Angular distribution of atoms sputtered from alloys

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ABSTRACT

The angular distribution of atoms sputtered from Co_5Sm alloy under 3 keV Ar^+ and 10 keV Xe^+ ion bombardment (normal incidence) has been studied experimentally. RBS was used to analyze a material deposited on a collector. The surface composition of Ni_xPd_y alloys (x, y = 1, 3, 5) irradiated by 3 keV Ar^+ ions was also investigated *in situ* by Auger electron spectroscopy. Results of these measurements compared with our previous data on component angular distributions sputtered from Ni_5Pd alloy. Sm segregation was found to effect on component angular distributions sputtered from Co_5Sm alloy. It has been found by using AES that Pd segregates to the surface in Ni_5Pd – $NiPd_3$ alloys, while Ni – in $NiPd_5$ alloy. A new approach to description of component angular distributions sputtered from Ni_xPd_y and Co_5Sm alloys presented.

1. Introduction

Sputtering and accompanying this phenomenon processes play a significant role in numerous applications such as ion beam analysis and technologies. The particular interest from this point of view consists in a study of multicomponent sputtering. Preferential (or selective) sputtering of one of the target components, observed for the first time more then half century ago [1], leads to the formation of an alternated layer which composition is different from the bulk. Therefore, one of the key problems in sputtering of compound targets was to reveal depth profiles of component concentrations within the alternated layer.

It has been shown in a number of researches that depth distributions of components could be approximated by monotonous functions: exponential [2,3] or linear [4]. However the researches of CuNi alloy sputtering distinctly demonstrated that the distributions, formed under bombardment by 5 keV Ar^+ ions at elevated temperatures in subsurface layers, are nonmonotonic [5]. Authors suggested that due to Gibbsian segregation Cu enriched the topmost surface layer from where Cu atoms were preferentially sputtered. In turn, Gibbsian segregation tries to fill in Cu losses arisen from sputtering, depleting the second and deeper subsurface layers. Such pronounced Cu depletion (Ni enrichment) was revealed in subsurface layers of CuNi alloy under steady state sputtering by using Auger electron spectroscopy. Thus, this study showed that Gibbsian segregation together with the effect of selective sputtering played an important role in formation of the alternated layer composition.

Another key aspect of multicomponent sputtering is studying of a spatial distribution of emitted components. The first experiments on research in this direction have yielded unexpected result [6]. It has been found, that angular distributions of components, sputtered from textured AgAu alloy and from texture-free CuPt alloy by Ar^+ ions with energy 80 keV, are different. Moreover, the analysis has revealed that angular distributions of both components sputtered from CuPt alloy are overcosine [6,7]. Underline that targets were irradiated in these experiments at temperatures, when Gibbsian segregation hardly occurs.

The first important step to understanding of processes leading to differences in the angular distribution of components has been made in [7]. Here was suggested that ion bombardment enhanced Gibbsian segregation. As a result of radiation enhanced Gibbsian segregation on the scenario, described in details in [5], the target surface is enriched with Cu (depleted with Pt), and the second and subsurface layers are on contrary depleted by Cu and enriched with Pt. The only difference in these two approaches consists that Gibbsian segregation is thermally activated in the one case [5], and in the second one – by ion irradiation of a target [7]. It is important to note that both approaches assume an existence of a gradient of component concentration in the topmost surface layers. Nearly at the same time it has been theoretically demonstrated that if such concentration gradient exists, the angular distribution of component, which is depleted in the topmost layer, will be more forward peaked, while the angular distribution of another component will be more wide [8]. This theoretical finding in combination with the hypothesis about radiation enhanced Gibbsian segregation explained the differences in angular distribution of the components, sputtered from CuPt alloy.

Later the idea about radiation enhanced Gibbsian segregation developed by the authors in study of component angular

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distributions to investigation of metal alloy with regard to alloys with a different composition and, in particular, to sputtering of CuPt alloy at elevated temperature [9,10].

