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The Effect of the Modulation Period on the Diffusion Intermixing and Magnetic Properties Behavior of Pt/Co Thin Films Induced by Ion Pre-irradiation

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The effects of modulation period and N^+ ion pre-irradiation on the layers intermixing and magnetic properties modification of Pt/Co and Pt/Co/Pt/Co stacks were investigated. The correlation between structural changes, diffusion intermixing, and magnetic properties behavior was discussed as a function of ion fluence. An experimental analysis was performed using X-ray diffraction, secondary ions mass-spectrometry, and in-plane vibrating sample magnetometry characterization. The nonlinear behavior of the saturation magnetization values for the bi- and four-layered stacks was observed. Based on the results of the study, it was found that a significant increase in coercivity (498 Oe) of the four-layered stack after the pre-irradiation with a fluence of 5×10^{14} ions/cm², related to the ion-induced collisional intermixing and more pronounced formation of the structural defects. It was demonstrated that employing a significant enhancement of the formation of additional defects and intrinsic strains may stretch the crystal lattice of the $A1$-CoPt phase, resulting in a modification of exchange interaction between Co atoms in the thin-film composition.

Keywords: magnetic thin films, ion irradiation, diffusion, Co-Pt alloy, interface, phase formation.

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Introduction

CoPt-based thin multilayered or alloyed films are promising materials for designing spintronic devices such as ultrahigh density magnetic storage media [1], magnetic tunnel junctions [2-6], exchange-coupled composites [7, 8], spin-orbit torque devices [9, 10], spin valves [2], and skyrmion-based media [11]. Such widespread applications are attributed to the unique magnetic properties of CoPt alloy, including high coercivity and magnetization saturation, as well as pronounced corrosion resistance.

However, the magnetic properties of CoPt thin films are substantially sensitive to composition, synthesis, and post-deposition treatment conditions. Therefore, the fabrication of ferromagnetic CoPt thin films becomes challenging due to specific diffusion kinetics and the necessity of additional thermal processing [12]. Regardless of whether it is a single-layer CoPt alloyed film [13, 14] or a layered stack prepared by the sequential deposition of individual Co and Pt layers [15-19], thermal processing leading to the formation of various ferromagnetic phases such as soft magnetic cubic $A1$-CoPt [16], $L1_2$-Co_3Pt [20], $L1_2$-$CoPt_3$ [15], and the hard-magnetic tetragonal $L1_0$-CoPt [17] or hexagonal Co_3Pt $D0_{19}$ [21] are taking place.

Ion irradiation is a unique tool for post-deposition tailoring of thin film's structural and magnetic properties [22-24]. For example, it has been effectively used either for designing high-resolution magnetically patterned planar nanostructures suitable for information storage applications [25] or irradiation-induced intermixing and atomic displacement for subsequent ordering into $L1_0$ structure with out-of-plane perpendicular magnetic anisotropy [23, 26, 27].

Recent studies are mainly devoted to irradiating Co/Pt heterostructures using various types of ions at different energies and fluences to tailor the phase composition and magnetic properties of thin-film material. A variety of ion modification mechanisms are under discussion. For instance, Som et al. [28] have attributed the structural and magnetic changes in Co/Pt stacks under irradiation with 150 keV Ar^+ ions to the clustering of irradiation-induced point defects. Bonder et al. [29] have observed a shift in magnetization orientation from out-of-plane to in-plane in Co/Pt multilayered stacks irradiated with 40 keV Ar^+ ions due to the specific distribution of recoils atoms into adjacent layers, followed by roughening and mixing of the interfaces. Vieu et al. have reported [30] that magnetic changes in Co/Pt ultrathin films under uniform irradiation by Ga^+ ions at low fluence in the 20 - 100 keV range are related to the effect of ion-induced collisional intermixing, leading to the formation of stable Co–Pt alloys with the composition that varying across the interfaces. Meanwhile, the film could become paramagnetic at room temperature due to the Pt enrichment of the Co layer. Ion irradiation has also been successfully employed for low-temperature ordering of intermetallic ferromagnetic phases. As has been reported by Maziewski et al. [31], the Ga^+ irradiation-induced intermixing at Co/Pt interfaces leads to the formation of the ordered $Co_{1-x}Pt_x$ alloys exhibiting high out-of-plane magnetic anisotropy.

An alternative prospective approach to achieve favorable magnetic properties is forming a chemically ordered CoPt alloy with the $L1_0$ structure. The conversion of the films from their multilayered structure to a final ordered alloy product is typically realized via heat treatment [32-34]. However, ion irradiation is an attractive supplement to heat treatment for converting magnetic thin films to their required structural-phase state. This may be realized by employing a significant enhancement of interdiffusion at lower temperatures due to the formation of additional defects and intrinsic strains [35]. For instance, Som et al. have shown [36] that 100 keV Kr ion irradiation of nanoscale Co/Pt multilayers leads to CoPt ordered/disordered phase formation at different temperatures. In particular, higher defect mobility is caused by the thermally-induced diffusion process at higher temperatures. Ghosh et al. [37] have reported that the ion-induced formation of the ordered CoPt phase in Co/Pt bi-layered stacks is more pronounced at elevated temperatures, resulting in significant shape anisotropy.

