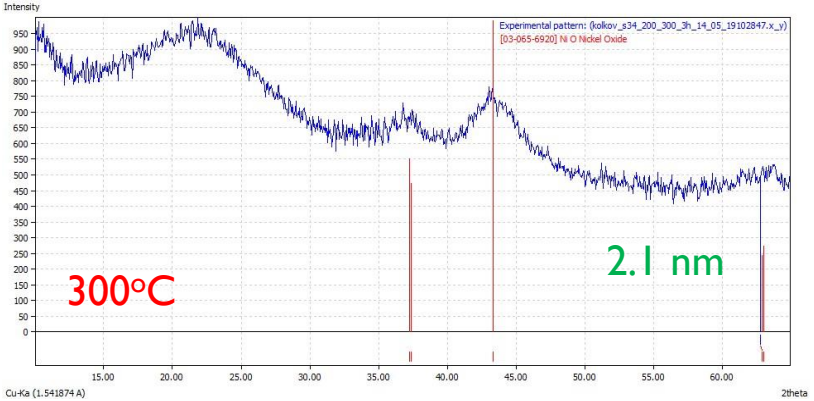
# *Ni-clusters in porous carbon matrix*

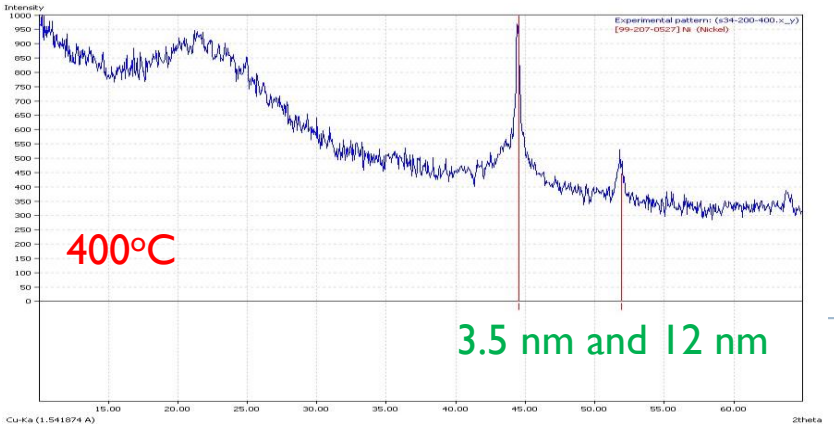
## *Synthesis protocol:*

Activated NaY zeolite was mixed with an nickelocene $Ni(C_5H_5)_2$ and heated at 130°C for 10 h to enable $Ni(C_5H_5)_2$ sublimation and adsorption in the zeolite pores. The $Ni(C_5H_5)_2$ adsorbing zeolite was exposed to ultraviolet light at room temperature for 3-5 days to organic ligand decomposion. The material was reduced under $Ar/H_2$ flow at 300°C.



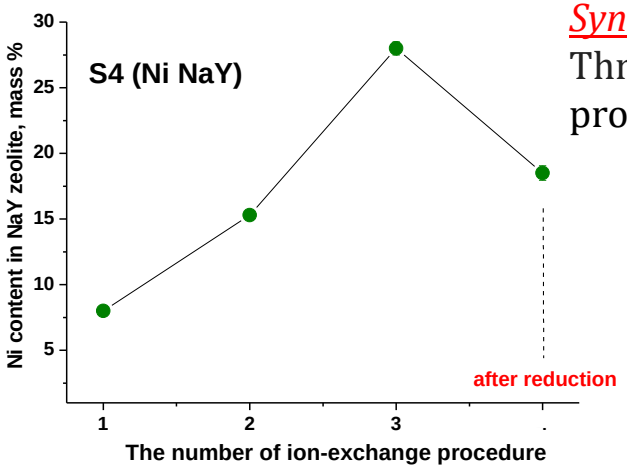




Inokawa, H., Maeda, M., Nishimoto, S., Kameshima, Y., Miyake, M., Ichikawa, T., ... & Miyaoka, H. (2013). Synthesis of nickel nanoparticles with excellent thermal stability in micropores of zeolite. International Journal of Hydrogen Energy, 38(31), 13579-13586.

