Influence of surface topography on RBS measurements: case studies of (Cu/Fe/Pd) multilayers and FePdCu alloys nanopatterned by self-assembly

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Abstract
In this paper the influence of surface topography on Rutherford backscattering spectrometry (RBS) is discussed. (Cu/Fe/Pd) multilayers with total thickness of about 10 nm were deposited by physical vapor deposition on self-organized array of SiO₂ nanoparticles with the size of 50 nm and 100 nm. As a reference, the multilayered systems were also prepared on flat substrates under the same conditions. After the deposition, morphology of the systems was studied by scanning electron microscopy (SEM), while chemical analysis was performed using Rutherford backscattering spectrometry. It was found that the RBS spectra and determined compositions for flat and patterned multilayers differ. The difference is discussed by taking into account the effect of additional inelastic scattering and energy straggling occurring due to developed topography of patterned systems. Then, the multilayers were annealed in 600 °C in order to obtain FePdCu alloy. The phenomenon of solid-state dewetting resulted in the formation of isolated alloy islands on the top of SiO₂ nanoparticles. The SEM and RBS analysis were repeated showing correlation between the size distribution of obtained alloy islands and broadening of peaks appearing in RBS spectra.

Keywords: RBS, patterning, nanostructures, dewetting, FePdCu alloy
Classification numbers: 2.01, 4.02, 4.11, 5.02, 5.12

1. Introduction
Development of sensors, transistors, recording media, and other electronic components and devices is connected with synthesis and use of innovated patterned films with at least one characteristic dimension measured in nanometers. Their size allows them to exhibit novel and significantly improved physical properties and phenomena. When characteristic structural features are in the range of about 100 nm, the objects often exhibit physical attributes substantially different from those revealed by bulk materials. Therefore, the new patterned films are extensively studied by chemists, physicists and engineers.

However, the characterization of patterned materials is challenging because of the difficulties in adapting conventional experimental techniques to samples with 3D topography. In particular, this problem concerns scattering techniques such as Rutherford backscattering or x-ray reflectivity. Additional scattering introduced by nanostructures causes new features in spectra making conventional analysis of data difficult or even impossible. Therefore, new approaches and methodologies for conventional techniques must be developed to determine the properties of patterned materials.

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2. Experimental

In order to prepare nanopatterned FePdCu samples, Fe, Pd and Cu metals were deposited on substrates coated with SiO₂ particles (commercially available from Polysciences, Inc.) with the size of 100 nm and 50 nm. Arrays of these particles were prepared on flat Si(100) surfaces using a method proposed by Micheletto et al [17], which based on deposition of a droplet of particle-water mixture onto a carefully cleaned substrate. Before the application of nanoparticle solution, it is important to ensure good wetting properties of the substrates, providing sufficient attractive capillary forces during the self-assembly process. Therefore, the Si wafers were washed in an ultrasonic cleaner for 30 min in a solution of hydrogen peroxide and ammonia at 80 °C. The solution was prepared by mixing 1 part of 30% hydrogen peroxide, 1 part of 25% ammonia and 5 parts of distilled water. Then, the substrates were transferred into a mixture of hydrogen peroxide and hydrochloric acid placed in an ultrasonic cleaner, where they were rinsed for 10 min at 80 °C. The composition of the second solution was as follows: 1 part of 30% hydrogen peroxide, 1 part of 36% hydrochloric acid and 6 parts of distilled water. In the last stage, substrates were washed with distilled water for 15 min at room temperature, and then dried with nitrogen. Shortly after this preparation process, a droplet of the SiO₂ nanoparticle solution was placed onto the Si substrate. During the evaporation of solvent a hexagonal close packed particle array was formed.

