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Role of structural defects on the magnetic properties of annealed Fe₅₆Pd₄₄ thin films upon development of the ordered *L*₁₀ phase

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ABSTRACT

Fe₅₆Pd₄₄ thin films grown on a silicon substrate by thermal evaporation were submitted to annealing in furnace in vacuum at 550 °C for increasing time in order to transform the initial disordered fcc phase into an ordered *L*₁₀ one. Starting from a detailed understanding of the time evolution of the microstructure, the complex resulting magnetic properties have been interpreted. Besides the expected increase of the coercive field due to the development of a magnetically hard phase, a non-monotonous behaviour of the remanence-to-saturation ratio has been attributed to the effects of the ordering of the developing *L*₁₀ phase first, and of the progressive ordering of the alloy as a whole thereafter, once the *L*₁₀ phase was already fully developed. The process of ordering of the alloy is characterised by the appearance of microstructure defects (twinned domains, twin boundaries, antiphase boundaries, polytwins), that promote a further increase of the coercive field by pinning domain walls, but also affect the remanence-to-saturation ratio and the arrangement of the magnetic domains by locally tilting and reducing the magnetic anisotropy.

1. Introduction

Ferromagnetic *L*₁₀ ordered alloys, such as FePt and FePd, have been extensively studied for applications as recording media, where large uniaxial magnetocrystalline anisotropy (of the order of 10⁶-10⁷erg/cm³) is required [1], as well as good thermal stability. The *L*₁₀ ordered FePd systems have been fabricated as thin films and nanoparticles. In the former case, several techniques such as molecular-beam epitaxy, sputtering, thermal evaporation [2–6], as well as electro-deposition [7] have been exploited. For the preparation of FePd nanoparticles, electron beam deposition, postdeposition annealing [8], chemical synthesis [9], and modified polyol process [10] are among the most commonly used techniques.

However, most synthesis or growth methods develop FePd systems that exhibit a disordered face centered cubic (fcc) structure at room temperature, which behaves as a soft ferromagnetic material. It is therefore required to anneal the system, usually at 400–700 °C, for transforming the disordered fcc structure into an ordered face centered tetragonal (fct) one, the *L*₁₀ phase, that is chemically ordered [11,12]. This phase is constituted by alternating pure Fe and Pd atomic planes along the c-axis direction of the face centered cubic lattice, which is therefore distorted: the lattice parameters of the *L*₁₀ FePd phase are *a* = *b* = 0.3871 nm and *c* = 0.3660 nm, giving an axial ratio, *c/a* = 0.945 [6].

The relevant magnetic properties of the FePd system depend, then, on its form (particles or thin films), degree of order of the *L*₁₀ phase, size (for particles), kind of substrate (for thin films), spanning relatively large intervals [4,13–15].

Such large variations (for example from below 1 kOe [4] to more than 8 kOe [14] for coercivity, from 3.8 MGOe of maximum energy product [15] to 10.3 MGOe [13]) cannot be explained without a detailed description of the systems microstructure. For example, Shima et al. [16] have obtained a sufficiently high coercivity from ultrathin films. Grain boundaries and twins have been reported to enhance coercivity in polycrystalline thin films [17]. Sablik [18] showed that coercive field increases with increasing dislocation

density and may decrease as grain size increases. The experimental results of Wang et al. [19] showed that the hard magnetic properties of FePt thin films are improved when another element such as Nitrogen (N) is added. The large coercivity in the $L1_0$ ordered alloys was implicated in the presence of structural defects, in the form of antiphase boundaries and twin boundaries [20]. Recently, it was shown that the perpendicular coercivity can be enhanced by increasing the stacking fault densities [21].

The role of structural defects (antiphase boundaries, twins, tweed structures, etc.) has therefore attracted increasingly more attention, as they can affect the magnetic properties of chemically ordered alloys in unexpected ways [20,22–24]. The presence of these defects has been investigated by the Lorentz microscopy imaging technique associated with the conventional transmission electron microscopy, which allows to visualize the magnetic domains [20,23]. Similar results were obtained using Mössbauer spectroscopy on FePd alloys prepared as a thin film [6]. These defects play a crucial role in determining the coercivity mechanism and the magnetization reversal [20,22–24], as the coercivity has been shown to increase due to the pinning of the magnetic domains walls by the antiphase boundaries and the twin boundaries, both experimentally [20,23] and numerically [25].