Despite extensive studies of ion irradiation's influence on the changes in magnetic properties, the effect of two-stage processing involving ion pre-irradiation and subsequent heat treatment on the structural and magnetic properties remains insufficiently studied. It has been recently shown [38] that ion bombardment of Co/Pt and Pt/Co thin films with 110 keV Ar^+/N^+ ions followed by the post-annealing in vacuum at 550°C for 30 min led to slowing down diffusion intermixing of the components. However, such a two-step treatment leads to enhancement of the film's coercivity. It is supposed that selecting the optimal combination of “ferromagnetic/non-magnetic” layer thicknesses and ion irradiation fluence can have a decisive impact on diffusion kinetics. Moreover, Moog et al. [39] have already reported that the Pt layer thickness directly contributes to the perpendicular magnetic anisotropy in the Pt/Co multilayers.

Therefore, this study is devoted to investigation of the effect of the two-stage N^+ ion pre-irradiation and subsequent thermal treatment on the structural and magnetic properties of Pt/Co and Pt/Co/Pt/Co multilayered stacks. This research is motivated by our previous work [38] and is meant to reveal how additional Pt/Co interfaces and various N^+ ion fluence will affect the diffusion intermixing and exchange coupling behavior.

I. Experimental procedure

The bi-layered Pt(10 nm)/Co(10 nm) and four-layered Pt(7 nm)/Co(5 nm)/Pt(7 nm)/Co(5 nm) stacks were prepared on SiO_2/Si(001) substrates by magnetron sputtering at room temperature. The vacuum chamber was maintained at a background pressure of 5×10^{-8} mbar, with an Ar^+ sputtering pressure set to 8×10^{-4} mbar during deposition. Thin films were deposited employing high-purity (99.9 %) Co and Pt targets using DC and RF sputtering, respectively. Deposition rates were 2 Å/s for Co and 1.5 Å/s for Pt.

Post-growth ion pre-irradiation was performed using N^+ ions at Balzers MPB 202 implantation system. The ion energy was set at 110 keV, with three irradiation fluences of 1×10^{14} ions/cm², 5×10^{14} ions/cm² (correspond to modulation period 20 nm), and 1×10^{15} ions/cm² (correspond to modulation period 12 nm). The irradiation spot size was 3 cm in diameter. Pre-irradiated stacks were annealed in a vacuum of 10^{-6} mbar at 550 °C for 30 minutes. Non-irradiated samples subjected to the same annealing process were also analyzed for comparison. The thickness of the individual layers and the irradiation dose were selected to ensure a sufficient ion mean free path depth without surface degradation. Both synthesis and processing conditions, except for the layer modulation period and various ion fluences, are chosen to be the same as in the previous study [38] to reach a homogenization of the element and phase composition. Fig. 1 schematically shows the sequence of the treatment processing applied for the investigated thin-film samples.

The structural properties and phase composition of the stacks after deposition, ion pre-irradiation, and post-annealing were examined using the X-ray diffraction technique (XRD) at Rigaku Ultima IV diffractometer with tube anode, utilizing CuK_α radiation (λ = 1,5406 Å) in θ–2θ Bragg-Brentano geometry [40]. Secondary ions mass spectrometry (SIMS) depth profiling was performed at Ion ToF IV analyzer equipped with a Cs^- primary ion source to determine the content of elemental species as a function of the sample's depth. The impact energy of the beam was 2 keV, and negative secondary ions were detected. The apparatus was pumped down to a vacuum of 10^{-9} mbar before the operation of the spectrometer. The magnetic properties of the as-deposited and ion/heat-treated samples were measured at room temperature using a vibrating sample magnetometer with a magnetic field of up to 1 kOe. The magnetic field was applied in two geometries related to the sample surface: in-plane and out-of-plane.

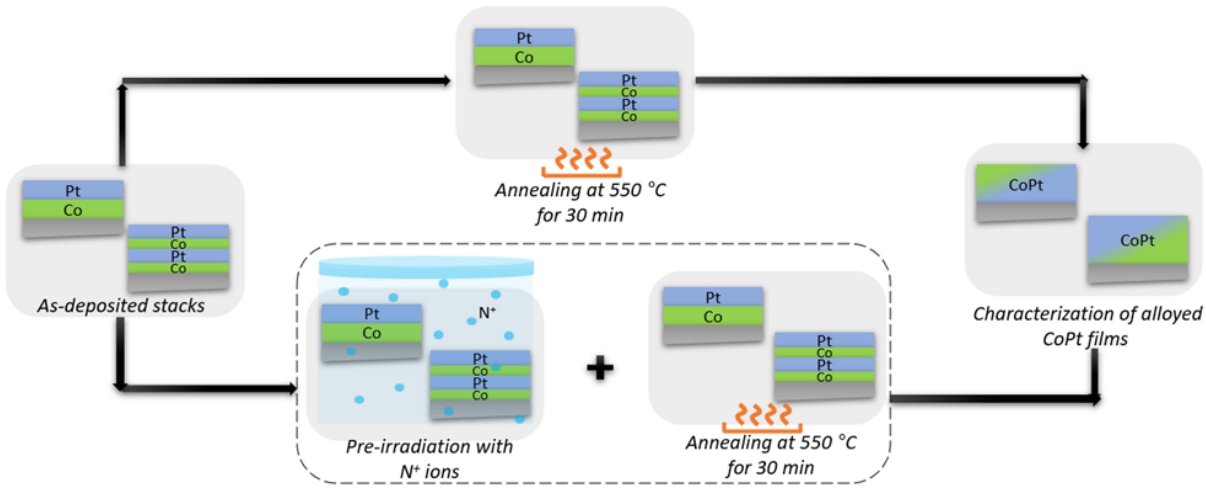

Fig. 1. The schematic illustration of the sequence of post-deposition ion irradiation and subsequent thermal processing of the bi-layered Pt/Co and four-layered Pt/Co/Pt/Co stacks.