The Si substrates with arrays of SiO₂ particles were inserted to ultra high vacuum preparation chamber and (Cu/Fe/Pd) multilayers were deposited. The (Cu(t nm)/Fe(0.86 nm)/Pd(1.14 nm)) × 5 multilayers (shown schematically in figure 1) were deposited by sequential thermal evaporation. The thickness of Fe and Pd layers was chosen as corresponding to 48:52 atomic stoichiometry, necessary to obtain the L₁₀ structure. Copper was introduced into each Cu/Fe/Pd trilayer in an amount corresponding to the thickness t of 0.1 nm or 0.2 nm, which resulted in two contents of copper in the system: approximately 5 at.% and 10 at.%. Layers were deposited at room temperature, with the evaporation rates of 0.5 nm min⁻¹ for Fe and Pd, and 0.3 nm min⁻¹ for Cu. The purity of source materials used for the evaporation was 99.9% or higher. The working pressure was in the range of 10⁻⁹ mbar and the film thickness was controlled during evaporation with a quartz thickness monitor. In addition to the nanopatterned samples, the same multilayer systems were deposited also on flat Si(100) substrates covered by 100 nm thick film of SiO₂. The parameters of the deposition were the same as described above.

After the characterization, the multilayers deposited on the flat and patterned substrates were annealed in vacuum with base pressure of 10⁻⁷ mbar. The rate of 10 °C min⁻¹ was applied to heat up the samples to the temperature of 600 °C. After reaching this temperature the sample was kept 1 min at 600 °C. Then the power was turned off and the samples were left under vacuum until they reached room temperature. The annealing resulted in the creation of the FePdCu alloys with a nominal copper content of approximately 5 and 10 at.%.

2.2. RBS analysis

The chemical composition of the systems was determined with Rutherford backscattering spectrometry. The RBS measurements were performed with ⁴He⁺ ions accelerated to the energy of E₀ = 900 keV with energy spread of 0.3 keV. The scattering angle was fixed to θ = 165°, while the incident angle to α = 0°. The beam was approximately 1 mm in diameter in size. Resolution of the detector used in the experiment was 12 keV. The pressure during the experiments was approximately 10⁻⁷ mbar. The analysis of the spectra was done with the software SIMNRA [18, 19], which is intended for the...
3. Results

3.1. Surface analysis of as deposited nanopatterned (Cu/Fe/Pd) multilayers

The multilayered systems surface was studied by electron scanning microscope (SEM). SEM images of SiO2 nanosphere arrays covered by (Cu0.1 nm/Fe/Pd) × 5 multilayers are shown in figure 2. Figures 2(a) and (b) show the images of the sample nanopatterned using 100 nm nanospheres, while figures 2(c) and (d) of the sample nanopatterned using 50 nm nanospheres.

A well-ordered monolayer of nanospheres with hexagonal symmetry is shown in figure 2(a), reflecting a close packed structure on the surface. Since the nanospheres do not cover the whole Si substrate surface, in the lower left corner of the image the boundary between the flat part of the sample and monolayer of nanospheres covered by (Cu/Fe/Pd) multilayers can be observed. The edges of the particles are brighter due to the greater exposure, which leads to increased emission of secondary electrons. Image shown in figure 2(b) was made in a different location on the same sample. It shows the area of particles layers arranged one above other creating a multilayer. In this place the order is smaller, although hexagonally ordered particles can be found locally.

In figure 2(c), a monolayer of 50 nm nanospheres covered by (Cu/Fe/Pd) multilayer is shown. Also in this case the hexagonal arrangement is noticed, but the order is much smaller than that observed for nanospheres of 100 nm. This is mainly because of the greater dispersion of sizes and shapes for 50 nm nanospheres. Image shown in figure 2(d) was made in a different location on the same sample, where the multilayer of particles was formed. This is the area of the greatest disorder, but also a local hexagonal symmetry can be found there.

The results discussed so far concerned multilayers with a copper layer with the thickness of 0.1 nm. However, SEM studies provided qualitatively the same observations for the (Cu0.2 nm/Fe/Pd) × 5 multilayers.

3.2. RBS analysis of as deposited nanopatterned (Cu/Fe/Pd) multilayers

Experimental and simulated RBS spectra of (Cu/Fe/Pd) multilayers, collected for the flat reference systems, are shown in figures 3(a) and (b). Figures 3(c)–(f) present spectra for the patterned systems. Arrows indicate the positions of Cu, Fe, Pd, Si and O signals. Since the flat silicon substrate is covered by 100nm layer of SiO2, the backscattered signal from the Si atoms consists of two steps visible in figures 3(a) and (b). The step at about 500keV comes from the silicon nuclei located in the outermost silicon oxide, while the step at about 440keV originates from pure silicon below the oxide. Furthermore, the
signals for Cu and Fe are overlapped due to the similar masses of iron and copper nuclei, and thus similar energies of back-scattered \( ^4\text{He}^+ \) ions. This effect is more visible in figure 4, which shows the magnification of the spectra displaying the signals of Fe, Cu and Pd.