In this research, we investigate the role of structural defects in determining the magnetic properties of a $\text{Fe}_{56}\text{Pd}_{44}$ alloy prepared in thin films form. The numerous studies reported in literature, including those reported above, confirm the complexity of the relationship among microstructure, defects and magnetisation processes. With this paper we aim at addressing this subject on FePd alloys thermally evaporated and then submitted to annealing to promote the phase transformation of the disordered phase into the ordered one. During the transformation, the coercive field progressively increases as a function of the annealing time, whereas the hysteresis loops shape evolves displaying a rich features set, resulting in a non-monotonous behaviour of the remanence-to-saturation ratio and of the maximum energy product. The rapid transformation of the soft fcc phase into the hard fct one is not sufficient to explain the complex phenomenology that has been observed. Instead, the degree of order of the $L1_0$ phase and of the whole alloy have been taken into account, to infer the role of structural defects in determining the magnetic properties.

2. Experimental

$\text{Fe}_{56}\text{Pd}_{44}$ thin films have been deposited on Si substrates by means of thermal evaporation. Instead of the more common $\text{MgO}(001)$ or $\text{SrTiO}_3(001)$ substrates, Si is a cheaper option, even though it does not allow oriented growth of Fe-Pd films. The deposition system consists in a high vacuum chamber (base pressure 10^{-5} Pa) in which the substrate and the tungsten crucible are placed approximately 200 mm apart. In the crucible, the elemental Fe and Pd powders (with 99.99 % purity) are heated by a current of 124 A, resulting in an operating pressure of $7 \cdot 10^{-4}$ Pa.

In order to measure the thickness of the deposited film, a piece of the substrate has been cut and placed into a scanning electron microscope in a 90° tilted position, in order to reveal the metallic deposit cross section. The electron beam energy was 5 keV. The measured sample thickness turned out to be 80 nm with a relative error of 4 %.

The samples composition has been determined by means of energy dispersive X-ray spectroscopy, resulting in 56 at.% Fe and 44 at.% Pd, with a relative error of 2 %; the composition is slightly richer in Fe than the stoichiometric alloy, but still permits the development of the $L1_0$ phase.

Thermal treatments, aimed at promoting phase transformations in the as-deposited material, have been conducted at 550°C in a furnace in vacuum (base pressure 10^{-5} Pa), for selected annealing times from 1 min to 120 min. Annealing has been performed all the time on the same sample, progressively submitted to additional treatments in furnace summing up to the desired time, and characterised with all the available techniques at each annealing step.

Structural information has been gathered using grazing incidence X-ray diffraction, as detailed in [6], and microstructure, phase transformation with annealing time, and degree of order of the $L1_0$ phase and of the alloy have been studied in [26].

Magnetic hysteresis loops have been measured by means of a PMC MicroMag 2900 Alternating Gradient Field Magnetometer (AGFM), at room temperature, with the magnetic field applied in the sample plane. Besides the usual hysteresis parameters (coercive field, saturation, remanence), the maximum energy product has also been calculated, in the second quadrant of the hysteresis loop, as the field at which a magnetisation value equal to half of the remanence is reached, times that magnetisation value.

Finally, a Bruker Multimode V Nanoscope 8 Atomic Force / Magnetic Force Microscope (AFM/MFM) has been used to image the morphology and the magnetic domains of the as-deposited and annealed samples.

The tip (Si pyramidal, with CoCr coating and 400 Oe coercive field) was magnetised along the tip axis (i.e. perpendicularly to the sample plane), and operated in intermittent contact, two-pass lift mode. Topography and magnetic domains have been acquired at room temperature using the height channel in pass 1 and the phase channel in pass 2 respectively.