# *Pd- and Ni-clusters in zeolite matrix*



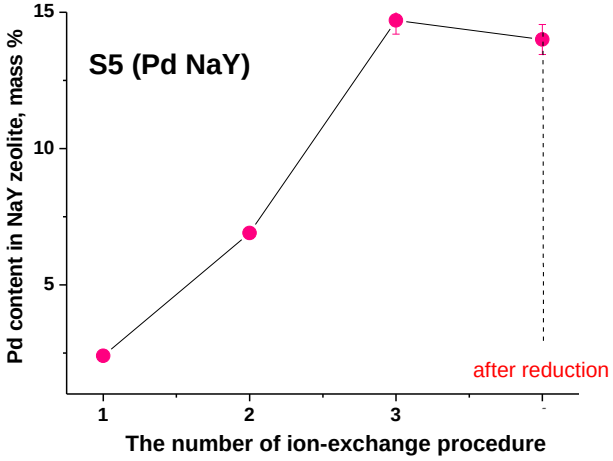

*Syntesis protocol* :

Three ion exchange procedure using Nickel Chloride solution, reduction procedures in $H_2$/Ar flow at 200°C for 4 hours

Final Ni content is about 18.5 mass %

XRD data

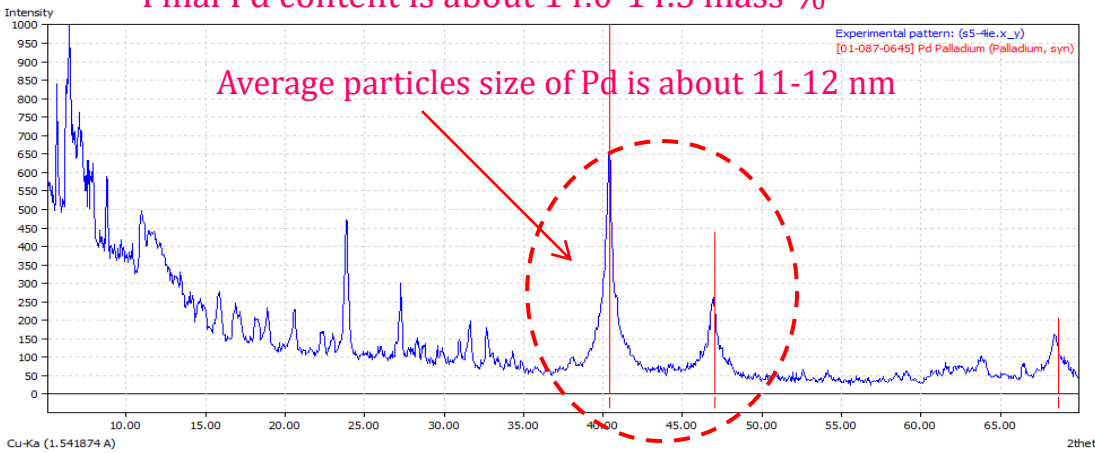
No traces of X-ray crystal nickel-containing phases were observed.




*Syntesis protocol:*

Three ion exchange procedure using $(NH_3)_4Cl_2Pd$ aqueous solution, reduction procedures in $H_2$/Ar flow at 200°C for 4 hours