In figure 3 clear difference between the initial range of spectra for the flat and patterned systems can be observed, which is associated with the different types of the used substrate. In the case of flat samples, the substrate is pure monocrystalline silicon covered by a homogeneous thin oxide layer. This results in well-defined oxygen peak for energy of about 300 keV. On the contrary, the substrate of patterned samples is covered with mono- and multilayers of SiO\(_2\) nanoparticles, which results in scattering events on oxygen nuclei occurring at various depths in the sample and causes the appearance of additional features in the spectrum for the energy range of 150–350 keV.

In figure 4 one can also notice that all the Fe, Pd, Cu peaks observed for patterned samples are left-side broadened. This effect is directly related to the 3D topography of the sample caused by the patterning. Due to the topography, the path lengths of the incoming and outgoing ions depend on the positions of the scattering nuclei, which is shown in figure 5. In the case of nuclei located on the top of nanospheres (path \#1 in figure 5), scattering processes are the same as for the flat sample. However, particles scattered from the nuclei located between the nanospheres or on the side walls of nanospheres have to pass through SiO\(_2\) material on their way towards the detector, losing extra energy (path \#2 in figure 5). Therefore, the corresponding counts in the RBS spectrum appear at a lower energy, causing left-sided tail of Fe, Cu and Pd signals and leading to a redistribution of the recoil events in the energy spectrum in comparison to the case of a flat surface.

Simulated RBS spectra are shown in figures 3 and 4 as red lines. Since SIMNRA software does not allow the simulation with 3D surface topography, a flat sample model was assumed. For spectra of patterned samples, it was necessary to introduce the roughness of the outer layer to the model. The effect of roughness is similar to that of energy straggling and leads to the additional broadening of spectral features. This gives a similar effect as the broadening caused by patterning [20, 21]. Therefore, the introduction of large roughness comparable with the nanoparticle size (approximately 100 nm) allows the reproduction of low-energy tail of Fe, Cu and Pd peaks. The results of the RBS simulation are shown in table 1. Errors indicated for element contributions take into account the statistical error of the fit, as well as the uncertainty of experimental parameters determination such as energy calibration error. It should be noted that the overlap of the signals coming from Fe and Cu is the main source of the large relative uncertainty of the Cu concentration.
This approach, unfortunately, does not allow to simulate correctly the spectrum for low energies corresponding to O and Si signals. This part of the spectrum, however, does not affect the determination of the metals concentrations in the multilayer.

For the flat samples, the received Fe:Pd concentration ratios are in good agreement with the nominal 48:52. On the other hand, results for all patterned samples showed a slightly lower Fe:Pd concentration ratio when compared with data for the flat samples. This can be caused by the additional inelastic scattering of $^4\text{He}^+$ ions in the SiO$_2$ material (path #3 in figure 5). Helium ions backscattered on iron nuclei have lower energy than the ions backscattered on palladium nuclei. The backscattered ion energy influences the Rutherford cross-section, which is given in the laboratory frame of reference by:

$$\sigma = \frac{mb}{sr} \left( \frac{Z_{\text{He}} Z_{\text{T}}}{E(\text{keV})} \right)^2 \left( \frac{M_{\text{T}}^2 - M_{\text{He}}^2 \sin^2 \theta + M_{\text{T}} \cos \theta}{M_{\text{T}}^4 \sin^4 \theta \sqrt{M_{\text{T}}^2 - M_{\text{He}}^2 \sin^2 \theta}} \right)^2,$$

where $\theta$ is the scattering angle, $Z_{\text{He}}$, $M_{\text{He}}$, and $E$ are the nuclear charge, the mass, and the energy of the projectile, respectively, and $Z_{\text{T}}$ and $M_{\text{T}}$ are the nuclear charge and the mass of the target atom, respectively. According to the formula, in the considered energy range, the higher is the energy of projectile, the smaller is the inelastic scattering cross-section. This means that helium ions backscattered on palladium nuclei (with energy of about 770 keV) are more likely to reach the detector than helium ions backscattered on iron nuclei (with energy of about 675 keV). This causes the observed overestimation of palladium concentration in relation to the concentration of iron. It is worth noting, that this effect does not occur for the thin flat samples.