3. Results

X-ray diffractograms of a few selected samples are shown in Fig. 1. The complete dataset and the full data analysis can be found in [26]. Apart from the peaks of the Si substrate, the as-prepared sample displays a few peaks of the disordered fcc FePd phase, while the annealed samples all display peaks of the $L1_0$ phase, with different intensities and sharpness as a function of the annealing time. From the XRD data, the tetragonal ratio c/a of the $L1_0$ phase can be calculated (it is assumed equal to 1 in the as prepared sample, where only the fcc phase is present), as reported in Fig. 1(a). The c/a ratio is then used to calculate the order parameter of the tetragonal phase, S_{tetr} , as reported in [26] and in Fig. 1(b).

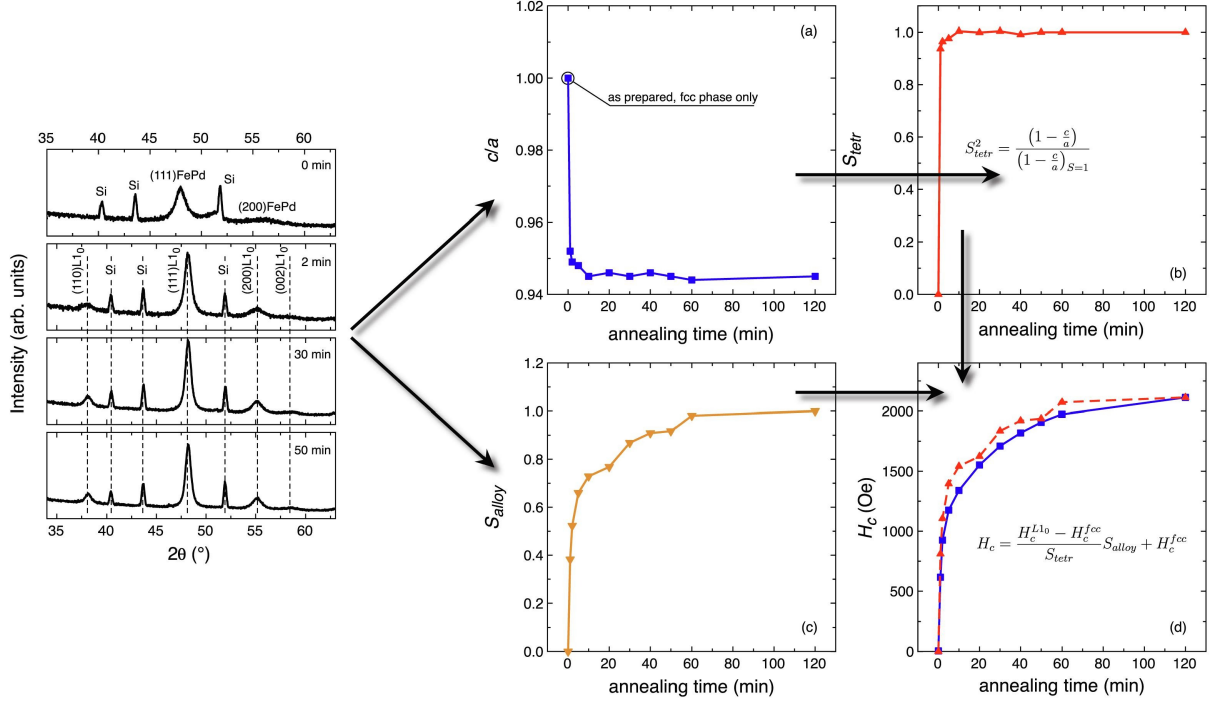


Fig. 1. XRD of selected Fe₅₄Pd₄₆ alloys for different annealing times. From XRD, the tetragonal ratio (c/a) (panel (a)) and the order parameter of the alloy (panel (c)) can be calculated. From the c/a ratio the tetragonal order parameter (panel (b)) is derived. From S_{tetr} and S_{alloy} , the coercive field of the alloy can be calculated (panel (d), red line and symbols), and compared with the experimental values (panel (d), blue line and symbols). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Without going into the details, it is important to mark the difference between two order parameters relevant for the discussion. S_{tetr} , previously introduced, describes the degree of ordering of the Fe and Pd atoms in the chemically ordered regions (the $L1_0$ grains). Conversely, a second order parameter, S_{alloy} , can be introduced, to represent the volume average of S_{tetr} , calculated by multiplying it by the volume fraction of the $L1_0$ phase [26]. S_{alloy} is then shown in Fig. 1(c).