While a considerable progress in researches of alloy and compound sputtering has been achieved, many aspects of this problem are still studied incompletely. Recently an effect of component concentration on angular distributions of elements sputtered from Ni_xPd_y alloys (x, y = 1, 5) was investigated experimentally and by using computer simulations [11]. It has been found that the relative yield Y_{Pd}/Y_{Ni} increased with the polar emission angle θ for Ni_5Pd and $NiPd$ targets bombarded by 3 and 10 keV Ar^+ ions. The similar behavior of the relative yield was observed for Ni_5Pd alloy irradiated by 15 keV Ar^+ ions and was explained in terms of radiation enhanced Gibbsian segregation of Pd [9]. However, for $NiPd_5$ alloy the component relative yield was found to decrease with increasing of the emission angle. This peculiarity explained in [11] by a reverse atomic segregation in Ni_xPd_y alloys. Undoubtedly, a conformation of this conclusion obtained by a different way would be very useful. Therefore, we used AES in the present work to study a surface composition of Ni_xPd_y alloys bombarded with Ar^+ ions.

Further, analysis of experimental data on sputtering of Ni_xPd_y alloys shown that the angular distribution $Y(\theta)$ of segregated element (Pd for Ni_5Pd and $NiPd$ alloys, and Ni for $NiPd_5$) could be described well by cosine power law:

$$Y(\theta) = Y_0 \times \cos^n \theta \qquad (1)$$

where Y_0 is sputtering yield for emission angle θ = 0° and n is fitting parameter. This expression successfully used to approximate the measured distributions, for example, in cases of Si and Ge sputtering [12,13]. However, this expression has appeared unfit for the description of the angular distribution of the other (nonsegregating) component. In order to understand whether this situation is intrinsic for Ni_xPd_y alloys only, investigations of the angular distributions of the components sputtered from Co_5Sm alloy were performed in the present work.

2. Experimental

Sputtering experiments were carried out in an UHV ion-beam experimental setup [14] consisting of a duoplasmatron ion source, electrostatic lenses and a magnetic separator for the primary beam formation and a sample chamber. The scheme of the experiment is shown in Fig. 1. A mass-separated beam of 3 keV Ar^+ and 10 keV Xe^+ ions bombarded polycrystalline Co_5Sm target along the normal to the surface. A background pressure in the target chamber was 2×10^{-6} Pa. The ion beam diameter was 1.5 mm, and the beam current changed from 6 µA for Ar^+ to 1 µA in the case of Xe^+
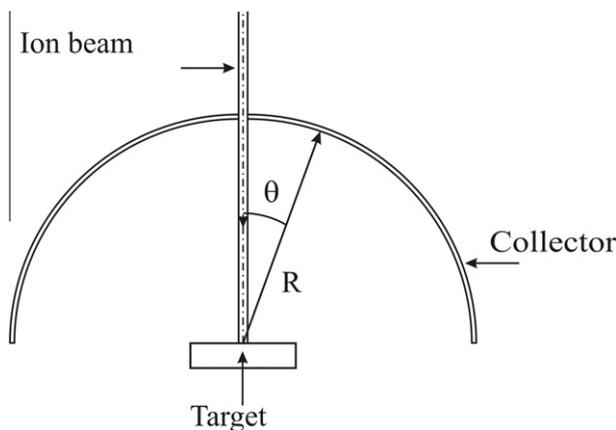

Fig. 1. The scheme of the sputtering experiment.

bombardment. Fluences of bombarding ions were $1–2 \times 10^{18}$ ions/cm^2, which is sufficient to measure the angular distribution of sputtered particles for steady state sputtering conditions.

Polycrystalline Ni_5Pd, Ni_3Pd, $NiPd$, $NiPd_3$, $NiPd_5$ and Co_5Sm alloys with a purity 99.99 at.% were prepared for these experiment in Institute of Metal Physics (Ekaterinburg, Russia). The special attention was given to alloys processing to dispose of a possible texture. X-ray analysis has shown the absence of a texture in all fabricated samples. By using ion etching of targets in a glow discharge it has been found that a grain size of the alloys does not exceed 20 µm. Targets were cut out as disks with a diameter about 9 mm and thickness – 2 mm, and one of the top sides of samples was mechanically polished. Before experiments the targets were cleaned in organic solvents.

A collector technique was used to measure the angular distributions of sputtered atoms. A semi-cylindrical collector with radius about 15 mm made of 0.1 mm-thick high purity Al foil. The material sputtered on the collector was analyzed using Rutherford backscattering spectroscopy (RBS) of a 1.5 MeV He^+ ion beam with a rectangular cross-section of 1×2 mm. The exposure to create RBS spectrum was chosen to provide an accuracy less than 5% for the emission angles $\theta > 70°$ where sputtering yield was low.

