Structural ordering of laser-processed FePdCu thin alloy films

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Abstract: The Cu/Fe/Pd multilayers were transformed into L10-ordered FePdCu alloy by pulsed laser annealing. The initial multilayers were irradiated with 1, 10, 100, and 1000 laser pulses with duration time of 10 ns and energy density of 235 mJ/cm². The gradual change of the number of laser pulses allowed to investigate the structural and magnetic properties at early stages of the transformation and L10-ordering processes. The measurements were carried out using X-Ray Diffraction, SQUID magnetometry, and Magnetic Force Microscopy. We found that L10 FePdCu (111)-oriented nanograins are formed by ordering of the coherent domains present in the as-deposited multilayer. The irradiation does not change the vertical size of the (111) crystalites. The L10 (002)-oriented grains appear at the later stages of the transformation and their size increases with the number of applied laser pulses. Additionally, the laser annealing induces the magnetic ordering of the irradiated material, which was observed as an increase of the saturation magnetisation and the Curie temperature with the rising number of pulses. We also observed that irradiation with 1000 pulses leads to the loss of order, which is reflected in the drop of the Curie temperature.

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1 Introduction

The development of new materials for the magnetic storage industry requires investigation of the new methods of their fabrication. Magnetic thin alloy films with L10 crystal structure such as FePd, FePt and CoPt, which exhibit a large uniaxial magnetic anisotropy of a few MJ/m³, are considered as potential materials for the bit pattern high-density recording media with information density over 1 Tbit/in². Application of these materials in data storage devices with perpendicular recording requires the fabrication of (001)-textured film. In order to design a material with desired properties it is essential to investigate the mechanisms leading to the formation of the L10-ordered alloy. In the following paper we focus on the FePd alloy: it is less expensive than the other two Pt-based materials; it has a lower order-disorder transition temperature, while having similar saturation magnetisation; and it is considered a model system for an exchange coupled nanomagnet.

One possible way to obtain an L10-ordered FePd thin film is an epitaxial growth. However, this method is relatively complicated and therefore it is hard to apply it for mass production. Another way to fabricate a film is the co-deposition of the constituent materials, or the deposition of the multilayer and its transformation into L10-ordered alloy. The latter can be done by the conventional long annealing, rapid thermal annealing, or by the ion beam irradiation. In this paper we present a new approach for fabrication of the L10-ordered FePd thin films, which is based on the pulsed laser annealing. Among the magnetic materials with L10 structure only for FePt thin alloy films results were previously obtained with this method by application of microsecond or sub-microsecond laser pulses showing that it leads to the formation of an alloy with the desired crystal structure.

The application of the laser annealing gives high heating and cooling rates of the irradiated material, which cannot be obtained with other methods. The increase of a number of laser pulses allows the investigation of the material properties at the consecutive stages of the transformation between a multilayer and an alloy film. Information gained during these studies is essential for understanding of the processes leading to the creation of the ordered L10 alloys with particular crystallographic texture, which is of great interest from the point of view of materials engineering. Moreover, such results can be exploited in experiments on Direct Laser Interference Patterning of metallic thin films.

The FePd thin films investigated here have an addition of 10 at% of copper, since it was found that such addition lowers the ordering temperature and facilitates the fabrication of an L10-ordered alloy.

2 Experimental details

The Cu(0.2 nm)/Fe(0.9 nm)/Pd(1.1 nm)10 multilayers were deposited by thermal evaporation on the Si(100) substrates with a native SiO₂ oxide layer. The thicknesses of Fe and Pd layers were chosen in order to obtain equiatomic FePd alloy, and the Cu thickness corresponded to atomic amount of 10%. Before the deposition the Si substrates were ultrasonically cleaned in acetone and ethanol, and rinsed in de-ionised water. The pressure in the evaporation chamber during the deposition was of the order of 10⁻⁷ Pa. The constituent layers were deposited sequentially, with the evaporation rates of 0.5 nm/min for Fe and Pd, and 0.2 nm/min for Cu. The thickness of layers was controlled in-situ by quartz crystal microbalance. The samples were 5 mm by 5 mm in size in order to fit with the diameter of a laser spot. After the deposition the chemical composition of the evaporated multilayers was checked by Rutherford Backscattering...
Spectrometry, confirming the assumed stoichiometry with an average accuracy of ±4 at.%. The thickness and roughness of the deposited layers were verified by X-Ray Reflectometry (XRR). The measurements confirmed the nominal thicknesses of the layers with average accuracy of 5%. XRR experiments also showed that the roughness of a single layer was approximately 0.6 nm. This result indicates the presence of a significant intermixing between layers during the deposition process and suggests that deposited volume of Cu is not a continuous layer and all Cu atoms are mixed with surrounding Fe and Pd.