Final Pd content is about 14.0-14.5 mass %

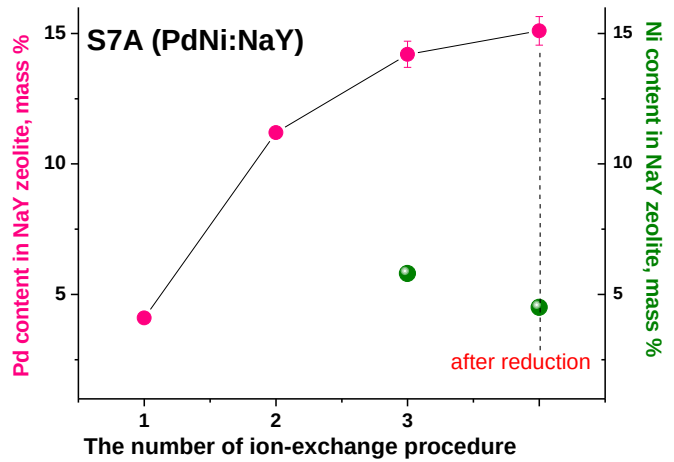


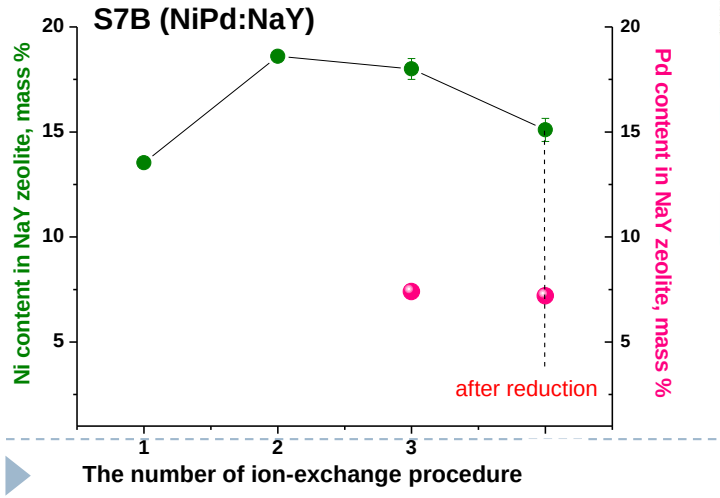
# PdNi-clusters in zeolite matrix

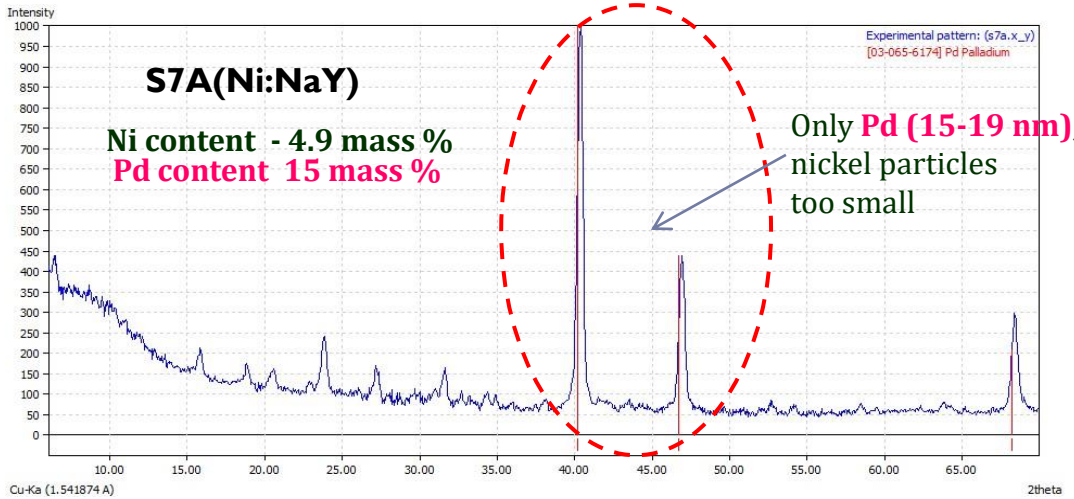
## *Syntesis protocol:*

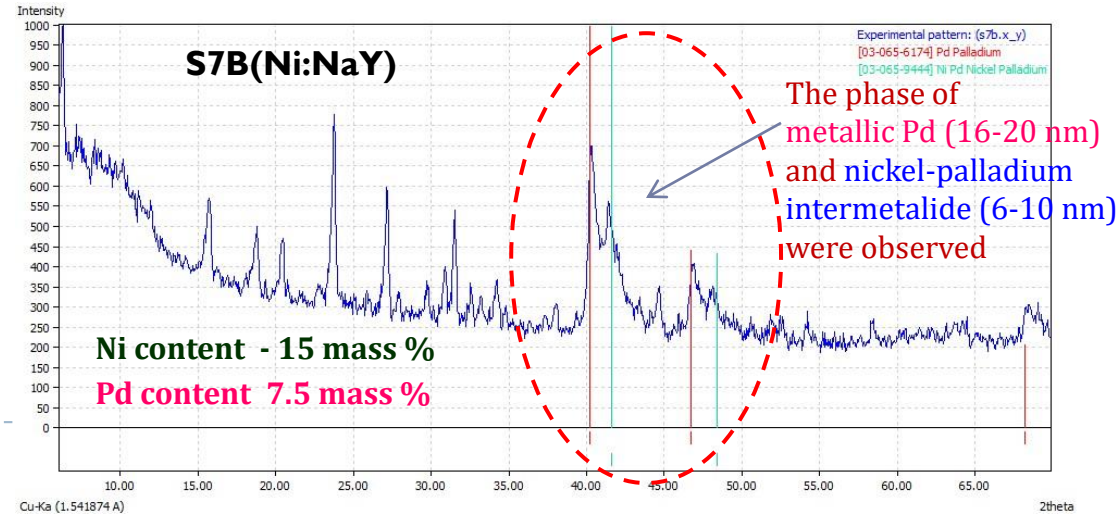
**S7a (Pd-Ni) NaY–** ***two*** ion exchanges with ***tetraamin palladium dichloride*** and ***one*** exchanges with ***nickel acetate***