Five Ni_xPd_y samples with the different composition were mounted side by side on a goniometer head of Auger spectrometer. Spectra of virgin samples were recorded in Varian auger electron spectrometer at the residual pressure of 2×10^{-7} Pa. Then spectrally pure argon was inserted into the spectrometer chamber (up to a pressure of 6×10^{-3} Pa), and an ion gun was switched on. The samples were irradiated with 3 keV Ar^+ ions at normal incidence to the surface. In order to irradiate all samples simultaneously the ion gun functioned in defocused mode. The ion beam current density was about 0.03 mA/cm^2. The irradiation fluence was 2×10^{17} ion/cm^2, that is enough to reach the steady state sputtering regime. After irradiation, argon was evacuated from the chamber, and auger spectrum for each sample was recorded again.

3. Results and discussions

3.1. Surface composition of Ni_xPd_y alloys

Peaks of surface and bulk impurities C (272 eV), N (380 eV), O (510 eV), and S (152 eV) were observed in the auger spectra of the virgin samples as well as the peaks of main components: Ni (61 and 848 eV) and Pd (330 eV). Since we used high purities targets, AES peaks of C, N and O detected for virgin targets originated mainly from gases absorbed on the surface, while S is a satellite impurity originated from Ni and Pd rich ores. Estimated concentrations of the surface impurities from auger spectra of virgin samples are of order of 14 at.% for C, 2% for O and N. A dependence of the ratio of the Pd (330 eV) and Ni (848 eV) peak height on the Pd content in the targets, shown in Fig. 2a, represents the content ratio of these elements in alloy. A deviation of this curve from a linier caused probably with a different content of impurities adsorbed on the various samples.

The same situation is seen in Fig. 2b, if we concentrate our attention at the first four points only. The peak ratio gradually increases with Pd concentration in the alloy. Let us pay attention to that the angle of slope of the curve in Fig. 2b became less then in Fig. 2a. As already mentioned, our previous study of the component angular distributions indicated that Pd is the element segregated to the surface and preferentially sputtered in cases of Ni_5Pd and $NiPd$ alloys [11]. Hence, subsurface layers of these alloys have to be depleted with Pd due to radiation enhanced Gibbsian segregation. The thickness of the alternated layer is determined by bombarding ion penetration depth and can be characterized by value of ions
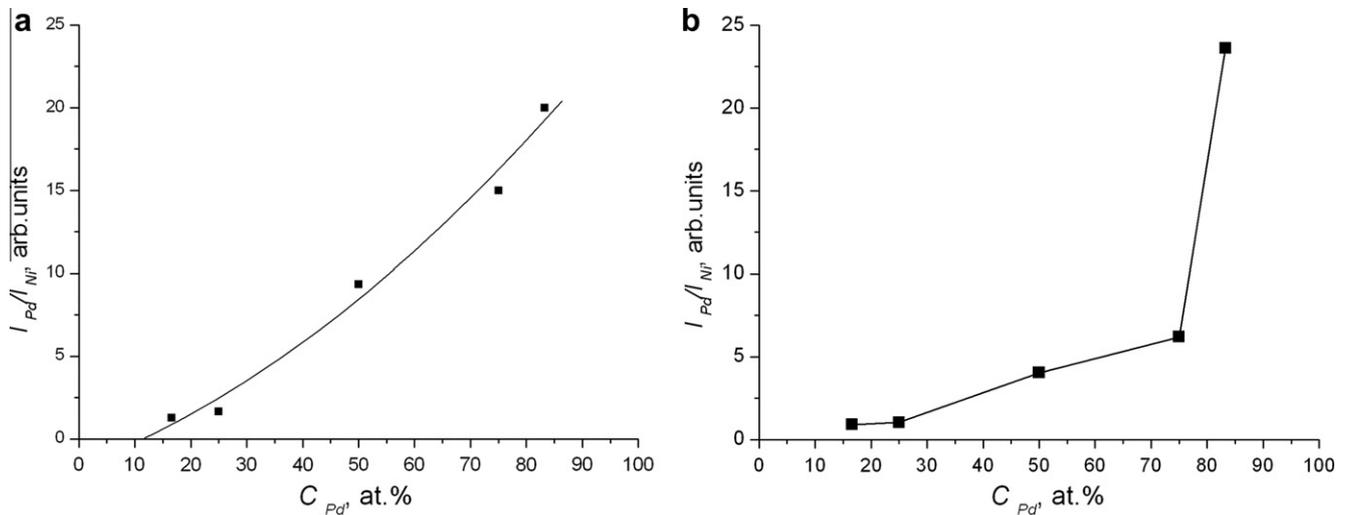
Fig. 2. The ratio of the Pd (330 eV) and Ni (848 eV) peak height on the Pd content in Ni_xPd_y alloys: (a) before and (b) after ion bombardment with 3 keV Ar^+ ion.