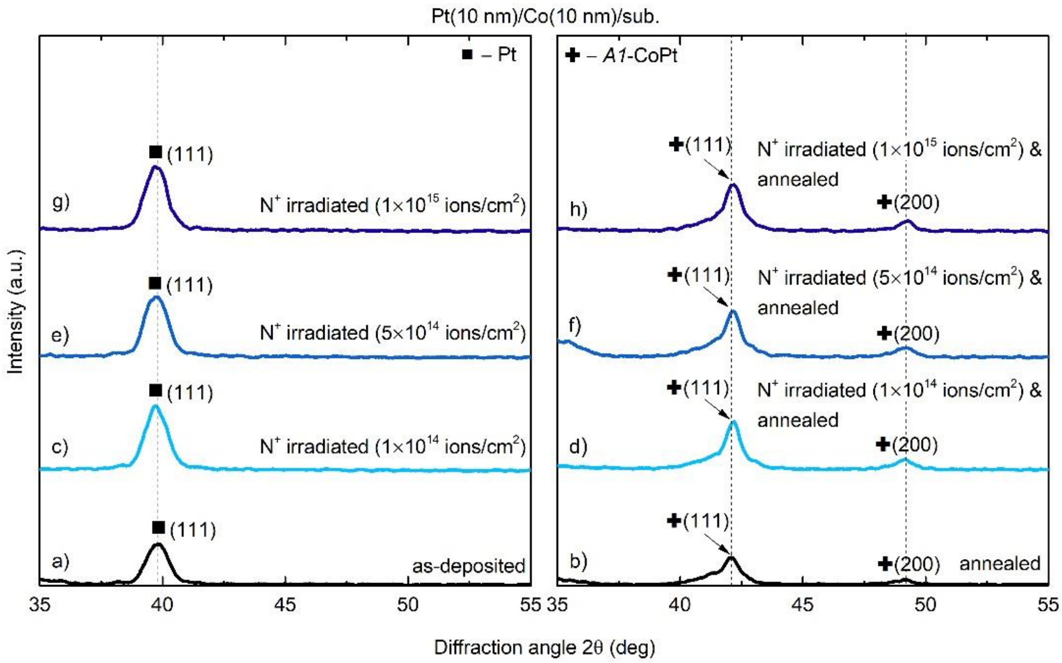

Fig. 2. XRD patterns of the bi-layered Pt/Co stack after deposition (a), N^+ ion irradiation (c, e, g), single-stage annealing (b), and pre-irradiation followed by the post-annealing (d, f, h).

II. Results and discussion

2.1. Structural characterization

Fig. 2 shows XRD patterns of the bi-layered Pt/Co stacks after deposition, N^+ ion pre-irradiation with various fluences, single-stage annealing, and irradiation followed by post-annealing. At the as-deposited state (Fig. 2a), the film consists of the face-centered cubic (fcc) Pt phase, and the corresponding diffraction peak (111) is detected at a 39.8° 2θ angle. Peaks from Co are not observed due to the limited thickness of the corresponding layer and its atomic scattering factor. Another reason is that when using the copper source, the observed cobalt intensity is less than the intensity expected due to the high linear attenuation coefficient and mass absorption coefficient compared to Pt. An annealing of the bi-layered stack leads to sufficient intermixing between Co and Pt layers, inducing a disordered *A*1-CoPt alloy phase formation, resulting in a growth of fcc-CoPt (111) and (200) diffraction peaks at corresponding XRD spectra (2θ = 42.1° and 49.2°, respectively). The ion irradiation of the as-deposited bi-layered stack does not lead to notable structural changes. As the ion fluence increases, the Pt(111) diffraction peak asymmetry changes due to displacing Co and Pt atoms across the interface (Fig. 2c, e, g). As a result of the two-stage processing (pre-irradiation followed by post-annealing), a disordered CoPt alloy phase is forming. Furthermore, the CoPt (111) peak shifts toward a slightly higher angle for a fluence of 1×10^{15} ions/cm² which is likely due to the stress induced by the neighboring of the Pt and Co atoms.

Co/Pt/Co stack after deposition, N^+ ion pre-irradiation, single-stage annealing, and irradiation followed by post-annealing. It follows from the comparison of the XRD