In order to form FePdCu thin alloy films the as-deposited Cu/Fe/Pd multilayers were annealed using the pulsed laser irradiation. The irradiation was carried out using the multimode Quantel YG980 Nd:YAG laser operated at first harmonic with a wavelength of 1064 nm. The duration of a single pulse was 10 nm with a repetition frequency of 10 Hz. The spatial divergence of an unfocused beam was 0.07 rad, and the beam spot diameter, measured at half of the maximum intensity, was 4.5 nm. The energy density of a laser beam was set by the Q-switch device and was controlled by Coherent FieldMax II energy meter. The energy distribution in the beam spot was not perfectly homogeneous; however, the irradiation with an increasing number of pulses should result in an uniform areal distribution of the deposited energy. Initial multilayers were irradiated with 1, 10, 100, and 1000 laser pulses. To prevent oxidation the annealing process was carried out in atmosphere of a flowing nitrogen. The energy density was 235 mJ/cm² per single pulse. The Θ/2Θ X-Ray Diffraction (XRD) patterns were collected using X’PertPro PANalytical diffractometer, equipped with X-ray source with Cu anode (Kα1 line, wavelength λ=0.154 nm) operated at 40 kV and 30 mA. Experimental conditions were the same as described in Ref. [23].

The measurements of magnetisation as a function of the external magnetic field (hysteresis loops) as well as the temperature measurements of the magnetisation (field cooling, FC) were carried out using Quantum Design MPMS XL SQUID device. The hysteresis loops were collected at 300 K and FC curves were measured in temperature range from 5 K up to 300 K with step of 3 K. For hysteresis loops the external magnetic field step was 5 Oe, 100 Oe, 500 Oe, and 5 kOe and was dependent on the field range and on the measurement geometry. The FC curves were acquired for in-plane geometry with the external magnetic field of 100 Oe. Hysteresis loops were measured for in-plane and out-of-plane geometries, with the external magnetic field directed at angles between 0° and 90° with respect to the sample plane. The atomic and magnetic force microscopy (AFM/MFM) images were taken using Digital Instruments NanoScope device. The AFM measurements were done in the contact mode, while the MFM scans were collected in the non-contact phase contrast mode. In the MFM the distance between the probing tip and the sample surface was approximately 200 nm, the frequency of unloaded cantilever free oscillations was 73 kHz. The probing tip was magnetised vertically with respect to the sample plane. Both AFM and MFM images were collected sequentially for the same area on the sample surface. The probing frequency was 0.6 Hz.

3 Results and discussion
The energy of the laser pulses was transferred by electronic excitations to the crystal lattice, which led to the rise of the sample temperature and enabled the rapid diffusion processes within the irradiated material. The applied laser energy density corresponded to the maximal temperature of the sample surface of approximately 500°C. This temperature is necessary for transformation of the Cu/Fe/Pd multilayer into an ordered FePdCu alloy with L1₀ structure. The temperature during irradiation was estimated using theory of the pulsed laser annealing and a heat transfer equation. The absorption coefficient for wavelength of 1064 nm, essential for the calculations, was 28% and was determined by optical measurements. According to the applied computational model of the annealing process the temperature of the irradiated sample rose from the room temperature up to the maximum value in 10 ns and dropped down to the room temperature before the next pulse was switched on. It is worth mentioning that the model applied here does not take into account the temperature dependencies of the material properties, such as the radiation absorption, density, specific heat and thermal conductivity, and assumes that between the subsequent laser pulses the values of these parameters do not change.

3.1 Structural ordering
The XRD patterns of the as-deposited multilayer and irradiated samples are shown in Fig. 1. For the as-deposited multilayer two reflections at angles 2Θ = 37.6° and 41.7° were observed, and were identified as 1-st and 9-th order peaks related to the periodically layered structure of the as-deposited sample. The (111), (002), and (200) reflections from the L1₀-ordered crystallites are indicated in the figure. Patterns were shifted vertically for clarity.