projectile range. The mean range of 3 keV Ar^+ ions, calculated by using SRIM [15], changes from 28 to 26 Å for Ni_5Pd and $NiPd_5$, correspondently. Since the escape depth of the auger electron does not exceed 20 Å, decreasing the angle of slope of the curve I_{Pd}/I_{Ni} argues that Pd relative concentration has decreased, and Ni one – increased in the alternated layer. The absence of any particular futures at this part of the curve makes it possible to conclude that Pd is also segregating and preferentially sputtered component of Ni_3Pd and $NiPd_3$ alloys.

However, this ratio increased five times for $NiPd_5$ alloy compare to $NiPd_3$ alloy. It is impossible to explain such a change of the peak ratio by influence of surface impurities. For all samples, the height of impurities peaks decreased significantly after ion bombardment with fluence 2×10^{17} ion/cm^2: C peak height decreased about five times, O – two times and N peak was not observed at all. To eliminate possible effect of impurities samples were bombarded additionally with fluence 2×10^{17} ion/cm^2 and the peak ratio was again measured for $NiPd_3$ and $NiPd_5$ alloys. Results of these measurements coincided with the previous one within a few percents.

On the other hand, our previous results pointed out that Ni segregates to the surface and preferentially sputtered from $NiPd_5$ alloy [11]. If Ni is the segregating element, then subsurface layers have to be enriched with Pd atoms and, hence, depleted with Ni. Such a change in the component concentrations leads to a large increasing of Pd auger signal and, vice versa, to a decreasing of Ni auger signal, and, consequently, explains the observed jump in the dependence presented in Fig. 2b.

Thus, AES researches of the surface composition of Ni_xPd_y alloys modified by ion bombardment have shown that Pd is a segregating element in Ni_5Pd – $NiPd_3$ alloys, while Ni – in $NiPd_5$ alloy. This conclusion fully corresponds to the assumption based on our study of angular distributions of sputtered components [11].

3.2. Angular distributions of components sputtered from Co_5Sm alloy

Yield composition ratio (or relative yield) Y_{Sm}/Y_{Co} of sputtered material is plotted in Fig. 3 versus emission angle θ for Co_5Sm alloy bombarded by 3 keV Ar^+ and 10 keV Xe^+ ions. The same dependence for Ni_5Pd alloy, measured in [11] under 3 keV Ar^+ ion bombardment, is also shown here. All dependences are normalized to the bulk composition of the samples, and, as is seen from Fig. 3, exhibit a very similar behavior. According to [9,11], such behavior of the relative yield demonstrates that Sm segregates to the surface under ion bombardment of Co_5Sm alloy. It is also seen from
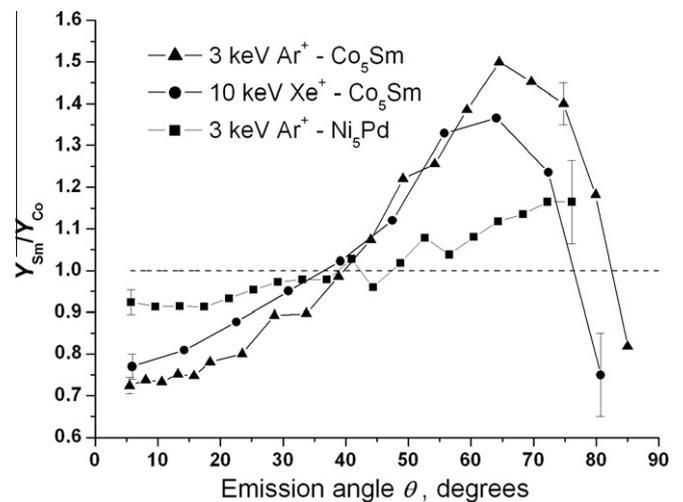

Fig. 3. Dependences of the relative yield of components sputtered by 3 keV Ar^+ and 10 keV Xe^+ ions from Co_5Sm. The relative yield Y_{Pd}/Y_{Ni} versus emission angle θ, measured in Ref. [11], is also presented.