Both order parameters, combined with the coercivity values of the pure fcc and $L1_0$ phases, permit to calculate the coercive field of the alloy for the different annealing times (Fig. 1(d), red curve and symbols), which can be compared with the coercive field obtained from hysteresis loops measurements (Fig. 1(d) blue curve and symbols). The detailed discussion on how the order parameters and the dependence of coercivity on them are derived can be found in [26].

The morphology of representative samples (see later for details) is shown in Fig. 2, where AFM images are reported for the as deposited (0 min) three annealed (2, 30, 50 min) samples. Overall, the samples display a rather homogeneous surface, without remarkable features, except for some crystals at long annealing times.

The root mean square roughness, calculated without considering the crystals for the samples annealed for 30 and 50 min, decreases with annealing time.

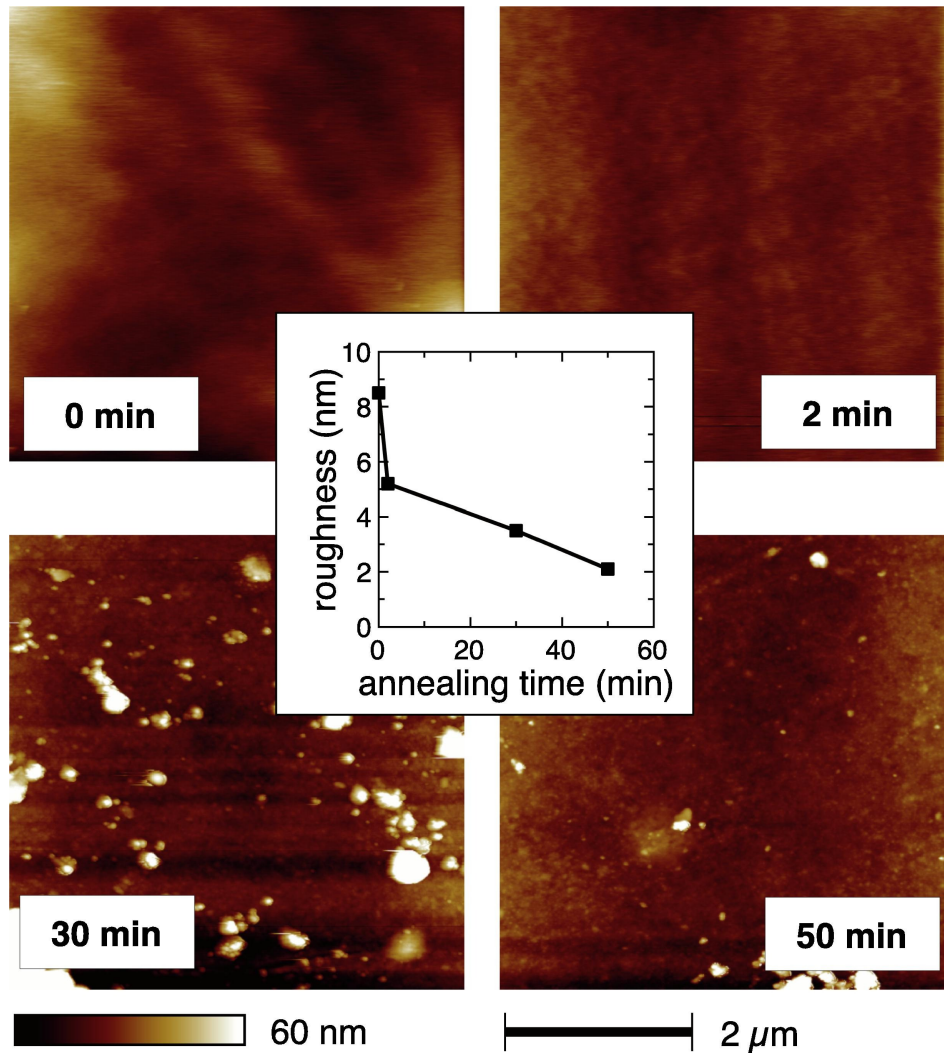


Fig. 2. AFM images of selected Fe₅₄Pd₄₆ alloys for different annealing times. In the inset: root mean square roughness as a function of annealing time, calculated excluding the white crystals on the surface in the samples annealed for 30 and 50 min.

The hysteresis loops of the as-prepared and annealed samples, shown in Fig. 3, fall into four families, depending on the annealing time. The first family is composed by the as-deposited sample alone, which displays a soft magnetic behaviour, with a coercive field of approximately 5 Oe (see Fig. 1(d) as well). Its relatively small remanence to saturation ratio suggests a disordered polycrystalline microstructure, involving progressive rotation of the magnetisation inside individual grains, overcoming the local (weak) anisotropy field. This behaviour is consistent with previous X-ray and Mössbauer studies that report the presence of a disordered fcc FePd phase (see [6,26] and Fig. 1).