**S7b (Ni-Pd) NaY** – ***two*** ion exchanges with ***nickel acetate*** and ***one*** exchanges with ***tetraamin palladium dichloride***



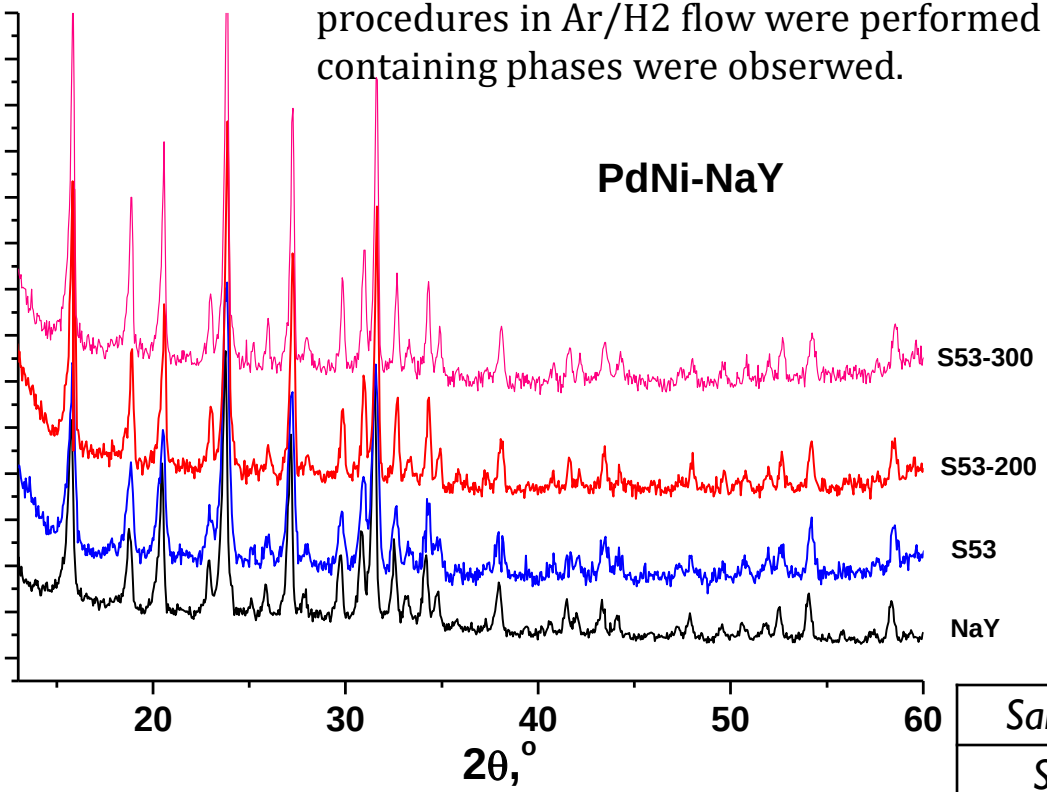



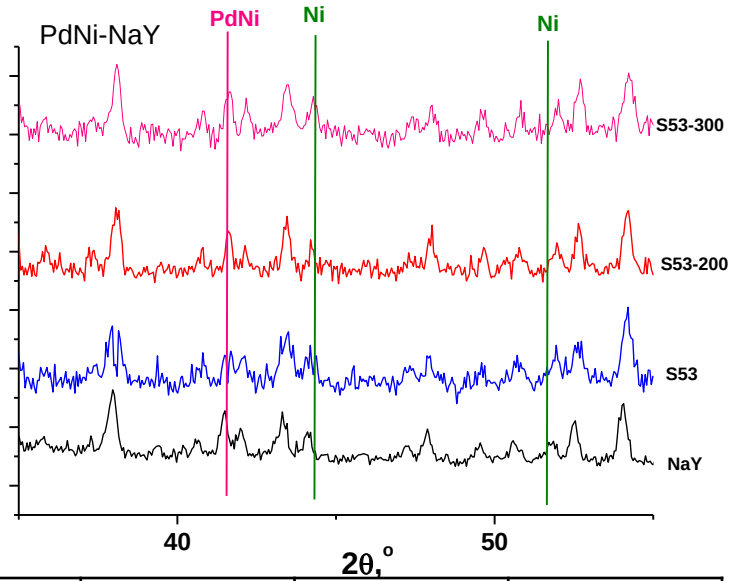



# PdNi-clusters in zeolite matrix

*Syntesis protocol:*

The NaY zeolite was used as a template for adsorption and decomposition of a sublimated organometallic compound (tetraamine palladium chloride and nickelocene). Reduction procedures in Ar/H2 flow were performed at 200, 300 and 350°C. No traces of Pd or Ni-containing phases were obserwed.