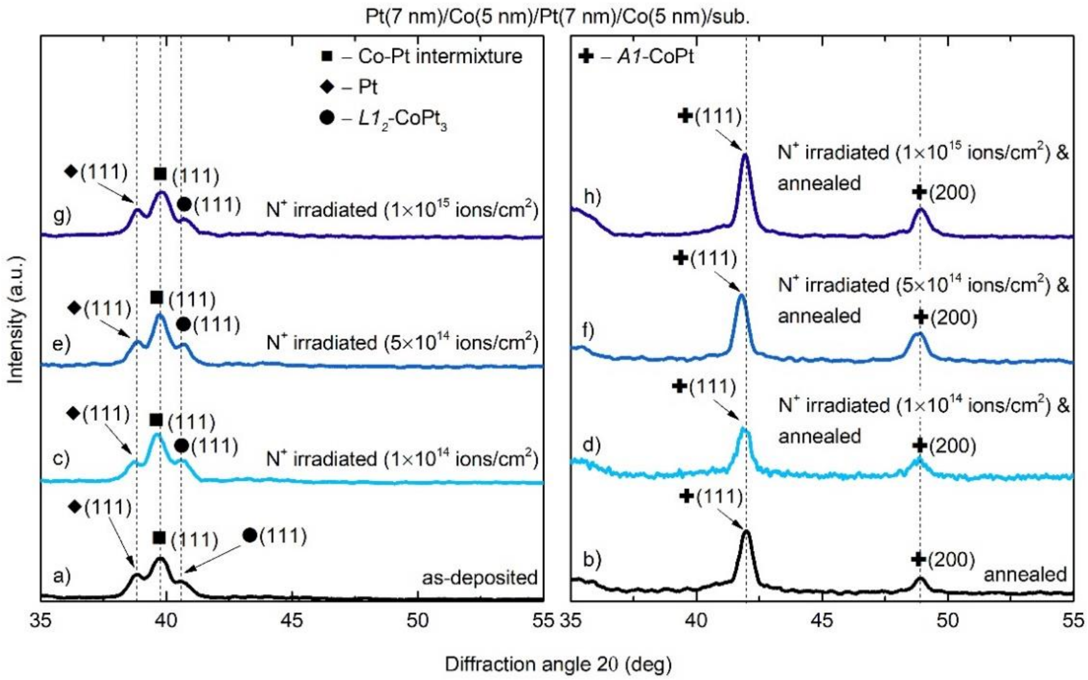

Fig. 3 . XRD patterns of the four-layered Pt/Co/Pt/Co stack after deposition (a), N^+ ion irradiation (c, e, g), single-stage annealing (b), and pre-irradiation followed by the post-annealing (d, f, h).

patterns of the as-grown bi-layered stack (Fig. 2a) with a four-layered one (Fig. 3a) that the decrease of modulation period (thickness of the Pt/Co bilayer) induces layers intermixing even in the as-deposited state due to diffusion activation along the interfaces. Won et al. have already reported the possibility of intermixing at the Co/Pt interface during the deposition process [41]. In our case, the film consists of a mixture of pure Pt ($2\theta_{(111)} = 39.8°$) and Pt-rich $CoPt_3$ ($2\theta_{(111)} = 40.64°$) phases formed by the displaced Co and Pt atoms across the interface. It is assumed that the diffraction peak that appeared at a lower angle ($2\theta = 38.84°$) corresponds to the metastable non-equiatomic Co-Pt mixture. The coexistence of CoPt with the $CoPt_3$ phases under ion irradiation has been reported by Kavita et al. [42]. Another explanation for the presence of this peak is a result of an interference phenomenon related to crystal coherence called Laue oscillations or thickness fringe [43]. After single-stage annealing (Fig. 3b), similar to the bi-layered stack (Fig. 2b), the formation of the disordered *A*1-CoPt alloy phase was detected, as suggested by the appearance of fcc-CoPt (111) and (200) diffraction peaks ($2\theta = 42°$ and 48.9°, respectively). The total intensity of (111) and (200) diffraction peaks from this phase is twice higher compared to the bi-layered stack, indicating a higher degree of lattice ordering. Upon ion irradiation, with an increase of the ion fluence (Fig. 3c, e, g), no noticeable changes are recorded in the peak positions nor the appearance of any additional peaks. Thus, the irradiation did not induce any noticeable changes in the phase composition. For the pre-irradiated stack next subjected to the thermal annealing at 550 °C, the presence of fcc-CoPt (111) and (200) peaks at $2\theta = 42°$ and 48.9°, respectively, were observed for the smallest fluence of 1×10^{14} ions/cm² (Fig. 3d). It is important to note that pre-irradiation with an intermediate fluence of 5×10^{14} ions/cm² results in a slight shift in the position of the (111) and (200) peaks from 2θ = 42° and 48.9° to $2\theta = 41.8°$ and 48.7°, respectively (Fig. 3f). The observed peak shift signifies the atomic rearrangement due to the recoil cascade process induced by ion irradiation. An increase in the ion fluence to 1×10^{15} ions/cm² results in the rise of the peak intensity; however, the peak shift is hardly noticeable (Fig. 3h). It can be concluded that the reduction of the modulation period from 20 nm to 12 nm leads to pronounced structure modification in the case of ion pre-irradiation with the fluence of 5×10^{14} ions/cm². It can be explained by the maximum intermixing induced by ion pre-irradiation.

2.2. Chemical depth profiling

Using SIMS chemical analysis, the distribution of Co, Pt, and Si secondary ions through the depth are recorded to provide a qualitative understanding of the interlayer intermixing across the stack interfaces. Fig. 4 shows the SIMS depth profiles of the bi-layered Pt/Co stack after deposition, N^+ ion irradiation, single-stage annealing, and irradiation followed by post-annealing. It can be seen that film in the as-deposited state consists of two alternating Pt and Co layers (Fig. 4*a*). A moderate blurring of the Pt/Co interface results in the appearance of a clear peak from complex CoPt ion. This is evidence of some limited layer intermixing along the interface during the deposition process. The rise of Si secondary ions intensity on the outer surface is an artifact associated with an ionic mass match between silicon and hydrocarbonates [38]. Post-growth annealing leads to the homogenization of Co and Pt atoms through the film depth (Fig. 4b). Meanwhile, similar Co and Pt profiles are observed in the samples irradiated at all three studied fluences. SIMS profiles after the two-stage processing (Fig. 4d, f, h) show the mutual migration of the Pt and Co signals upon annealing at 550°C across all samples, suggesting the composition