Figure 1: XRD patterns of the multilayer and samples irradiated with 1, 10, 100, and 1000 laser pulses. The open circles are the measurement data, the solid lines are fits of the pseudo-Voigt functions. The (111), (002), and (200) reflections from the L1₀-ordered crystallites are indicated in the figure. Patterns were shifted vertically for clarity.
an evidence for the disappearance of the periodic multilayer structure. However, the observation of only one reflection makes the precise determination of the crystal structure impossible, since the angular position of this reflection matches both (111) reflection from an L1₀-ordered alloy and from a disordered fcc FePd system.⁹⁰⁰⁰

After irradiation with larger number of laser pulses two reflections were observed for 2θ of 41,7° and 48,9°. These peaks were identified as (111) and (002) reflections originating from the L1₀-ordered crystallites. For sample irradiated with 10 pulses the additional reflection was recorded at 47,5°, and it was identified as the L1₀ (200) peak. The lattice parameters of the irradiated material were calculated using the Bragg equation and the angular positions of the reflections, and they had the values of a = 0,380 ± 0,001 nm and c = 0,371 ± 0,001 nm, giving the lattice distortion c/a = 0,98 ± 0,01. Both lattice parameters and distortion are close to the values for bulk FePd alloy⁹⁴ despite the presence of Cu, although it is known that the Cu addition changes the parameters and increases the lattice distortion.⁹⁵⁹⁶ In this case the observed effect may indicate a low chemical order in the irradiated samples. The appearance of the reflection at 41,7° in each irradiated sample means that the structural domains with interplanar distance of about 0,217 nm are present in all cases. The effect of the pulsed annealing was the gradual diffusion of the elements leading to the transformation of the as-deposited multilayer into the a thermodynamically stable grained alloy phase with an L1₀-ordered crystal structure.

The observed XRD reflections were fitted with pseudo-Voigt functions. Applying the Scherrer equation

\[ D = \frac{K_S \lambda}{FWHM \cos \theta_0}, \]

and using the full width at half maximum of the Cauchy component \((FWHM_C)\) of the pseudo-Voigt function, the information about coherence length \(D\) (grain size along the normal to the sample plane) was obtained. The value of the Scherrer constant \(K_S\) was 0,95.⁹⁷ The wavelength \(\lambda\) was 0,154 nm, and the \(2\theta_0\) was the angular position of the reflection maximum. The microstrains \(\sqrt{\langle \varepsilon^2 \rangle}\), being a root-mean-square of the interplanar distance distribution around the average value, were calculated using the equation

\[ \sqrt{\langle \varepsilon^2 \rangle} = \frac{w_G}{\sqrt{8 \ln 2 \tan \theta_0}}, \]

where \(2w_G\) is the full width at half maximum of the Gauss component of the pseudo-Voigt function. The results of the calculations are shown in Fig. 2.

The vertical size \(D\) of the structural domains with interplanar spacings of approximately 0,217 nm does not change significantly with an increasing number of laser pulses, and has an average value of approximately 9 nm. On the other hand the microstrain \(\sqrt{\langle \varepsilon^2 \rangle}\) decreases from 0,013 ± 0,001 for the as-deposited multilayer to 0,007 ± 0,001 after application of at least 10 pulses. This means that the laser annealing results in the strain release for (111) grains, which in turn leads to the structural ordering of the material. The (111)-oriented L1₀-ordered nanocrystallites with narrow height distribution originate from structural superlattice domains present in the as-deposited multilayer. For the L1₀-ordered FePdCu structure the (111) crystallographic plane has the lowest surface energy. Since the system naturally tends to decrease its total energy, the appearance and ordering of the crystallites with such planes aligned parallel to the sample surface is favourable. After application of more than 10 laser pulses there was no further change of height of the (111)-oriented grains.

The (002)-oriented L1₀-ordered crystallites appeared after irradiation with at least 10 laser pulses. The height of these crystallites increased monotonically with a number of laser pulses, while no clear relation between number of pulses and microstrains was observed. In our previous study⁹⁸ we found that (002) crystallites are elongated in the direction parallel to the sample surface. Thus, it can be figured out that these crystallites appeared as the result of the heterogeneous nucleation at the interfaces of irradiated multilayer. This indicates that in case of the (002) crystallites the laser annealing leads to their creation and growth; however, it does not remove the microstrain, which is demonstrated by the lack of relation between a number of laser pulses and microstrains. The appearance of these grains can be attributed to the rapid temperature changes during heating and cooling of the sample, which could promote the occurrence of the nonequilibrium thermodynamic conditions favouring the mechanical stress and formation of (002)-oriented instead of (111)-oriented crystallites.