Fig. 3 that the dependence of the relative yield for Co_5Sm target is weakly affected by energy and type of projectiles. Note that a weak sensitivity of the relative yield dependence on emission angle to ion bombarding energy was earlier observed for Ni_5Pd and $NiPd$ alloys [11].
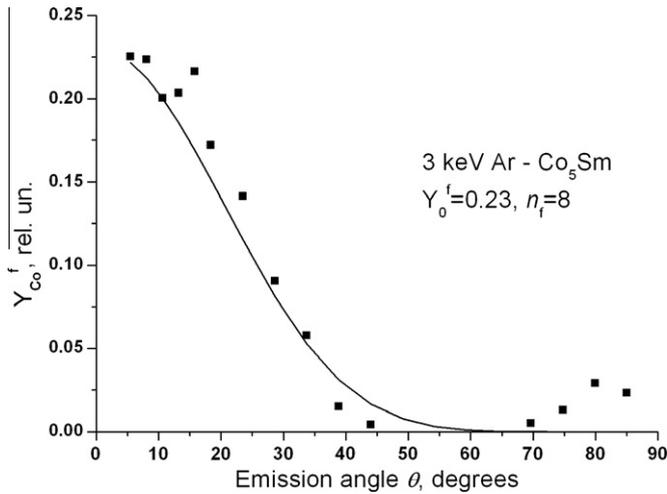
The normalized angular distribution of sputtered Sm measured for 3 keV Ar^+ ion bombardment of Co_5Sm alloy is shown in Fig. 4a. The distribution is rather smooth, has no particular features typical for textured targets and can be approximated well by the cosine power law (1) with $n = 1.05$.

However, the angular distribution of sputtered Co as it is clearly seen from Fig. 4b cannot be approximated by using the expression (1). As mentioned above we have met the same problems earlier trying to describe the angular distribution of nonsegregating element in case of Ni_xPd_y alloys sputtering. In all cases attempts to fit expression (1) by normalizing at small emission angles led to n values much higher than 2.

The same time the power cosine law describes experimental data well at emission angles $\theta > 40°$ with $Y_0 = 0.78$ and $n = 1.5$ (see Fig. 4b). The difference between the measured angular distribution of Co atoms and the approximation by the cosine power law with represented parameters is shown in Fig. 5. This fraction of
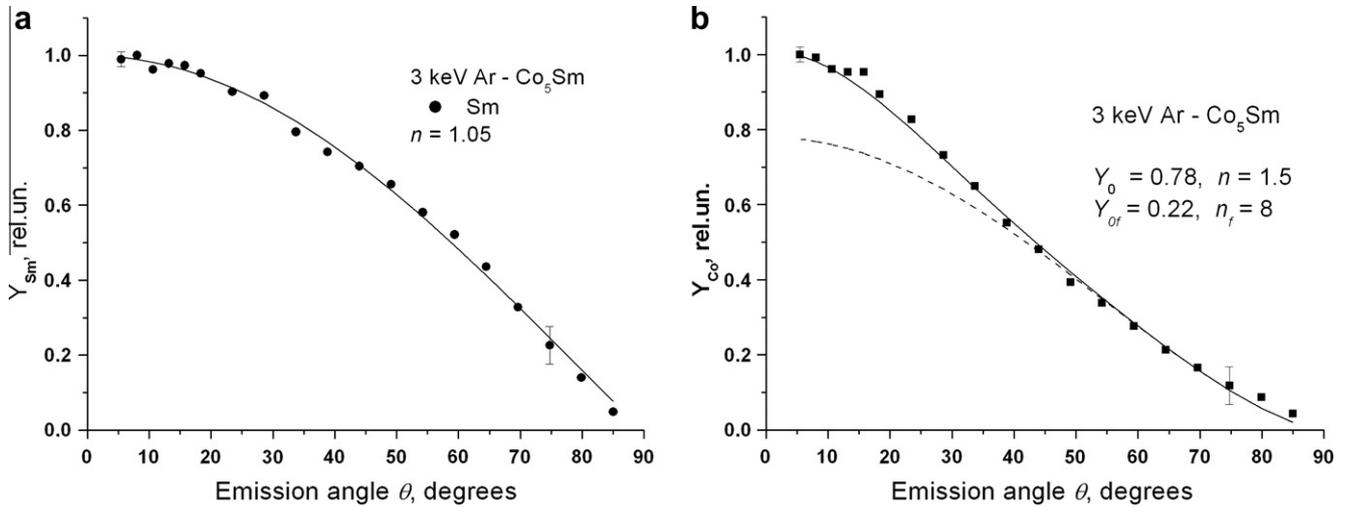
Fig. 4. (a) The angular distribution of Sm atoms sputtered from Co_5Sm alloy by 3 keV Ar^+ ions: ● experiment, — approximation by the expression (1) with $n = 1.05$; (b) the distribution of Co atoms sputtered from Co_5Sm alloy by 3 keV Ar^+ ions: ● experiment, —— approximation by expression (1) and — approximation by the expression (2) with $Y_0 = 0.78$, $n = 1.5$, $Y_{f0} = 0.22$ and $f = 8$.

