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Chemical ordering of FePt films using millisecond flash-lamp annealing

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The structural and magnetic properties of 20-nm-thick $\text{Fe}_x\text{Pt}_{100-x}$ films that were processed by 20 ms flash-lamp annealing were investigated. A maximum in coercivity of (10.4 ± 0.5) kOe was achieved for a composition of $\text{Fe}_{53}\text{Pt}_{47}$, which shows also a high degree of $L1_0$ chemical order. A variation of the chemical composition toward either higher or lower Fe content leads to a lowering of the coercivity, which can be attributed to a reduction in $L1_0$ ordered volume fraction. Thus, in the millisecond time regime, the fastest ordering transformation occurs for slightly Fe-rich FePt films. © 2012 American Institute of Physics. [doi:10.1063/1.3677991]

I. INTRODUCTION

FePt in the chemically ordered $L1_0$ phase is considered to be a promising material for future data storage devices,^{1,2} magnetic sensors,³ or permanent magnets^{4,5} due to its high uniaxial magnetocrystalline anisotropy constant of $\sim 10^8$ erg/cm³, large saturation magnetization, and corrosion resistance superior to rare-earth based magnets.⁶ However, when FePt is deposited at room temperature, it grows in the chemically disordered A1 phase and the transformation into the chemically ordered $L1_0$ phase typically involves a post-annealing step to temperatures between 500 °C and 900 °C.⁷⁻⁹ In the last years, a continuous effort has been made not only to reduce the required annealing temperature but also to reduce the required annealing time utilizing laser annealing or millisecond plasma arc pulses. These short annealing times can effectively prevent interdiffusion and grain growth and would enable a large throughput minimizing the production costs. While no ordering was observed when the annealing time of the laser pulses was in the nanosecond time scale,¹⁰ partial order can be induced when a pulse width in the range of 2.5 ms to 10 ms was employed.¹¹⁻¹³ Partial ordering was also observed using plasma arc annealing with annealing times of 50 ms and 100 ms.¹⁴ Very recently, the time-temperature-transformation (TTT) diagram for a binary FePt film with a thickness of 10 nm has been experimentally determined, which shows that the onset of chemical ordering occurs after 1.1 ms at an annealing temperature of 700 °C.¹⁵ These results are in good agreement with TTT calculations additionally pointing out the impact of the chemical composition on the processing time required for a complete ordering transformation.¹⁶ Flash-lamp annealing is another millisecond annealing process capable to heat thin films to temperatures above 1300 °C.^{17,18} and the prospects of this method for advanced semiconductor processing have recently been demonstrated.¹⁸⁻²⁰

In the present study, flash-lamp annealing with 20 ms light pulses was employed to process 20-nm-thick $\text{Fe}_x\text{Pt}_{100-x}$ films (with $x = 42 - 60$) sputter deposited at room temperature. The determination of the magnetic and structural properties with respect to chemical composition and annealing temperature gives further insight into the relevant parameters of the ordering transformation in the millisecond time regime.

II. EXPERIMENTAL

20-nm-thick $\text{Fe}_x\text{Pt}_{100-x}$ films of various composition with $x = 42 - 60$ have been deposited at room temperature by magnetron co-sputtering (Ar pressure 3.5×10^{-3} mbar) from individual Fe and Pt targets on temperature stable (up to 650 °C) OHARA TS-10SX glass-ceramic substrates. The composition was controlled by individually adjusting the sputter rates of Fe (Pt) and later on verified by Rutherford backscattering spectrometry (RBS). The samples were transferred to a separate flash-annealing setup, where the annealing was performed using 20 ms light pulses under N_2 atmosphere. No pre-heating was applied and the energy density of the light pulse was controlled via the capacitor charge. The irradiation of the utilized Xe flash-lamps is primarily in the visible spectrum with two maxima around 470 nm and 570 nm and the shape of the light pulse reveals typical rise and fall times of around 4 ms and 10 ms, respectively. Further details of the flash-annealing setup are described elsewhere.¹⁷⁻¹⁹ The structural properties of the samples were investigated by x-ray diffraction using beamline G3 of HASYLAB at DESY (wavelength $\lambda = 1.54185$ Å) and their surface morphology was analyzed by atomic force microscopy (AFM). The integral magnetic characterization was performed using superconducting quantum interference device (SQUID) magnetometry with magnetic fields up to 70 kOe.

III. RESULTS AND DISCUSSION

In the as-grown state, all $\text{Fe}_x\text{Pt}_{100-x}$ films reveal only one diffraction peak corresponding to the (111) reflection.