3.2 Magnetic properties

Following the laser annealing a set of magnetic measurements was carried out to obtain information about the change of magnetic properties. The hysteresis loops obtained for out-of-plane and in-plane geometries of the as-deposited multilayer and laser-annealed samples are demonstrated in Fig. 3. For the in-plane geometry the saturation field had value of approximately 3 kOe, while for out-of-plane configuration the value of about 20 kOe was recorded. This suggests, that the easy axis of magnetisation is parallel to the sample plane.

The measurements of the hysteresis loops were supported with the field-cooling experiments, which are shown in Fig. 4. The values of the Curie temperature \(T_C\) were obtained from the fit of the field-cooling curves with the function:

\[ \frac{M(T)}{M(T = 0 \text{K})} = \left[1 - \left(\frac{T}{T_C}\right)^\beta\right]^\gamma, \]

where \(\beta\) is the critical exponent for ferro-para magnetic phase transition. In this case \(\beta\) had a value of 0,33 ± 0,01,
Figure 3: The magnetic hysteresis loops \( M(H) \) measured for in-plane and out-of-plane geometries of as-deposited and laser-irradiated samples.

Figure 4: An example of the field-cooling measurements for as-deposited multilayer and sample irradiated with 100 pulses, carried out for in-plane geometry with external magnetic field of 100 Oe. Points are data, black solid lines are the fits.

Figure 5: The saturation magnetisation \( M_s \) and Curie temperature \( T_C \) as a function of a number of laser pulses. The inset shows the SEM image of the sample surface after 1000 pulses.

Since there are significant changes in the film morphology, the measured value of the magnetisation was assigned to the larger volume of the film than the measured amount of the material. This effect is likely responsible for the lowering of saturation magnetisation after 1000 pulses, although it cannot be excluded that the loss of ordering induced by the laser annealing is also the reason for the lower \( M_s \) in this case.

The coercivity field \( H_c \) observed in hysteresis loops demonstrated in Fig. 3 shows that for the non-epitaxial \([\text{Cu}/\text{Fe/Pd}]_{10}\) multilayer, where ferromagnetic Fe layers were decoupled from each other by the non-ferromagnetic Pd/Cu layers with comparable thickness, the coercivity was 13 Oe for in-plane and 60 Oe for out-of-plane. It can be expected that after laser treatment inducing the ordering, there will be a significant increase of \( H_c \). However, laser annealing led only to a slight change of the coercivity resulting in the coercivity of 15 – 45 Oe for in-plane geometry, and 60 – 110 Oe for out-of-plane, which was about two orders of magnitude lower than for bulk FePd alloy.

Since the ordering induced...
by irradiation may result in the increase of the exchange interaction between magnetic atoms within structural domains, the low value of coercivity recorded after irradiation can be related to the weak dipole magnetic interaction between the newly created L1$_0$-ordered grains. Therefore, it can be assumed that the L1$_0$-ordered nanocrystallites are separated from each other, with disordered material filling the volume between them, which does not provide the significant change in coercivity.

3.3 Magnetic domains and the mechanism of magnetisation reorientation

The direction of the easy axis of magnetisation and the mechanism of the magnetisation reversal were measured by the angle-dependent hysteresis loops for different angles $\psi$ between the direction of the external magnetic field and the sample plane ($\psi = 0^\circ$ for in-plane geometry and $\psi = 90^\circ$ for out-of-plane). The results of the measurements are presented in Fig. 6. For the discussion the sample irradiated with 100 pulses was chosen, since it exhibited the largest structural order after laser treatment.

The relation between the ratio of magnetic remanence $M_R$ to saturation magnetisation $M_s$ and angle $\psi$ is shown in Fig. 7. The change of the $M_R/M_s$ ratio with increasing angle $\psi$ is proportional to $\cos^2\psi$. This fact, together with a monotonic increase of the saturation field as a function of an angle $\psi$ from approximately 2 kOe to 25 kOe (Fig. 6), is a direct evidence that the easy axis of magnetisation is in-plane of the sample. The change of the coercivity field $H_c$ with angle $\psi$ is presented in Fig. 7. The angle-dependent values of the coercivity were normalised to the coercivity value $H_{c0}$ measured with the external magnetic field directed along the easy axis of magnetisation. The rise of the coercivity observed for an increasing angle $\psi$ followed the Kondorsky $1/\cos^2\psi$ relation indicating that the magnetisation reorientation process takes place by the domain wall motion. The small deviation of the coercivity from the $1/\cos^2\psi$ function seen for larger angles $\psi$ can be related to the presence of domain wall pinning sites.

