

The use of ionic implantation in the synthesis nano palladium for biosensors

Abstract

In the work the possibility of primary ionic implantation using in the synthesis of palladium nanoparticles in the aspect of potential biosensors preparation was studied. The phase composition of implants was performed by means of XRD. The morphology of the samples surface was studied using optical microscopy and SEM. It was shown that after the implantation of molybdenum, nickel, and aluminum ions, it is possible to synthesize palladium nanoparticles in wide range of the sizes. It was established that the dispersion of the layer of the active component depends from the nature of the target ions and the number of treatments. It was shown that the nanoscale layers obtained by ionic implantation have significant prospects for palladium nanoparticles preparation and use as biosensor materials.

Keywords: biosensor materials, nanomaterials, nanoparticles, palladium, ionic implantation, XRD, SEM, optical microscopy

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Introduction

The rapid development of biosensor technologies and the expansion of the requirements determine the necessity for scientists to search of new materials and new technologies.¹⁻⁵ Moreover, these problems are caused not only by the interest of scientists in this topic, but also by an increase attention of industrial and medical workers.⁶ The development of biology, pharmacy, medicine and, in particular, biochemical and biophysical methods of analysis and control leads to the search for new designs of electrochemical, photosensitive, thermal sensors and biosensors.^{3,7-9} Recently, a promising direction in biosensor material science is the use of nanomaterials, such as nanoparticles, nanocomposites, nanotubes, etc.^{3,4,7,8,10} The most common non-organic components of nanoparticles are precious metals (Au, Pt, Pd) deposited on various supports (steel, polymer, etc.).^{3,4,7,8} However, the effectiveness of biosensors also depends on the technology of active particles producing. In this respect, ionic implantation proved to be very successful. This technology permits to modify the surface layer of various materials – supports (most often metals, alloys) by introducing target ions (Ti, Ni, Cr, Al, etc.)¹¹⁻¹⁴ and thereby create substrates for the active components deposition. Thus, the aim of the work is to study the possibility of ionic implantation using in the synthesis of palladium nanoparticles in the aspect of obtaining materials for biosensors.

Synthesis of samples and methods of investigation

Synthesis of the samples was carried out by ionic implantation. Ionic implantation is the technology of implanting target ions (Ti, Ni, Mo, Al, etc.) into the support surface. The process takes place in a vacuum (the pressure is about 10⁻⁴ Pa) in an electromagnetic field of high intensity (the field strength is about 20-40 kV/m). In result, the target ions are accelerated to high energies and penetrate the material to a depth of 1 μm, changing the properties of the surface layer. In this work, nickel (Ni), aluminum (Al) and molybdenum (Mo) ions were implanted in a stainless steel support (SS). Some of these samples, in

addition, were treated with aluminum ions (Al) and after implantation part of them were impregnated with palladium (Pd). The obtained samples are designated respectively Mo/SS, Ni/SS, Ni/Al/SS, Pd/Mo/SS, Pd/Ni/SS, Pd/Ni/Al/SS.

To study the state of the surface optical microscopy (microscope "MIM-7", additionally equipped with digital camera "Kodak Easy Share C1013"), XRD (diffractometer Philips PW 1830) and SEM (Hitachi S-4000) were used.

Results and discussion

Figure 1, as typical example, the diffractogram of a sample synthesized by impregnation a second component (Pd) onto a molybdenum implant (Mo/SS) is shown. It is possible to see that only reflexes of austenite (support) and compounds of deposited metal (phases of metallic palladium and its oxide) were observed in the X-ray. It is necessary to note that the absence of implants reflexes for substrates (Mo/SS, Ni/SS, Al/SS) with Mo, Al, Ni implanted was shown in.^{11,14} For palladium crystals, there are reflexes of planes (111), (200), (220) and (311).¹⁵ The calculation by the use of the Scherrer equation (plane (111) with the maximum reflex intensity) shows that the size of the primary crystals Pd is 19-21 nm, but they can form larger aggregates (crystallites). Low-intensity reflexes of palladium oxide refer to planes (101) and (111).¹⁶ The calculation shows that palladium oxide exists in the form of larger crystals (about 86 nm) than the metal. Thus, the deposition of palladium does not lead to a change in the state of the implanted metal, whose reflexes are still absent in the diffractogram. The study of substrates surface using optical microscopy shows that the implantation of nickel ions (Figure 2) and molybdenum (Figure 3) leads to a double effect (two different samples are presented for each of the elements, synthesized under the same conditions). The fact that the effect of the formation of secondary defective structures is related to the nature of the implant is confirmed by the data obtained for samples that were synthesized by consecutive implantation of aluminum and nickel (Figure 3). On