**Figure 4.** Magnification of RBS spectra obtained for (Cu/Fe/Pd) multilayers and depicted in figure 3. Measured data are presented as circles and simulation as red line. The left and the right column show spectra for the systems containing a layer of copper with the thickness of 0.1 nm and the thickness of 0.2 nm, respectively. Spectra (a) and (b) were collected for flat multilayers, (c) and (d) for multilayers deposited on SiO$_2$ nanospheres with the size of 100 nm, (e) and (f) for multilayers deposited on SiO$_2$ nanospheres with the size of 50 nm.

**Figure 5.** Cross-sectional view of SiO$_2$ nanoparticles (light gray) covered by (Cu/Fe/Pd) multilayer (red) with selected beam paths of backscattered $^4\text{He}^+$ ions.
and therefore the determined element concentrations for these samples are not vitiated by this measurement artifact.

3.3. Surface analysis of nanopatterned FePdCu alloys after annealing

The (Cu/Fe/Pd) multilayers deposited on the flat and patterned substrates were annealed in vacuum chamber. The annealing resulted in the creation of the FePdCu alloys with L10 structure, which was proven by x-ray diffraction measurements (not shown here).

Figures 6 and 7 show SEM images made for the FePdCu alloy deposited on the SiO2 nanoparticles with sizes of 100 nm and 50 nm, respectively, while figure 8 depicts the alloy deposited on the flat substrate. The presented images concern alloys with the nominal copper content of 5 at.%. However, SEM studies provided qualitatively the same images for the alloys containing larger content of copper admixture. Therefore, the description of the results is also applicable to the FePdCu alloys with 10 at.% of copper.

As shown in the images, pronounced changes of the surface morphology occurred during the annealing. Patterned layers lost the continuity and split up into isolated islands (bright areas). In the case of layers deposited on the 100 nm nanoparticles, the islands arrangement reflects the hexagonal order of the SiO2 nanoparticles, and on the top of each nanoparticle a single island of alloy is located. The same alloy deposited on 50 nm nanospheres created islands, which arrangement does not correlate with the topology of the prepatterned substrate.

The least noticeable morphological changes were observed for the systems deposited on the flat substrates. In comparison with as-deposited multilayers, in some places voids appeared. Similar changes of the sample morphology caused by annealing were reported for many thin film systems deposited on the SiO2 and Si substrates. The voids and island formation is reported for thin layers of copper [21], palladium [22], iron [23] as well as for FePd alloys [24]. All of the morphological changes are usually explained by the phenomenon of agglomeration, which can occur well below the melting point of the material by surface and volume diffusion [25, 26].

The agglomeration in thin film systems usually initiates with voids formation that occurs at the grain boundaries [25]. Further annealing results in the initial voids growth and generation of the holes in the film that expose parts of the substrate. If the temperature is sufficiently high, the agglomeration process continues, and the holes connect with each other. Finally, the film breaks up and small islands form on the substrate surface. At this point, the film is no longer continuous. Further annealing induces islands to grow and to slowly coalesce. From a thermodynamic point of view, a giant single island has the lowest free energy. In reality, however, small islands are preferably formed and further coalescence is slow [21].

The above process is common for many thin metallic systems [26], however, the annealing temperature needed to
initiate a given phase of film agglomeration varies with film thickness and depends on the composition of the annealed layer. Additionally, Petersen showed that the agglomeration on a prepatterned substrate takes place at lower temperatures than for the flat systems [27]. Due to the additional driving force of curvature induced diffusion, the system needs lower thermal energy to form voids and islands. This driving force is associated with an excess local chemical potential introduced by the prepatterned structure [28]. Consequently, the additional chemical potential alters the energy relations in the system and forces diffusion of the metal atoms already at lower temperatures.