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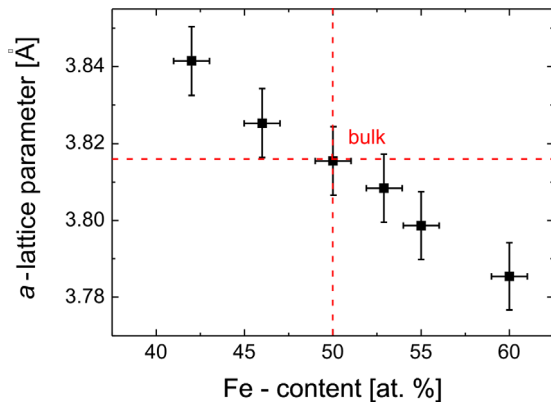


FIG. 1. (Color online) Dependence of the a lattice parameter on the Fe content. Please note that the (111) reflection was used for its determination.

The position of this peak was used to determine the a lattice parameter of the chemically disordered phase by applying the Bragg equation for fcc crystallographic systems.²¹ A linear dependence of the lattice parameter on the Fe content was observed (Fig. 1) following Vegard's law²² which is in agreement with the composition analysis performed by RBS.

For a chemical composition of $\text{Fe}_{53}\text{Pt}_{47}$, x-ray ($\theta - 2\theta$) diffraction patterns reveal no further diffraction peaks after flash-lamp annealing with a capacitor voltage (V_C) up to $V_C = 2.4$ kV (Fig. 2(a)). Increasing V_C to 3.0 kV, however, leads to the formation of pronounced (001) and (110) superstructure peaks, as well as to a separation of the (200) and (002) reflections which is a characteristic feature of polycrystalline $L1_0$ ordered FePt films (Fig. 2). From the later sample the ratio of the integral intensities between the (001) and (002) reflection was used to determine the chemical order parameter S .²³ When neglecting the Debye-Waller factor, a chemical order parameter of about $S = 0.8 \pm 0.1$ can be estimated. Furthermore, AFM investigations (not shown), which were performed after flash-annealing showed a steady

increase in root mean square roughness from 0.43 nm up to 1.15 nm with higher capacitor voltage. This increase in surface roughness is a first indication of grain growth.

The magnetic properties are consistent with the results of the structural investigation. In the as-grown state (not shown) the magnetic properties are dominated by the shape anisotropy and an in-plane easy axis with a coercivity H_C of less than 100 Oe is observed. The anisotropy field H_a was determined to $H_a = (11.5 \pm 0.5)$ kOe giving an effective anisotropy $K_{\text{eff}} = [1/2] M_s H_a = (5.7 \pm 0.6) \times 10^6 \text{ erg/cm}^3$ using $M_s = (990 \pm 160) \text{ emu/cm}^3$. This effective value contains the contributions of the magneto-crystalline anisotropy and shape anisotropy ($2\pi M_s^2 = (6.2 \pm 1.3) \times 10^6 \text{ erg/cm}^3$). By comparing the two values it is evident that the magnetic properties of the as-grown FePt films are dominated by shape anisotropy as expected for chemically disordered FePt films with low magneto-crystalline anisotropy. Whereas only little change in the magnetic properties is observed after flash-lamp annealing using $V_C = 2.4$ kV (Fig. 3(a)), both the in-plane and out-of-plane coercivity increase substantially when the capacitor charge is increased and large coercivities up to $H_C = (10.4 \pm 0.5)$ kOe are achieved by applying $V_C = 3.0$ kV (Fig. 3(d)). In addition, the magnetic response is becoming isotropic and only little difference between the in-plane and out-of-plane M-H hysteresis loops are observable. This confirms the development of a polycrystalline film structure with substantial $L1_0$ chemical order in which the high uniaxial magneto-crystalline anisotropies of the individual but randomly oriented crystallites predominate over the shape anisotropy and lead to a magnetically isotropic behavior.