Ni/Al/SS secondary defects occur in nickel implantation and this effect is increased in comparison with mono-implanted nickel. The emerging combs acquire larger values from height and area of their surface. When palladium is deposited, for example, where aluminum and nickel have been previously sequentially implanted, the grouping of defects is larger and less uniformly distributed over the surface (Figure 4). This is explained by the concentration of new formations near defects that occur after the implantation of nickel.

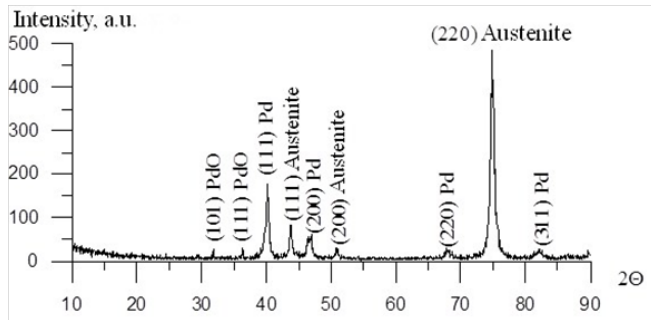


Figure 1 Diffractogram of Pd/Mo/SS sample.

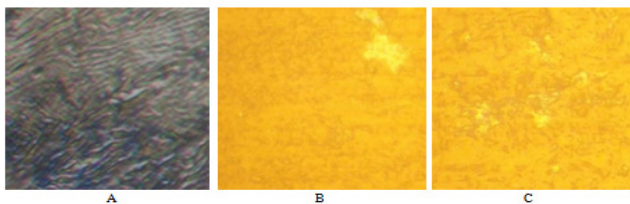


Figure 2 Microphotographies of stainless steel foil (A), Ni/SS (B, C). An increase of 1,500.

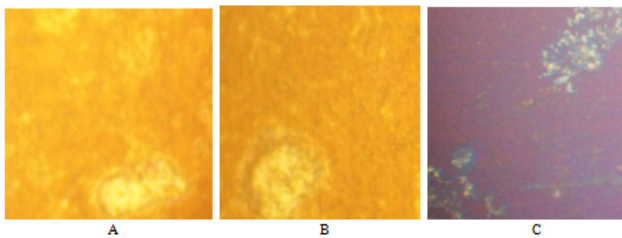


Figure 3 Microphotographies of Mo/SS (A, B), Ni/Al/SS (C). An increase of 1,500.

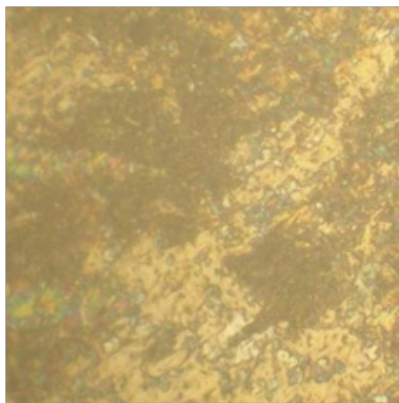


Figure 4 Microphotography of Pd/Ni/Al/SS. An increase of 1,500.