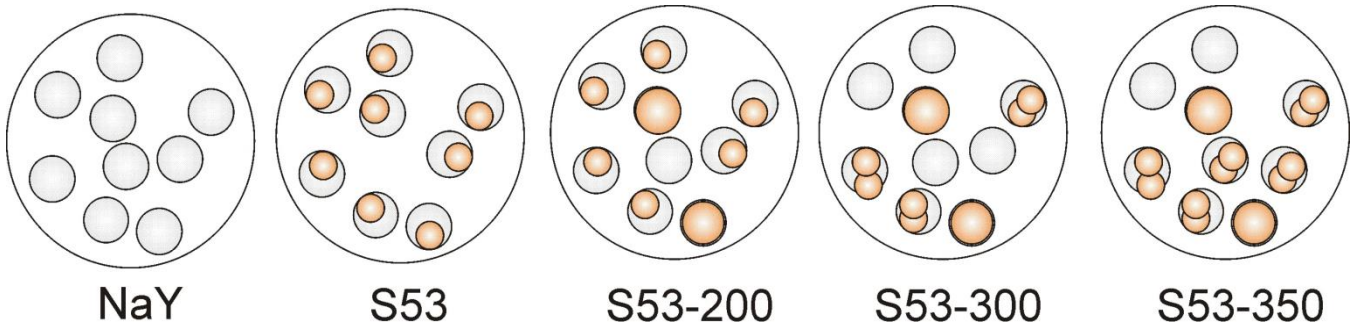


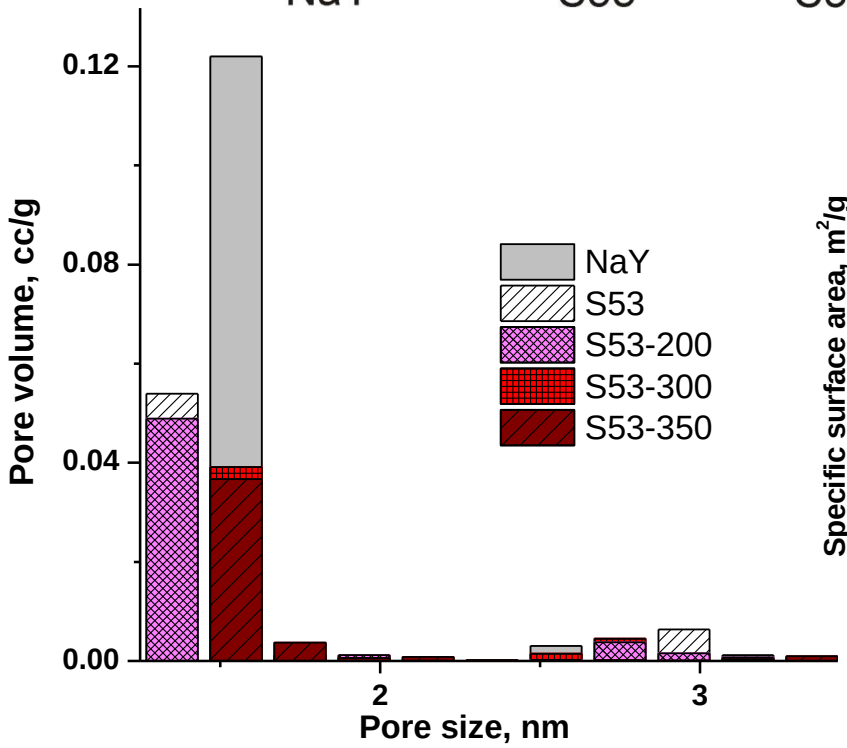



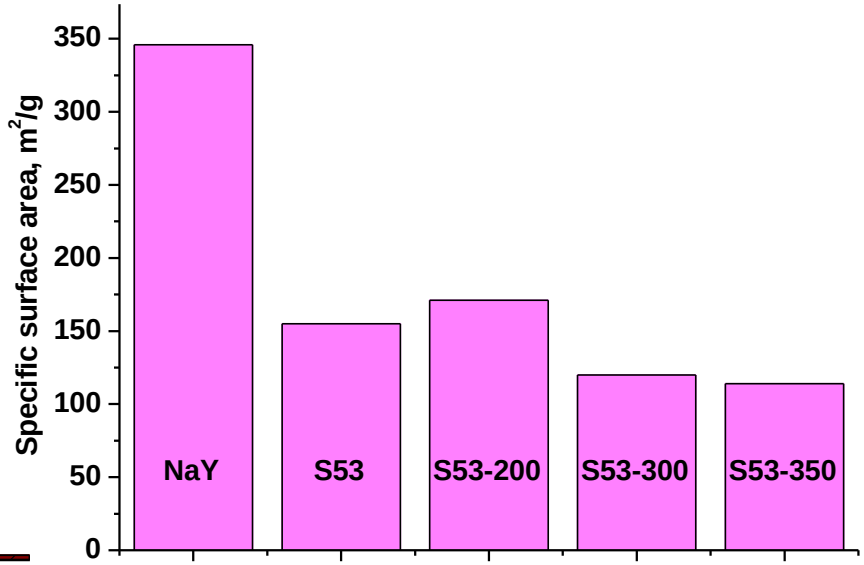

*XRD pattern of pure NaY zeolite and PdNi-doped zeolite after reduction procedure at different temperatures*

| Sample | Pd, mass % | Ni, mass % | Cl, mass % |
|---|---|---|---|
| S53 | 1.0 | 13.6 | 5.0 |
| S53-200 | 0.3 | 7.0 | 4.1 |
| S53-300 | 0.2 | 8.0 | 1.5 |
| S53-350 | 0.2 | 12.0 | 1.8 |