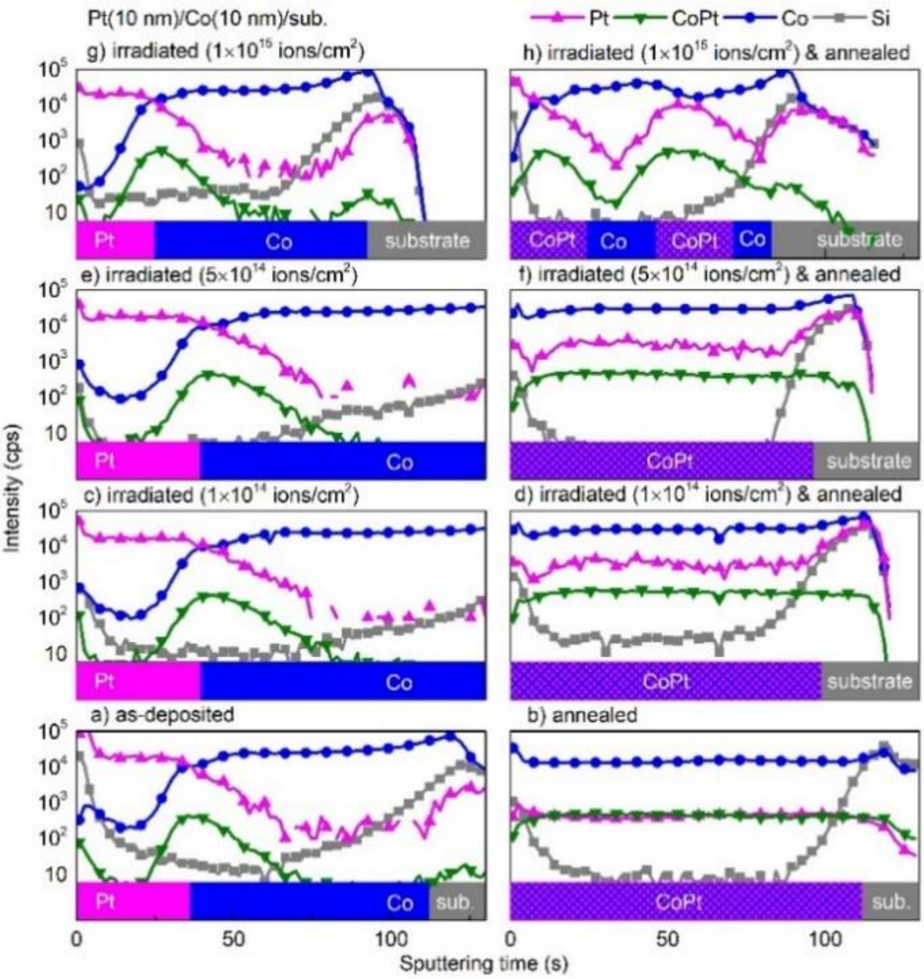

Fig. 4. SIMS chemical depth profiles of the bi-layered Pt/Co stack after deposition (a), N^+ ion irradiation (c, e, g), single-stage annealing (b), and irradiation followed by the post-annealing (d, f, h). Pt peak intensity was normalized to the total secondary ions' intensity.

homogenization. This may be understood as follows from the diffusion rate dependence of the atomic concentration gradient based on Fick's First Law. It should be noted that Co peak intensity across all samples is more uniform, which can be attributed to the higher diffusivity of the smaller and lighter Co atoms compared to Pt. It is important that more significant changes in Co and Pt depth profiles are observed at the higher fluence of 1×10^{15} ions/cm² (Fig. 4h). Co atoms penetrate deeply into the Pt overlayer, while Pt atoms migrate into the Co layer and segregate in the near substrate region.

The resulting Pt and Co distribution profiles of the four-layered Pt/Co/Pt/Co stack after deposition, N^+ ion irradiation, single-stage annealing, and irradiation followed by the post-annealing are shown in Fig. 5. The chemical depth profile of the as-deposited film does not show clear interfaces (Fig. 5a). Only the upper Pt/Co modulation may be identified as two separate metal layers. Meanwhile, the lower Pt/Co interface has almost disappeared, resulting in the appearance of the signal from the CoPt complex ion. A surge in CoPt peak intensity and its position corresponds to the Pt layer due to the diffusion of Co into the Pt layer. These results agree with XRD data (Fig. 3a) and confirm the $CoPt_3$ phase formation. The driving force of such intermixing could be caused by the existence of residual stresses due to the lattice mismatch of the individual layers. The surface stress relaxation can explain the lack of mixing of the upper Pt/Co modulation.

After annealing, a complete uniform intermixing of the components is observed (Fig. 5b). Upon ion irradiation with different fluences, the distribution of Pt and Co atoms through the depth has not changed significantly compared to the as-deposited sample (Fig. 5c, e, g). Even at the maximum ion fluence, the layered structure is still observed. The two-stage processing of ion pre-irradiation followed by the annealing provides a homogeneous distribution of Pt and Co atoms through the entire film depth (Fig. 5d, f, h).

2.3. Magnetic characterization

Fig. 6 shows the $M(H)$ hysteresis loops of the bi-layered stack after deposition, N^+ ion irradiation, single-stage annealing, and irradiation followed by the post-annealing. The changes observed in the *M-H* hysteresis loops provide further evidence of the structural phase transformations and the alloy formation after the ion pre-irradiation and post-annealing process. We examine the critical magnetic parameters – saturation magnetization (M_s) and coercivity (H_c), derived from the $M(H)$ loops as a function of the ion fluence. The analysis of the magnetic hysteresis loops of the

