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Characterization of the morphology and chemical composition of Fd_xPd_{1-x} nanorods by advanced FEGSEM and low-voltage EDS analyses

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Fe-Pd alloys have many potential applications due to their unique chemical and magnetic properties, which can be tailored by changing their composition. While the equiatomic composition is interesting due to its high magnetocrystalline anisotropy, the $Fe_{70}Pd_{30}$ has the potential to be used as a magnetic shape memory alloy, which is based on phase transformation from disordered austenite (fcc) to ordered martensite (fct) phase [1,2]. However, disordered Fe_xPd_{1-x} has a narrow fct-range ($x \approx 0.68–0.70$), and therefore an accurate compositional analysis is of vital importance. In this work we have applied advanced, high-resolution, field-emission-gun, scanning electron microscopy (FEGSEM) and elemental analyses using energy-dispersive X-ray spectroscopy (EDS) to study the morphology and the composition of the Fe-Pd nanorods that were prepared via the template-assisted electrodeposition method using an Al_2O_3 membrane with a 200-nm pore diameter. A JEOL JSM-7600F FEGSEM equipped with an Oxford INCA Energy 350 System and an EDS-SDD X-max-20 silicon drift detector were used for overall analyses. Our main aim in this work was to achieve a consistent submicrometer-scale characterization of the Fe-Pd nanorods utilizing modern, high-performance, SEM/EDS analytical equipment.

SEM micrographs of the FePd nanorods were recorded using various beam-voltages from 5 kV to 15 kV and beam currents between 0.3–0.8 nA with microscope column conditions being carefully optimized for high-resolution imaging. Particularly useful was the backscattered electron (BSE) imaging in compositional contrast mode, which revealed major compositional changes along the rods, as shown Figure 1. The short, “brighter”, initial part of the rods was found to be a Pd-Pt rich region near the edge of the Pt-deposited membrane, which ends with a distinct border towards the grey, regular part of the Fe-Pd deposited alloy. High-resolution top-view image of the morphology of the Fe-Pd nanorods after dissolving the Al_2O_3 membrane is shown in Figure 2 revealing that the nanorods end with uniform, flat surface.

Quantitative compositional analyses were carried out using the low-voltage EDS approach by analysing the Fe-L□ and Pd-L□ low-energy spectral lines. The comparison of the EDS spectra acquired at three accelerating voltages 8, 6.5 and 5 kV from analytical point #4 (see Figure 1) is shown in Figure 3. The Al and O signals that originate from the Al_2O_3 membrane increase with voltage due to the larger X-ray excitation volume at higher voltages. However, in this case the true stoichiometry of the Fe-Pd nanorod and the corresponding Fe/Pd atomic ratios can be determined by normalization of the Fe and Pd concentrations. The atomic Fe/Pd ratios obtained from the quantitative EDS analyses at 10 discrete points along a Fe-Pd nanorod (shown in Figure 1) are given in Figure 4 and reveal consistent results for all the applied voltages. The advantage of low-voltage EDS is the high spatial analytical resolution due to the reduced, submicrometer-sized, X-ray excitation volume at low beam energies. This allowed us to measure the composition and/or compositional variations along the Fe-Pd nanorods on a submicrometer scale with achieved lateral analytical resolution of ≈ 0.2 □m, which is comparable to the diameter of the nanorods. In selected sample quantitative EDS analysis clearly showed that considerable Fe and Pd concentration gradient along the nanorods is present. In conclusion we found that a high-resolution FEGSEM imaging combined with low-voltage EDS analyses are very appropriate tools for reliable analyses of the morphology and chemical composition of the electrodeposited Fe-Pd nanorods [3].

1. X.L. Fei *et al*, Solid State Communications 141 (2007) p. 27.
2. N. Tasaltin *et al*, Journal of Alloys and Compounds 509 (2011) p. 4701.
3. The authors acknowledge the support of this work from Slovenian Research Agency (ARRS) within project J2-4237.