Fig. 5. The dependence of the focused fraction of sputtered Co atoms on emission angle: ● difference between measured angular distribution and cosine power law (1), — fitting by using the additional term.

sputtered Co atoms can also be approximated well by the cosine power law with the exponent $f = 8$ and $Y_f = 0.23$. Evidently, that the angular distribution of sputtered nonsegregated element will be fitted well with an expression consisting from two terms:

$$Y(\theta) = Y_0 \times \cos^n \theta + Y_f \times \cos^f \theta \qquad (2)$$

The expression (2) has appeared to fit well for the description of the angular distribution of nonsegregating element in case of sputtering Co_5Sm alloy by 10 keV Xe^+ ions as well as for sputtering of Ni_xPd_y alloys. Values of n and f extracted from the best fitting of expressions (1) and (2) to experimental results for Co_5Sm and Ni_5Pd alloys gathered in Table 1.

Naturally, a question on physical meaning of expression (2) arises. As already mentioned above, the angular distribution of component, which is depleted in the topmost layer, was theoretically shown to be more forward peaked [8]. It means that atoms of nonsegregated element, emitted from the second layer, are focused by atoms of the topmost layer near to the surface normal. Thus, the second term in expression (2) describes this focused fraction of sputtered flux, while the fist term as well as expression (1) is responsible for a cascade fraction of sputtered flux.

Table 1
Values of n and f extracted from the best fitting of expressions (1) and (2) to measured angular distributions of components for sputtering of Co_5Sm and Ni_5Pd alloys by Ar^+ and Xe^+ ions with different energies. The calculated contribution of the second layer to sputtered flux of the segregated element is shown in the right hand column.

	n Pd	n Ni	f Ni	Contribution of the 2nd layer (%)
3 keV $Ar^+ \rightarrow Ni_5Pd$	1.45	1.6	10	7
3 kev $Ar^+ \rightarrow Co_5Sm$	1.05	1.5	8	14
10 keV $Xe^+ \rightarrow Co_5Sm$	1.3	1.6	11	10
	Sm	Co	Co	

The contribution of the nonsegregated component emitted from the second surface layer to sputter flux for different alloys is shown in Table 1. This contribution was calculated as a ratio of the area lying under a curve described by the second term in expression (2) to the total area limited by expression (2). Notes, it follows from computer simulations of sputtering that the topmost layer contributes to sputtered flux about 80–90% [16,17].

It is seen from Table 1, that the value of n for Co_5Sm target is lower then for Ni_5Pd under 3 keV Ar^+ bombardment, while increasing of target mass was demonstrated in [13] to lead to increasing of n values. This contradiction can be explained if we compare parameters of the targets. The surface binding energies of Ni and Co are very similar: $U_{Ni} = 4.46$ eV and $U_{Co} = 4.42$ eV. However, the corresponding values for Pd and Sm are very different: $U_{Pd} = 3.91$ eV and $U_{Sm} = 2.14$ eV. The mean mass of Co_5Sm and Ni_5Pd alloys differs slightly (about 12%). Thus, the lower values of n especially for Sm in the case of Co_5Sm sputtering are more probably connected with the influence of the surface binding energy on the angular distribution.