Since the resolution of the visible microscopy was small for the determination of local changes in the surface morphology during metal implantation. So, a study of samples was carried out using the SEM method. In accordance with the data obtained earlier, the results of SEM also show that the implantation of nickel leads to the formation of new, sufficiently large defects in the surface structure (Figure 5). At the same time, the number of these defects and their depth increase in the case of preliminary aluminum implantation in the surface layer of the carrier (Figure 5). That is the heterogeneity of the surface of the synthesized implants greatly increases. When palladium is deposited by impregnating on surface of samples with implanted nickel or with consecutive implanted aluminum and nickel the formation of its individual particles (metal and its oxide, according to the XRD given above) having a globular form was observed (Figure 6). In the case of Ni/SS substrate use, the distribution of particles of palladium (its oxide) in size is more uniform, from 300 to 500 nm. In the second case (using a substrate with successively implanted two elements), the difference in the sizes of the palladium particles is a greater and their dimensions change from 100 to 600 nm (Figure 6). Perhaps this is due to more heterogeneity of the substrate surface when two metals are implanted. At the same time was established that large particles of palladium (larger than 500 nm) present conglomerates from primary particles having a diameter of 50-100 nm (Figure 7) which confirms the calculations from XRD data given above. Thus, the realized studies shown that preliminary implantation of metal ions (Ni, Al, Mo) into steel creates the conditions for formation of palladium nanoparticles rounded form with sizes up to 100 nm on the support surface easy impregnation method. In the case of palladium deposition on support without preliminary metal implantation the indefinite particles of palladium with dimension more than 1 cm were formed (Figure 7B). So, the preliminary implantation permits to create particles of the smallest size.

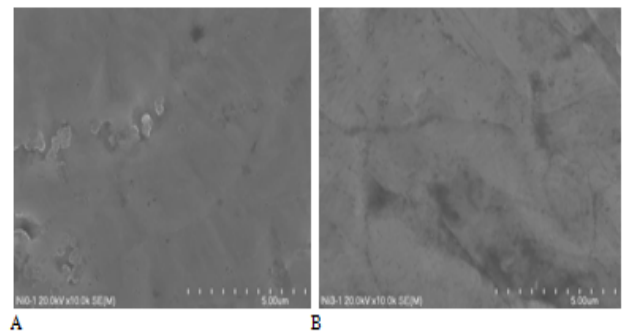


Figure 5 SEM patterns of Ni/SS (A), Ni/Al/SS (B)

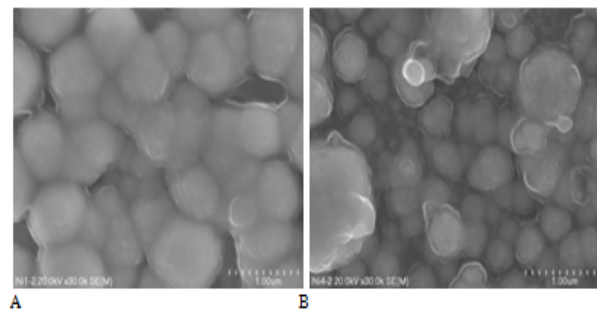


Figure 6 SEM patterns of Pd/Ni/SS (A), Pd/Ni/Al/SS (B)

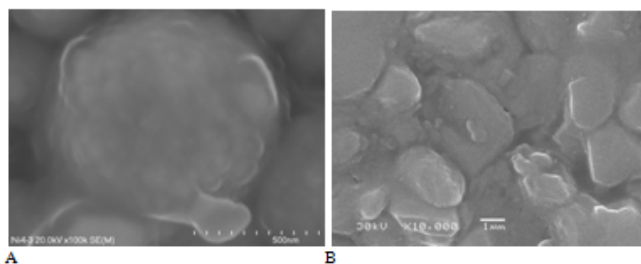


Figure 7 SEM pattern of Pd/Ni/Al/SS (A) and Pd/SS (B).

Conclusion

The supported nanoparticles of palladium (metal and oxide) on the steel surface were synthesized by use of preliminary metals (Mo, Ni, Al) implantation method. It was established that the dispersion of the layer of the active component depends on the nature of the target ions and the number of treatments. It was shown that the nanoscale layers of palladium obtained with preliminary ionic implantation has significant lower particles sizes that in case of deposition of palladium only and the prospects for use as biosensor materials.

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Conflict of interest

Authors declare that no have any conflict of interest.

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