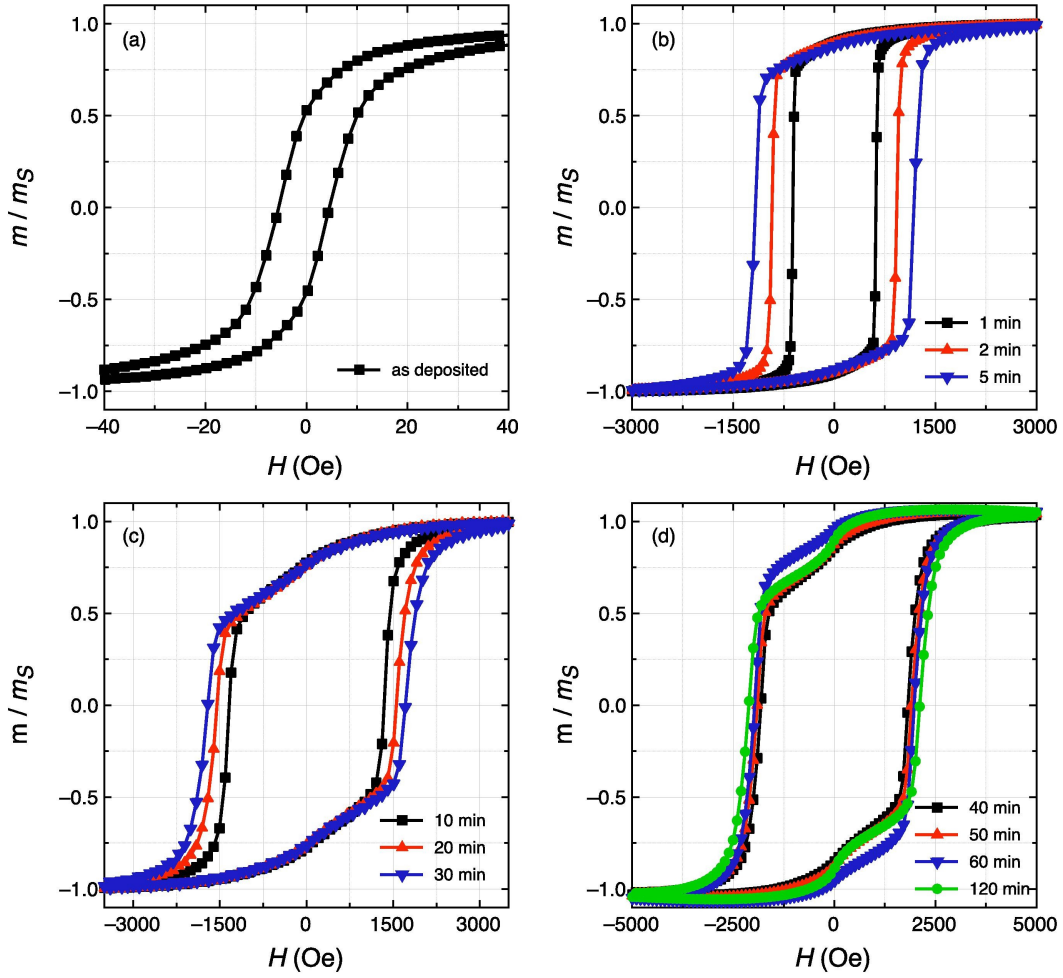


Fig. 3. Hysteresis loops of the Fe54Pd46 alloys as a function of the annealing time.

The second family of hysteresis loops consists of the shortest annealing times, from 1 to 5 min at 550 °C. These loops are characterised by a shape that is more rectangular, a sudden jump of coercivity, and a significant dependence of coercivity (that more than doubles its value) as a function of time. This behaviour is due to the development of the magnetically hard $L1_0$ phase, at the detriment of the fcc one, documented by the S_{tet} order parameter, which increases as a function of the annealing time, and by the c/a lattice parameter of the $L1_0$ phase, as reported in [6] and in Fig. 1.

The third family of hysteresis loops is populated by the samples annealed from 10 to 30 min, for which the coercive field increases (more slowly) up to approximately 1700 Oe, but a significant loop shape change takes place, with a reduced remanence to saturation ratio and a significant, reversible reduction of the magnetisation up to almost the coercive field. For these samples, the complete development of the $L1_0$ hard phase, and the complete disappearance of the fcc soft one, is assumed to be responsible for the observed magnetic behaviour.

Finally, the fourth family consists of the samples annealed from 40 to 120 min, for which the coercive field increases still some more, up to approximately 2100 Oe, and the remanence to saturation ratio increases again towards unity, but a “shoulder” appears in the magnetisation curves between the remanence and the coercive field that, according to Mössbauer results [6,23], can be attributed to the appearance of microstructural defects (antiphase boundaries and twin boundaries).