## *PdNi-clusters in zeolite matrix*



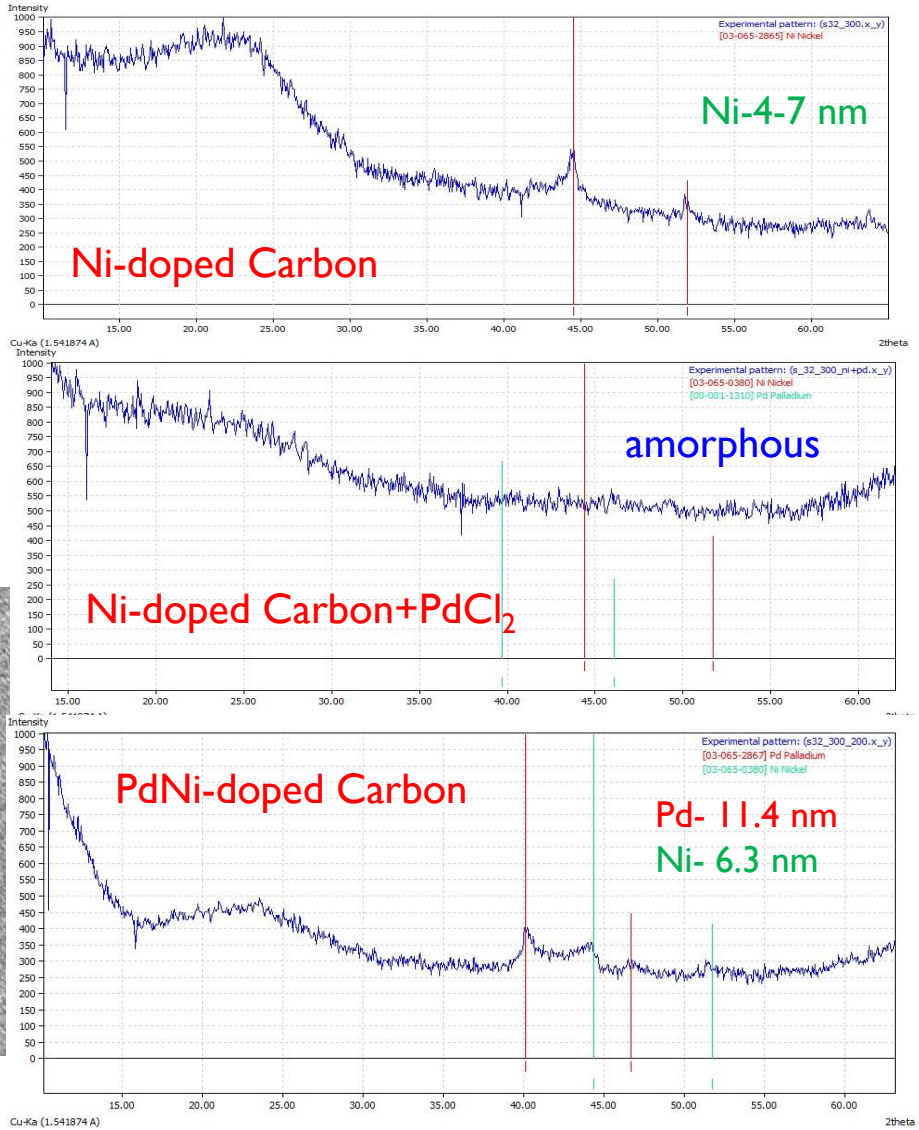

*The evolution of pore structure for PdNi-doped zeolite (S53 sample)*

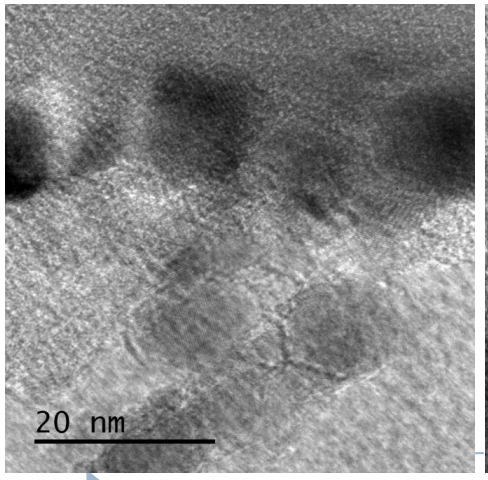
## *Synthesis protocol:*

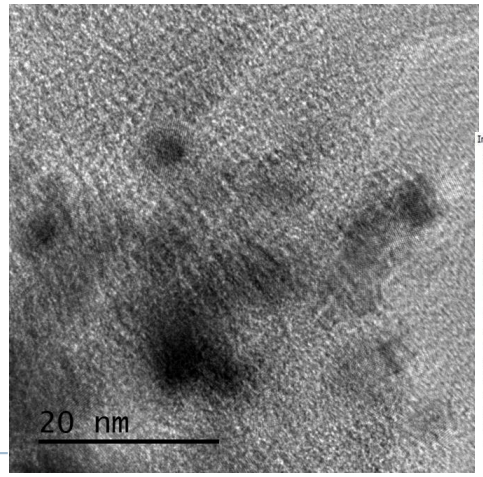
Two stages:

1.Activated carbon was used as a template for adsorption and decomposition of nickelocene with reduction procedures in Ar/H2 flow at 300°C .

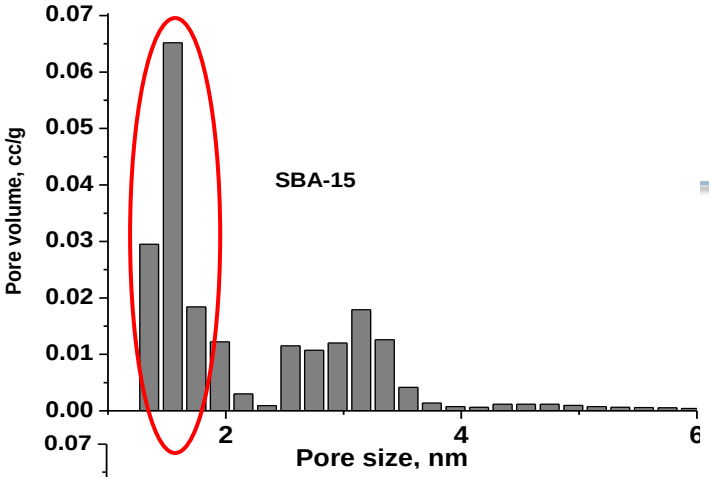
2.Ni-doped carbon was saturated with tetraamine palladium chloride solution reduced in Ar/H2 flow at 200°C