To study the dependence of the phase transformation on the chemical composition of 20-nm-thick $\text{Fe}_x\text{Pt}_{100-x}$ films, the perpendicular coherence length L_{Perp} and the in-plane coercivity was determined for various compositions and annealing conditions (Fig. 4). While L_{Perp} was calculated from the (111) peaks according to the Scherrer equation²⁴ and

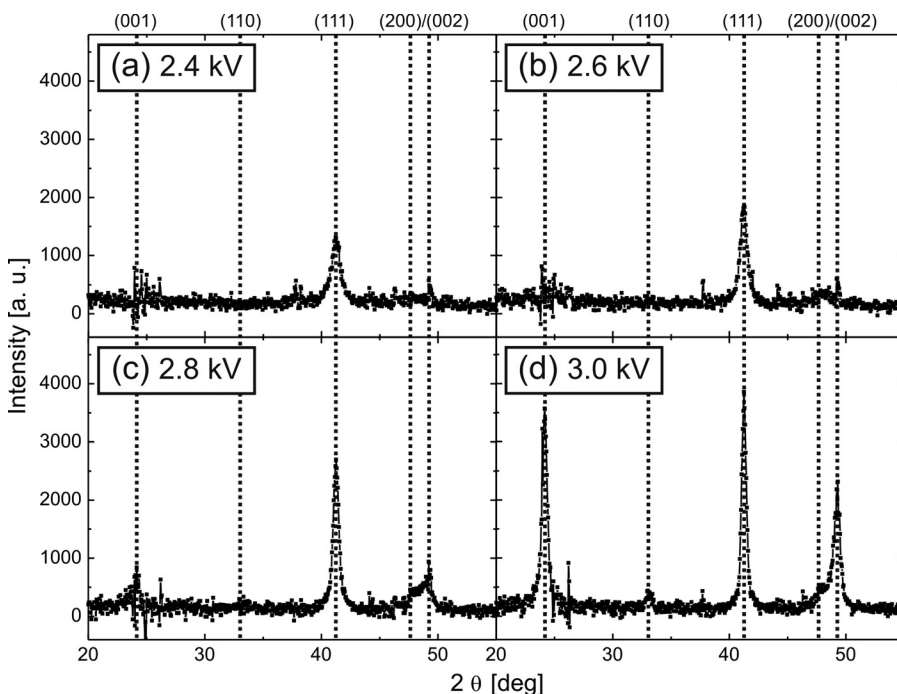


FIG. 2. X-ray ($\theta - 2\theta$) diffraction pattern of 20-nm-thick $\text{Fe}_{53}\text{Pt}_{47}$ films after flash-annealing using a capacitor voltage of (a) 2.4 kV, (b) 2.6 kV, (c) 2.8 kV, and (d) 3.0 kV. The positions of the (001), (110), (111), and (200)/(002) diffraction peaks of FePt in the $L1_0$ phase are indicated.

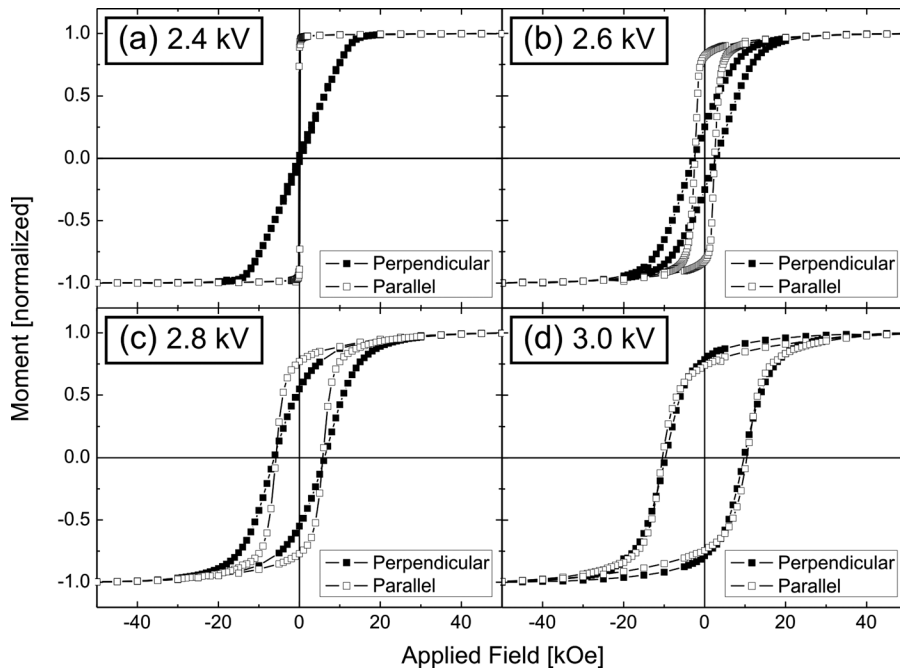


FIG. 3. M-H loops of 20-nm-thick $\text{Fe}_{53}\text{Pt}_{47}$ films after flash-annealing using a capacitor voltage of (a) 2.4 kV, (b) 2.6 kV, (c) 2.8 kV, and (d) 3.0 kV. The magnetic field was applied perpendicular and parallel to the film plane.