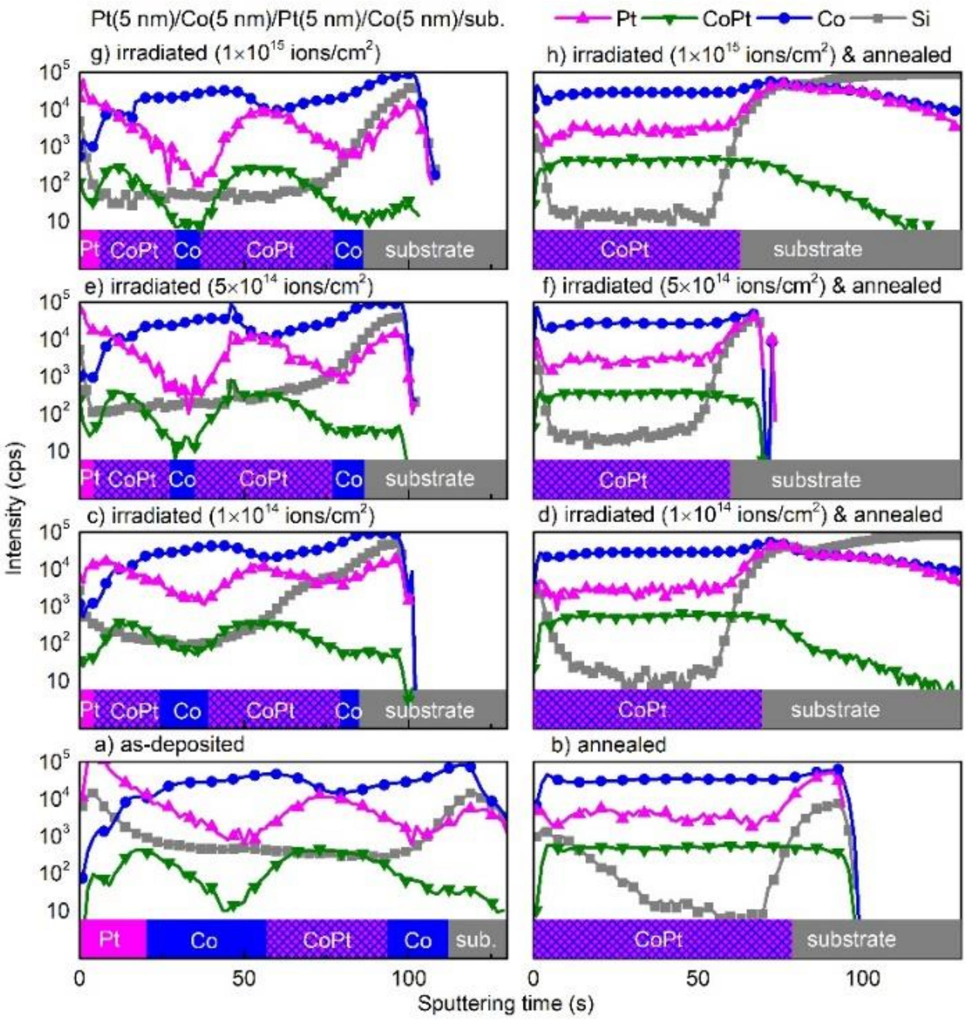

Fig. 5. SIMS chemical depth profiles of the four-layered Pt/Co/Pt/Co stack after deposition (a), N^+ ion irradiation (c, e, g), single-stage annealing (b), and irradiation followed by the post-annealing (d, f, h). Pt peak intensity was normalized to the total secondary ions' intensity.

films after deposition and post-growth treatment revealed the presence of in-plane magnetic anisotropy due to the shape anisotropy. The H_c and M_s values of the as-deposited film are 8.5 Oe and 527 emu/cc, respectively (Fig. 6a). While after annealing (Fig. 6b), the H_c significantly rises up to 302.5 Oe. This is well agreed with the mentioned XRD and SIMS data, which is conditioned by the formation of the soft magnetic *A*1-CoPt phase. There are no significant differences between the samples irradiated at 1×10^{14} ions/cm² and 5×10^{14} ions/cm² ion fluences in terms of the M_s and H_c values. Further increase of the ion fluence up to 1×10^{15} ions/cm² leads to a reduction of M_s to the value of 650 emu/cc, which means that ion-induced structural changes significantly affect the magnetic characteristics. In contrast, pre-irradiated and post-annealed bi-layered stack shows remarkable thermal stability of the magnetic properties upon increase of the ion fluence (Fig. 6d, f). After 1×10^{14} ions/cm² and 5×10^{14} ions/cm² ion pre-irradiation, H_c and M_s were 191 Oe, 1051 emu/cc, and 180.5 Oe, 1107.5 emu/cc, respectively. The increase of ion fluence up to 1×10^{15} ions/cm² leads to a drastic reduction of H_c to 34 Oe (Fig. 6h), which is associated with uneven distribution of paramagnetic Pt by the film depth and its segregation near the substrate.

Fig. 7 shows the $M(H)$ hysteresis loops of the four-layered stack after deposition, N^+ ion irradiation, single-stage annealing, and pre-irradiation, followed by post-annealing. At the as-deposited state (Fig. 7a) and after ion irradiation (Fig. 7c, e, g), noticeable changes in magnetic parameters are not observed, and magnetic properties remain similar to the ones observed for the bi-layered stack. In contrast, there is a lower H_c of 148 Oe after single-stage annealing, which is related to intermixing at the deposition stage and soft magnetic $CoPt_3$ phase formation (Fig. 7b). The irradiation-induced modification of the magnetic curves is visible upon the rise of the ion fluence. The N^+ ion irradiation followed by the post-annealing leads to a gradual increase in coercivity up to 251 Oe and 498 Oe for 1×10^{14} ions/cm² and 5×10^{14} ions/cm², respectively. Finally, for the highest fluence of 1×10^{15} ions/cm², the H_c drops to 212 Oe. All mentioned changes in magnetic properties are supposed to be attributed to the different effects of the ion pre-irradiation on the formation of structural defects and intrinsic strain states.