There is an opinion that 10 keV Xe irradiation, particularly on Sm-rich alloys, will give rise to a substantial amount of non-linear sputtering. The nonlinear effect in sputtering, observed for the first time in [18,19], was explained by overlapping of atomic collision cascades. Such overlapping occurs because the molecular ion falls to pieces at the first collision with a surface atom and subsequently each of atoms of the former molecular ion forms its own cascade. Further, the primary energy of ions E_0 in these experiments was chosen so that energy deposited in a layer responsible for sputtering has a maximum possible value for the ion-target combination. In particular, Te^+, Te_2^+, Sb^+ and Sb_2^+ ions bombarded Ag and Au tar-

gets used in these experiments. As both cascades arise simultaneously and are localized in immediate proximity, their overlapping is possible. Thus, in an overlapping zone moving atoms of one cascade interact with moving atoms of another cascade (so-called spike regime of sputtering), giving a contribution to sputtering yield increasing. It is of interest to note that an energy density in a cascade, that can be characterized by the mean energy deposited per target atom – θ_0 [20,21], was in the experiments [18,19] of order 0.3 eV/atom.

However, in our experiments an ion beam current density is such that overlapping of the cascades created by separate Xe$^+$ ions does not occur. Nevertheless, spikes possibly can arise under irradiation by heavy monatomic ions, in particular, Xe$^+$ ions due to high energy density in a cascade. Really, θ_0 value considerably increases with decreasing of E_0 [20,21]. For example, in a case of irradiation of Ag target by 10 keV Te$^+$ ions, which atomic weight is close to weight of Xe ions, a value of θ_0 increases up to 13 eV/atom. If it is truth, the situation looks as a collapse. A substance should evaporate from a cascade zone. It should lead to very high values of sputtering yield compare to a case of sputtering by argon ions. Besides, since cascade duration is of order of 10^{-13} s, a shock wave should be exited like a situation with a laser ablation. Very narrow angular distributions of ablated material with half-width from 15° to 50°, depending on laser pulse width and intensity, have been found for NiMoRe and Ni$_x$Pd$_y$ alloys [22,23]. It is of interest to note, that RBS analysis of a material deposited on a collector revealed that ablated flux is enriched with Pd when Ni$_5$Pd and NiPd alloys were irradiated by overlapping nanosecond laser pulses (overlapping means that a few pulses hit at the same area) [23]. No doubt that this enrichment occurs due to Gibbsian segregation.

However, experimental data [24] point out, that the difference of sputtering yield of Ag target for Ar$^+$ and Xe$^+$ bombarding ions is not so great at the range near 10 keV, as it was possible to expect in case of the spike effect. Unfortunately, we do not know researches directed on a motivated study spike effect on angular distributions of sputtered particles.

Coming back to discussion of sputtering of Co$_5$Sm alloy note, first of all, that an average atomic mass of the alloy is not too heavy – 74 amu. From a point of view of cascade initiation process, which is determined by energy transfer from projectiles to target atoms, a difference between Ar$^+$ and Xe$^+$ ion bombardments is insignificant. The kinematic factor is equal 0.91 and 0.92 for Ar$^+$ and Xe$^+$ ions, correspondingly. Obviously therefore the angular distributions of components sputtered from Co$_5$Sm alloy by Xe$^+$ ions contain all particular features revealed in case of Ar$^+$ irradiation. Thus, it can be concluded that the spike regime is not realized in case of Co$_5$Sm alloy bombarded by Xe$^+$ ions.

Contradictions of experimental data to theory predictions, concerning a role of spikes, are caused, most likely, that θ_0 value is overestimated in the theory [20,21]. In our opinion, such disagreement occurs for following reasons. First, in the theoretical consideration an energy dissipation of bombarding ions occurs within elastic collision cascade, while inelastic energy losses are not considered. Secondly, processes of energy transfer from the cascade to a target volume are not taken into account. Thus, a further progress

both theory and experiments needs for better understanding of this problem.

4. Conclusions

The investigation of radiation-enhanced Gibbsian segregation in Ni$_x$Pd$_y$ alloys has been performed by using AES. It was demonstrated that the segregating element changed in the alloys dependently on the component concentration. The obtained results are in good agreement with conclusions of [11] and confirm that the alloy composition determines sputtering mechanisms and in its turn the component angular distribution.

The angular distributions of components sputtered from polycrystalline Co$_5$Sm alloy were studied experimentally. It was found that radiation-enhanced segregation of Sm is responsible for the angular distribution of components sputtered from this alloy. The surface binding energy was found from a comparison experimental data for Co$_5$Sm and Ni$_5$Pd alloys to effect strongly on the angular distribution of the component that segregates to the target surface.

The new approach to description of the angular distribution of components sputtered from alloys suggested.

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