allows to probe the heat-induced grain growth, the coercivity in post-annealed FePt films shows typically a linear relationship with the L_{10} ordered volume fraction^{25,26} and therefore allows to probe the chemical ordering transformation. L_{Perp} of the as-grown films is around 8 nm for all chemical compositions. Substantial grain growth starts after flash-annealing

using a capacitor voltage $V_C \geq 2.4$ kV. Beside minor variations especially for lower annealing temperatures, all $\text{Fe}_x\text{Pt}_{100-x}$ films reveal a comparable L_{Perp} , which approaches the film thickness of 20 nm for $V_C = 3.0$ kV. As the evolution of the perpendicular coherence length is found to be independent on the chemical composition, it can be concluded that the evolution of L_{Perp} is also independent on the degree of chemical order. This indicates that the grain growth does not depend on the degree of chemical order and consequently L_{10} ordered grains do not grow more rapidly than the chemically disordered grains in the investigated thickness, time and composition range.

A pronounced maximum in coercivity is obtained for a Fe content of 53 at. % (Fig. 4(b)). However, previous studies have observed a rather broad compositional dependence of the coercivities in the limit of long annealing times.²⁷ Thus, the rather sharp maximum reveals that an annealing time of 20 ms is insufficient to induce a complete chemical ordering transformation over the entire investigated composition range. Additionally, the dependence of the coercivity on the chemical composition indicates that the highest volume fraction of chemically ordered grains has been formed for a composition of $\text{Fe}_{53}\text{Pt}_{47}$. Consequently, the ordering transformation has to occur substantially faster in slightly Fe-rich $\text{Fe}_x\text{Pt}_{100-x}$ films than in Pt-rich films. This behavior is in agreement with calorimetric studies on the phase transformation.^{28,29} However, the tendency that Pt-rich films should show a faster transformation kinetics in some temperature regimes in particular for ultra-thin films as indicated by model calculations of TTT diagrams¹⁶ could not be confirmed.

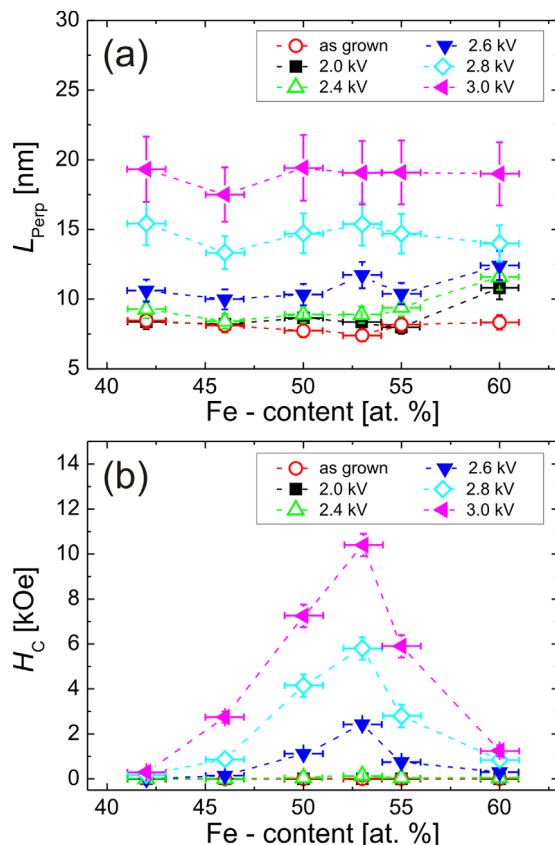


FIG. 4. (Color online) Dependence of (a) the perpendicular coherence length L_{Perp} extracted using the FWHM of the (111) reflection and (b) the in-plane coercivity H_C on the alloy composition and the capacitor voltage.

IV. CONCLUSION

It has been shown that flash-lamp annealing with 20 ms light pulses can be used to transform $\text{Fe}_x\text{Pt}_{100-x}$ films from the chemically disordered A1 phase into the chemically ordered L_{10} phase. A variation of the Fe content x from

$42 \leq x \leq 60$ revealed that the fastest ordering transformation occurs for a composition of $\text{Fe}_{53}\text{Pt}_{47}$, whereas the grain growth was observed to be independent on chemical composition and degree of chemical order.

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