To get a deeper insight into the origin of irradiation-induced modification of the magnetic properties, a relative

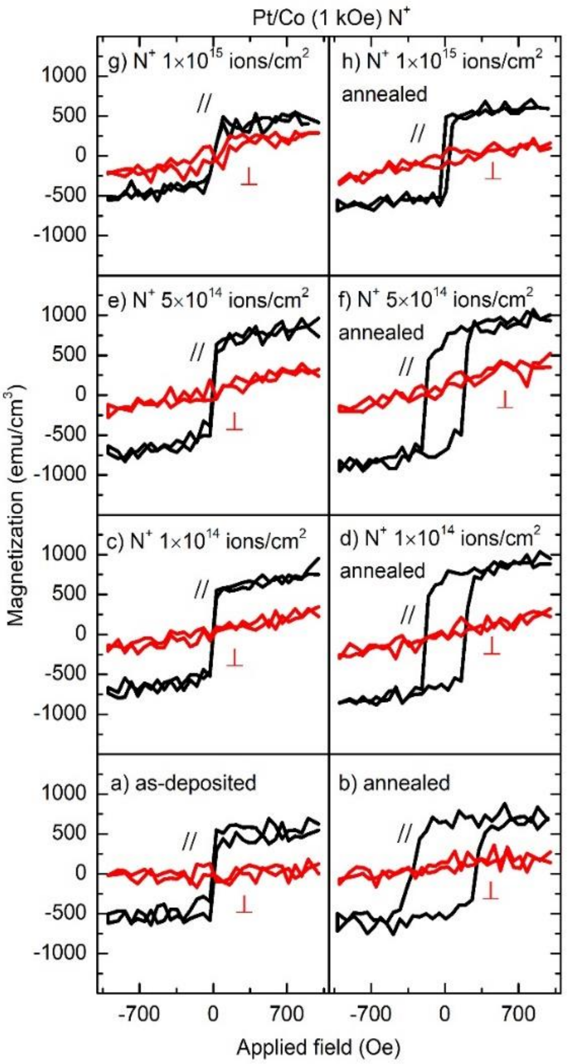

Fig. 6. *M-H* hysteresis loops of the bi-layered Pt/Co stack after deposition (a), N^+ ion irradiation (c, e, g), single-stage annealing (b), and pre-irradiation followed by the post-annealing (d, f, h).

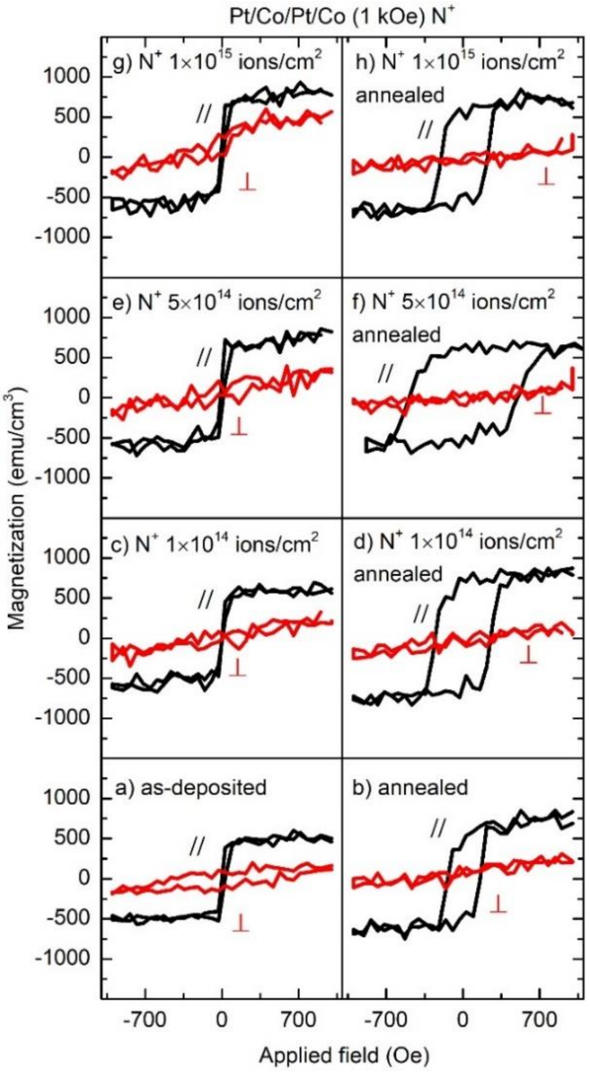

Fig. 7. *M-H* hysteresis loops of the four-layered Pt/Co/Pt/Co stack after deposition (a), N^+ ion irradiation (c, e, g), single-stage annealing (b), and pre-irradiation followed by the post-annealing (d, f, h).

comparison of structural changes caused by diffusion intermixing that is enhanced by additional defect generation with hysteresis loops is required. Fig. 8a, and 8b show plots of H_c and M_s of the bi-layered and four-layered stacks after different processing regimes as a function of *A*1-CoPt *d*-spacing and ion fluence. A non-linear change of saturation magnetization for all samples can be seen (Fig. 8a). In a bi-layered stack after two-stage processing, the H_c values degrade with the increase of the ion fluence compared to the single-stage annealing. At the same time, in the stack with a smaller modulation period, the H_c possesses the opposite trend towards its significant rise with the fluence. Since the irradiation induces additional point defects, it will likely enhance the atomic mobility, thereby favoring the thermally-induced intermixing. However, beyond certain fluence, it may change the nuclear arrangement. The results show the maximum $H_c = 498$ Oe for the four-layered stack irradiated with the fluence of 5×10^{14} ions/cm². The nature of these modified magnetic characteristics may be related to the increase of lattice *d*-spacing (2.164 Å) of the soft magnetic *A*1-CoPt phase. Thereby, such a lattice stretching could lead to an estrange of the part of Co atoms with a reduction of their exchange interaction, leading to the increment in coercivity.

