Production of C–Fe–Pd nanocomposites via Infra-red radiation and its structural characterization

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Abstract. The objective of the present study is to produce C–Fe–Pd nanocomposites using pyrolyzed infrared (IR) radiation. The structural characterization was analyzed using X-ray diffraction (XRD), transmission electron microscopy (TEM) and X-ray fluorescence analysis. In addition, the size of metal nanocomposites as a function of the pyrolysis temperature, and its distribution within the carbon matrix is also characterized.

1. Introduction
Currently, development of different process methods for the production of nanocomposites with tailored properties is one of the priority tasks of modern materials science. The peculiarity of such materials, structural characteristics and functional properties of nanosystems, depending on the conditions for obtaining is an important task. This due to this it opens a new research area that could possibility control the structure and consequently its properties [1].

Bimetalnanosized Fe-Pd particles systems possess excellent catalytic activity, which leads to their use at the present time. Furthermore, the C–Fe–Pd nanocomposites system is receiving increasing attention as it is being used to enhance magnetic media with high recording density and storing information [2].

In this regard, the aim of this work was to obtain the metal-carbon systems with bimetallic Fe-Pd particles, and to study their properties depending on the different temperatures (300, 400, 500, 600, 700 °C).This method for obtaining nanocomposites based on the pyrolysis of organic and inorganic substanceis the most efficient ways of obtaining nanocomposites for the perspective of industrial applications.

2. Experiment
Metal-carbon nanocomposite was obtained by thermal decomposition of the precursor under the influence of infrared heating to install Photon. The precursor was obtained by dissolving PAN, iron salts and palladium in a solution of DMF and subsequent drying. In addition the structural properties of C–Fe–Pd nanocomposites are analyzed under different temperatures (300, 400, 500, 600, 700 °C).
3. Results and discussion

The diffraction patterns of the samples presented in Figure 1 was produced by a qualitative phase analysis. X-ray phase analysis showed the presence in the system IR-PAN/FePd phase of amorphous carbon. The radiographs of the samples determined that at all temperatures the production of nanopowder is formed a solid solution of Fe in Pd.

![Diffraction Patterns](image)

**Figure 1.** The diffraction pattern of the samples.

The obtained samples were further annealed from temperature 200 to 700 °C. At temperature 700 °C, a peak corresponding to palladium, acquires a symmetric form, becomes more intense and narrow, indicating an increase in the size of crystallites and enhancement of the structure (Figure 2). At temperatures 200, 250 and 300 °C the X-ray lines observed with the "rush" from the side of smaller angles. Based on the above results it is assumed that the dissolution of hydrogen in palladium nanoparticles.
Figure 2. X-ray diffraction patterns of C–Fe–Pd nanocomposites.

The lattice constant of FCC-Pd in the nanocomposite IR-PAN/Fe/Pd is less than the values typical for coarse-grained FCC-Pd due to specific features of nano state of the metal (Table 1).

Table 1. The values of gratings Pd in a coarse-grained and nanocrystalline samples.

| Sample              | Lattice constant a (nm) |
|---------------------|-------------------------|
| Pd (nano)           | 0.3881                  |
| Pd (array)          | 0.3890                  |

According to the difference of periods of the lattice of palladium in coarse-grained and nanocrystalline states has been determined dependence for nanosized materials, which was performed quantitative estimation of the composition of the formed metallic phase. The concentration of iron in the solid solution increases with increasing pyrolysis temperature. This is evidenced by the shift in the peak intensity on X-ray film in the region of large angles and a decrease in grating period phase of palladium (Table 2).
Table 2. The lattice period for phase Pd under different temperature

| Annealing temperature, °C | The parameter a (nm) |
|--------------------------|----------------------|
| 300                      | 0.3885               |
| 400                      | 0.3863               |
| 500                      | 0.3855               |
| 600                      | 0.3845               |
| 700                      | 0.3833               |

To calculate the size of coherent scattering regions the Selivanov–Smyslov method was used for the broadened diffraction peaks. At 500 °C and 600 °C the peaks shifted strongly to higher values, hence the average size also increased drastically. In addition, it is clear from the Figure 3 that the increase in temperature leads to increase the crystallites size.

Figure 3. Distribution of crystallites size.
At 500 °C and especially at 600 °C graphics strongly blurred to higher values, the average size also increased drastically. As can be seen from the obtained data, the temperature increase leads to an increase in the size of crystallites.

Figure 4 shows the transmission electron microscopy micrograph of the obtained samples. It is clear from the images that the metal nanoparticles (dark areas on the micrograph) are rounded and encapsulated in a polymeric matrix (the gray area). In some areas there are coagulation of the particles also observed (Figure 4). The photomicrographs also reveals that the polymer matrix has a layered structure.

**Figure 4.** TEM images of C–Fe–Pd nanocomposites at different temperature (a) 400°C and (b) 500°C.

The image shows that there are large dark thickened backdown present. The large thickness are polymer layers or larger particles of the metallic phase. In the course of obtaining the nanocomposite undergoes various reactions and structural transformations, the nature and sequence of which may change with increasing temperatures, at which the gain material. As a consequence, in some areas there may be conditions for coagulation of the particles.

The photographs, obtained with the electron microscope, was used to measured the size of metal particles. Based on the data obtained histograms showing the distribution of nanoscale particles. The obtained results, reveals that with increasing pyrolysis temperature, the size of nanoparticles increases. This may be due to the coagulation of particles and the growth rate increases with increasing temperature of pyrolysis. The experimental curve (Figure 5) shows clearly that there is a long "tail" to larger particles, which is described by logarithmically normal law.
Figure 5. Histogram of size distribution of particles in the sample obtained at 500 °C.

References
[1] Ozkana S Zh, Dzidzigurib E L, Karpacheva G P, Bondarenko G N 2011 J. Nanotechnologies in Russia 6 750
[2] Bagdasarova K A, Perov N S, Karpacheva G P, Pile S E, Dzidziguri E L 2011 J. Advanced Materials 168 349