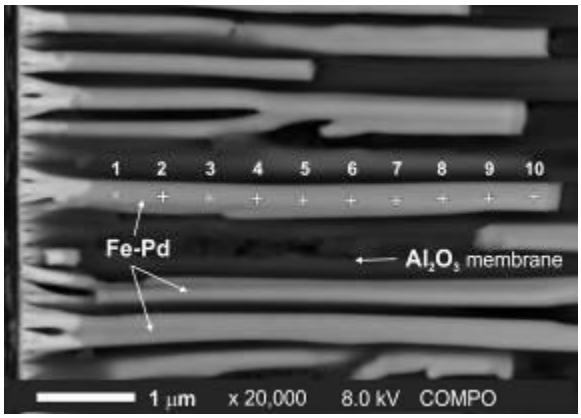

Figure 1. FEGSEM BSE micrograph of the Fe-Pd nanorods in Al_2O_3 membrane. Marked points 1-10 show the positions of point-beam EDS analyses along the nanorod.

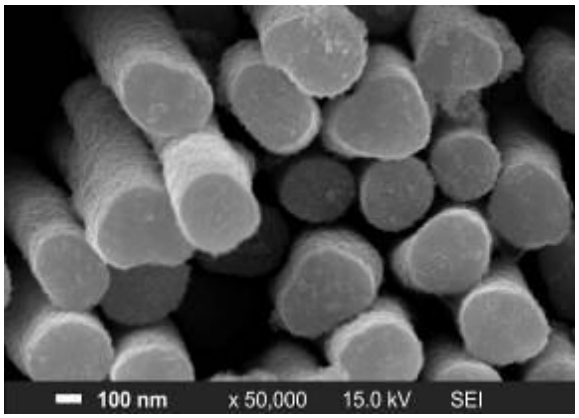

Figure 2. FEGSEM secondary electron micrograph of the Fe-Pd nanorods after dissolving the Al_2O_3 membrane

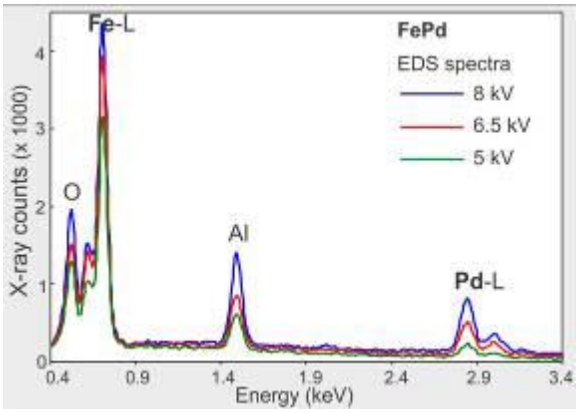

Figure 3. Comparison of low-voltage EDS spectra acquired at 8, 6.5 and 5 kV from the point 4, marked in Figure 1.

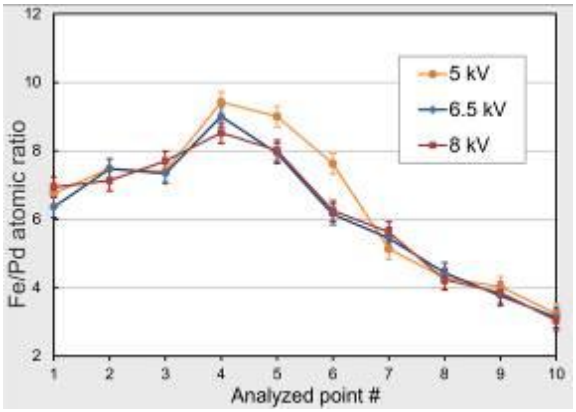

Figure 4. Variation of the atomic Fe/Pd ratios between the points 1-10 on the nanorod shown in Figure 1, calculated from the results of quantitative EDS analyses.