Conclusions

In summary, in this work, we investigated the effect of the modulation period (20 nm and 12 nm) of the Pt/Co thin films on the evolution of their structure and magnetic properties induced by N^+ ion pre-irradiation followed by the vacuum annealing (550°C for 30 min). The increase of the ion fluence from 1×10^{14} ions/cm² to 5×10^{14} ions/cm² leads to significant structural changes, resulting in a rise of *A*1-CoPt phase *d*-spacing up to 2.164 Å. The *M*(*H*) magnetic loop reveals the highest H_c of 498 Oe, with M_s of 739,5 emu/cc. The obtained results may be associated with the modification of exchange interaction between Co

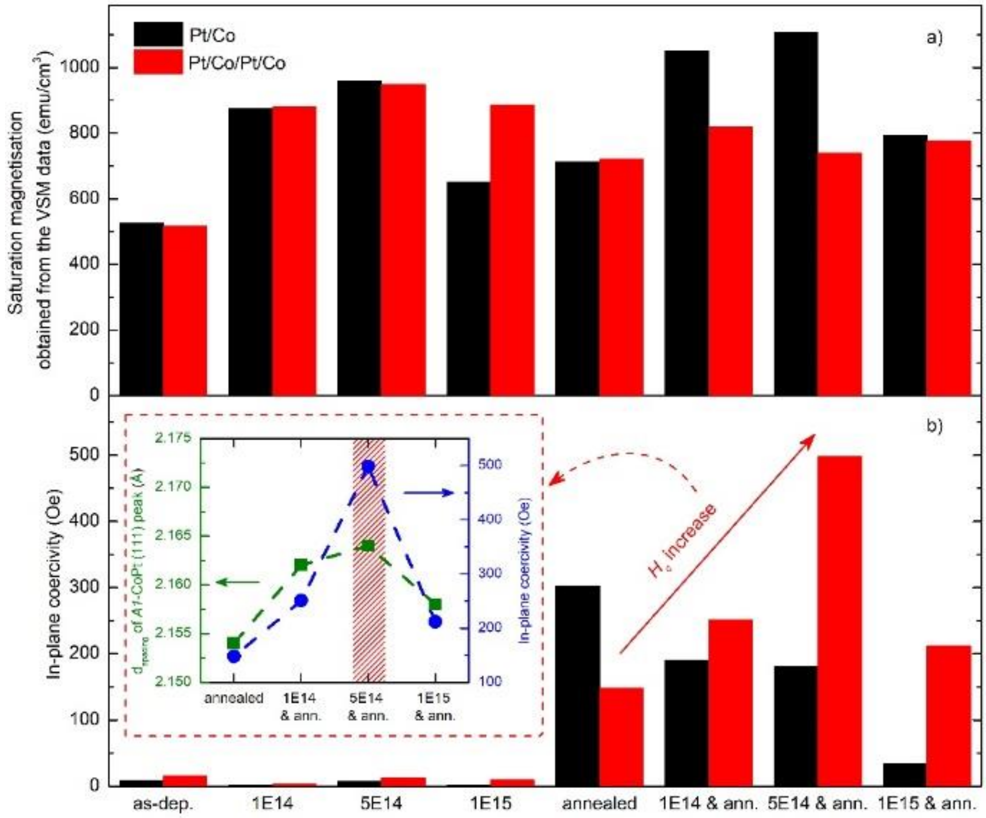

Fig. 8. Saturation magnetization (a) and in-plane coercivity (b) of the bi-layered Pt/Co and four-layered Pt/Co/Pt/Co stacks after deposition, N^+ ion irradiation, single-stage annealing, and pre-irradiation followed by the post-annealing. Inserts show the corresponding comparison of in-plane coercivity and *d*-spacing of the *A*1-CoPt phase as a function of ion fluence.

atoms due to the expansion of the crystal lattice of the ferromagnetic *A*1-CoPt phase. The stack with a smaller modulation period is shown to be more susceptible to ion-induced defect formation.

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Вплив періоду модуляції на іонно-індукований дифузійний масоперенос та магнітні властивості тонких плівок Pt/Co

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Досліджено вплив періоду модуляції та попереднього опромінення іонами N^+ на перемішування шарів і модифікацію магнітних властивостей плівок Pt/Co та Pt/Co/Pt/Co. В роботі описано вплив флюенсу на кореляцію між структурними змінами та особливостями перемішування шарів. Експериментальний аналіз проводився з використанням РСФА, ВІМС та вібраційної магнітометрії. Спостерігалась нелінійна зміна намагніченості насичення для дво- та чотиришарових плівок. З результатів дослідження встановлено, що значне збільшення коерцитивної сили (498 Ое), при флюенсі 5×10^{14} іонів/см² у чотиришаровій плівці, пов'язано з іонно-індукованим колізійним змішуванням і більш вираженим утворенням структурних дефектів. Продемонстровано, що значне збільшення додаткових структурних дефектів і внутрішніх напружень призводить до розтягування кристалічної гратки фази *A*1-CoPt та зміни обмінної взаємодії між атомами Co в тонкоплівковій композиції.

Ключові слова: магнітні тонкі плівки, іонне опромінення, дифузія, сплав Co-Pt, інтерфейс, фазоутворення.