Atomic and Magnetic Force Microscopy (AFM/MFM) measurements were carried out to obtain more information about domain structure and domain walls. An example of the MFM image obtained for sample irradiated with 100 laser pulses is presented in Fig. 5, together with a corresponding AFM image shown in Fig. 5. A cross-section of the line visible in MFM image is presented in Fig. 5. The asymmetrical shape of the cross-section is the evidence for the presence of magnetic stray fields, which are characteristic for Neel-type domain walls. The observed domain wall widths $d_w$, determined from MFM image, were in range from 80 nm to 150 nm with an average value of 125 ± 31 nm. Using the values of domain wall widths it is possible to estimate a value of the anisotropy energy $K_u$ by applying the equation:

$$K_u = \pi \mu M_s^2 \cos^2 \psi$$

where $J$ is the exchange interaction energy, taken for thin FePd alloy films and equal to $1 \cdot 10^{-11}$ J/m. The values of the anisotropy energy $K_u$ for different domain wall widths, calculated from (4), are in the range from $4 \cdot 10^4$ J/m$^3$ to $1 \cdot 10^5$ J/m$^3$, and are about two orders of magnitude lower than anisotropy energy for a well-ordered L1$_0$ FePd alloy. Such low anisotropy energy can be a result of the poor chemical order of the material and the grained sample microstructure with the nanocrystallites separated from each other.

The anisotropy energy could be compared with the magnetostatic energy $E_m$, expressed by the equation

$$E_m = \frac{\mu_0 M_s^2}{2} \cos^2 \psi$$

where $M_s$ is the saturation magnetisation. The maximum value of $E_m$ was obtained for the direction perpendicular to the sample plane (angle $\psi = 0^\circ$). Taking into account the measured saturation magnetisation (see Fig. 5) the magnetostatic energy $E_m$ for angle $\psi = 0^\circ$ after irradiation with 100 pulses has a value of $7 \cdot 10^4$ J/m$^3$. The value of the anisotropy energy $K_u$ is larger than the magnetostatic energy $E_m$ for angle $\psi$ greater than $85^\circ$ in agreement with the in-plane easy axis of magnetisation. Relatively low anisotropy energy caused the broadening of the domain wall, which also decreased the coercivity field, since the energy cost for domain wall movement was smaller than for well-ordered material with high anisotropy energy.

4 Conclusions

In this paper we present the results of the research on structural and magnetic ordering induced in Cu/Fe/Pd multilayers by the pulsed laser annealing. The laser annealing allowed us to study the structural and magnetic properties of the material at the early stages of the transformation from a multilayer to an alloy. The laser annealing of the Cu/Fe/Pd multilayers led to the formation of the partially L1$_0$-ordered FePdCu nanocrystallites. It was found, that single laser pulse causes the disappearance of the periodically layered structure of the initial sample, and after application of a larger number of laser pulses the L1$_0$-ordered nanocrystallites started to appear. First, as the result of the laser irradiation the (111)-oriented grains raised from the coherent domains present in the initial superlattice, and the annealing process led to partial structural ordering of these crystallites, which was reflected in the decrease of microstrains. However, the laser annealing did not change the vertical size of these grains. The (002)-oriented grains were formed after at least 10 laser pulses by ordering of the material at the interfaces of the multilayer. The further irradiation caused the monotonic increase of the vertical size
of these crystallites; however, it did not provide the well-ordered grains. The magnetic ordering of the irradiated material was observed as the increase of the saturation magnetisation and the Curie temperature for increasing number of the laser pulses. However, the degree of order induced by laser irradiation was not sufficient to provide the magnetocrystalline anisotropy large enough to overcome the magnetostatic energy, which resulted in orientation of the easy axis of magnetisation in-plane. The magnetisation reversal took place by the Neel-type domain wall motion. It was also found, that irradiation with 1000 pulses leads to the loss of ordering, which can be attributed to the annealing of the sample at higher temperature than predicted by the theoretical calculations. This results in a partial material ablation.

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