In order to determine the influence of the substrate curvature on the agglomeration process related to the different island sizes we calculated the distribution of the island area for the alloy deposited on the 50 nm and 100 nm nanoparticles. For 100 nm nanoparticles we observed a narrow distribution of the island area with the mean value of 5660 nm$^2$, while for 50 nm nanoparticles this value increased to 12760 nm$^2$. This difference can be explained by the fact that smaller sizes of the nanospheres result in a larger local curvature, which causes lower temperature at which the process of agglomeration starts. This leads to an earlier island formation and initiation of island coalescence process. As a result, the alloy deposited on the 50 nm nanoparticles has enough time and sufficient temperature to bring the islands together. Consequently, large irregular alloy islands are observed. In contrary, the alloy deposited on the 100 nm nanoparticles at the same annealing conditions does not show the presence of substantial contribution from coalescence process as was shown in [29].

### 3.4. RBS analysis of nanopatterned FePdCu alloys after annealing

The above-described processes are reflected in the RBS spectra collected for patterned FePdCu alloys (figure 9). Figures 9(a) and (b) show spectra collected for the system on SiO$_2$ nanoparticles with size of 100 nm, while figures 9(c) and (d) for the alloy deposited on 50 nm SiO$_2$ nanoparticles. The initial ranges of spectra (below 500 eV) for the annealed and as-deposited samples are very similar to spectra for as-deposited systems, therefore only the range displaying the signals of Fe, Cu and Pd is presented. As in the case of the multilayers, signals originating from Cu and Fe are overlapped due to the similar masses of iron and copper nuclei, and thus similar energies of backscattered $^4$He$^+$ ions.

One can notice, that all the peaks are strongly left-side broadened, however, the broadening is more pronounced for the alloys deposited on SiO$_2$ nanoparticles with the size of 50 nm. It correlates with broad distribution of island area for these samples. Helium particles scattered from the nuclei located on the bottom or inside the coalesced large alloy islands have to pass through thick part of the material on their way towards the detector. It causes additional loss of energy, which is demonstrated by counts in the RBS spectrum appearing at a lower energy.

After the annealing, the additional small left-side peak broadening occurred also in the spectra collected for the alloys deposited on 100 nm SiO$_2$ nanoparticles. The observed changes are less noticeable, which can be related to much smaller mean island area and narrower island area distribution. A large number of small islands does not increase significantly propagation path of projectiles in the material, which in consequence causes less visible changes in RBS spectra.

An attempt to simulate the spectra using the approach presented in section 3.2 did not bring a satisfactory result. An acceptable fit was not obtained both for alloys on 50 nm and 100 nm nanospheres, which means that this simple approach cannot be applied to patterned systems with island size distribution. It is difficult to use RBS technique for chemical composition determination of non continuous films and porous structures. The patterned nanostructure surface could result in an RBS spectrum identical to those obtained for continuous film but with smaller thickness since the empty space between the nanostuctures do...
not contribute to the scattering and to the energy loss. There are attempts to solve this problem by filling such empty space, as for example present in porous samples, by light elements like hydrogen [30]. However, this approach can lead to dubious results in the studied case. Therefore, further development of the RBS data analysis for such systems is necessary.

4. Conclusions

This work presents the preparation and characterization of the (Cu/Fe/Pd) multilayers and FePdCu alloys deposited on matrices of the SiO\textsubscript{2} nanoparticles, as well as on the flat Si/ SiO\textsubscript{2} substrates. We demonstrated that the selected method of patterning affects the RBS spectra and influences the obtained information concerning sample composition. The patterning leads to an additional broadening of spectral features and appearance of the low-energy tail of the peaks. It disturbs quantitative analysis, which results in a slightly lower Fe:Pd concentration ratio when compared with the data for the flat systems. Such effect is caused by the additional inelastic scattering of 4He ions in the SiO\textsubscript{2} material.

In the case of annealed samples, the phenomenon of solid-state dewetting, as observed in [29], resulted in the formation of the isolated alloy islands placed on top of the SiO\textsubscript{2} nanoparticles. For these samples, relationship between the width of island area distribution and changes in RBS signal collected for patterned alloys can be noticed. Broader island area distribution corresponds to the large low-energy tail of the peaks in RBS spectra. We demonstrated that in case of patterned samples consisting of arrays of well separated nanostructures the use of RBS technique is very difficult because of the lack of proper methods for spectra analysis.

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