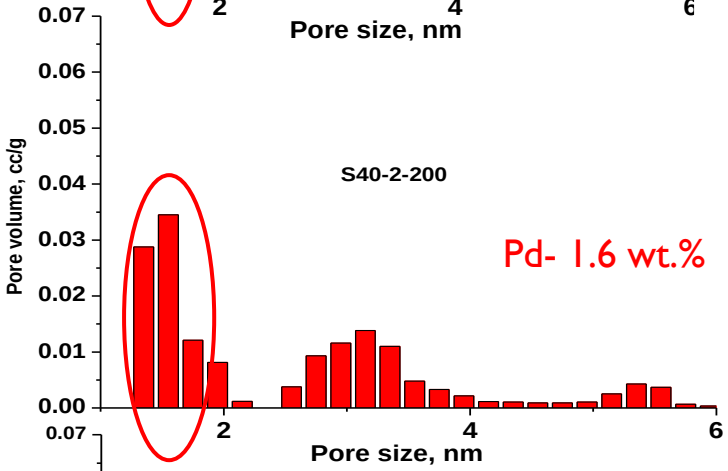


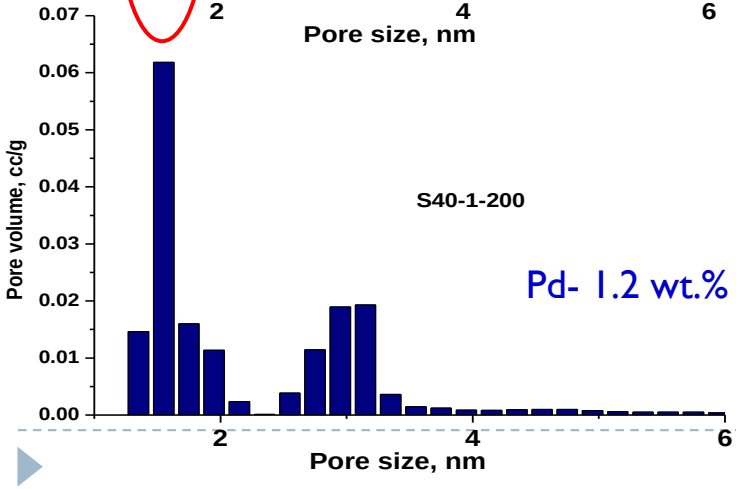



# *Pd-clusters in SBA-15 silica*

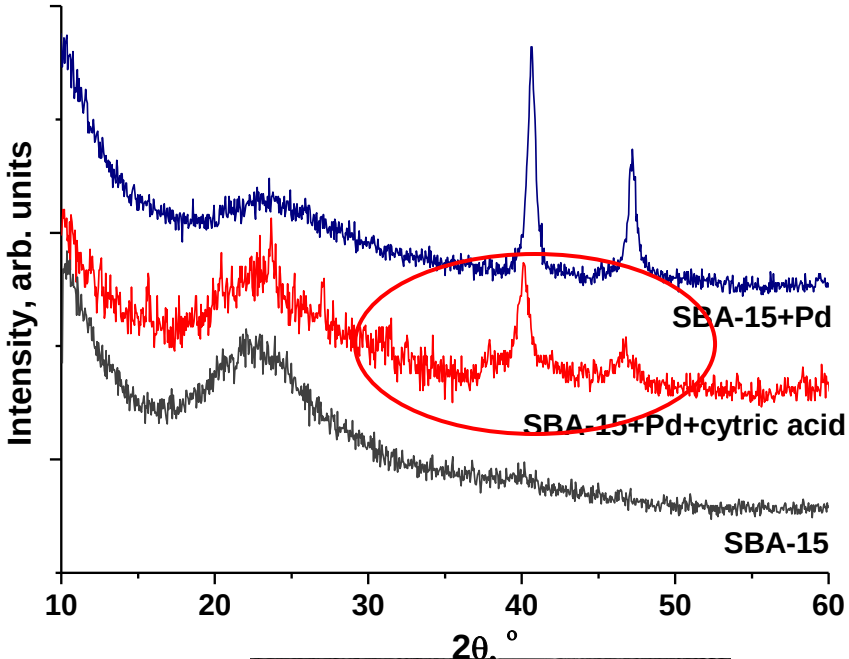



Pd- 1.6 wt.%



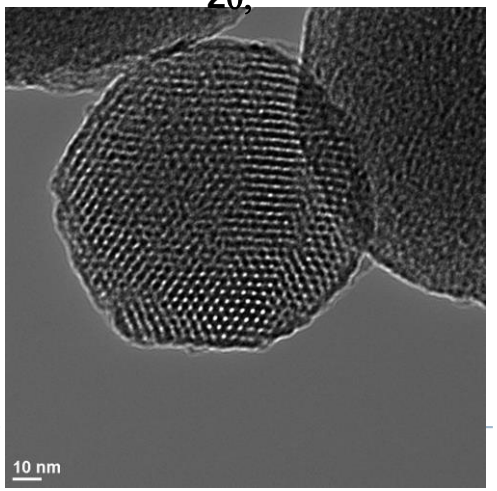

Pd- 1.2 wt.%




12 nm

1.4 nm

12 nm



## *Conclusions*

- *The methods of experimental obtaining of ultrasmall Pd and Ni, and also combined Pd-Ni clusters were successfully approbated.*

- *Porous carbon and zeolite matrixes were used for synthesis Pd and Ni nanoparticles with the average size about 1.5 nm.*

- *The analysis of hydrogen adsorption/desorption for synthesized Ni- Pd- doped carbons and zeolites was realized.*

- *The testing of obtained systems as a perspective LENR active materials with nuclear active sites due generation of discrete breathers or fast phase transformation of quasicrystals was started.*

*Thank you for attention*

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