These results suggest that, in the course of the thermal treatments, the progressively increasing high coercivity is induced initially by fcc to $L1_0$ transformation, then by the progressive ordering of the hard magnetic phase, and finally by the appearance of structural defects at the interfaces between grains [6]. These defects (antiphase boundaries, twin boundaries, order/disorder phase boundaries) are known to act as centres that hinder the motion of domain walls [20,23,24,27], therefore implying that an increase of coercivity can be expected even after the order parameter of the hard phase S_{tet} has already reached unity, as long as the order parameter of the alloy S_{alloy} as a whole is still increasing, as in our case (compare Fig. 1(d) and [26]).

4. Discussion

The significant change of magnetic behaviour occurring after 5 min of annealing time, when the $L1_0$ phase reaches complete order ($S_{tetr} = 1$ for annealing time equal to 10 min or above [26] and Fig. 1), is clearly visible in Fig. 4, where the saturation magnetic moment is plotted as a function of the order parameter of the alloy as a whole (S_{alloy}) [26]. As all the samples are just one sample submitted to subsequent annealing steps, it is not necessary to estimate the sample volume to obtain the magnetisation, the magnetic moment being sufficient for comparing the magnetic properties at different annealing times. A two-slope behaviour is observed, with a well-marked jump between 5 and 10 min annealing time. Below 5 min, the saturation magnetic moment remains almost constant, as expected as m_s of the hard phase should be almost equal to that of the fcc phase [5,28]. The slight decrease could be due to an increase of the density of antiphase boundaries [29]. Conversely, from 10 min on, when S_{tetr} becomes equal to 1, S_{alloy} keeps on gradually increasing [26], but m_s shows a marked reduction and a decreasing trend. The appearance and progressive development of antiphase boundaries and twins, for such annealing times, is responsible for pinning of domain walls. This not only increases coercivity, as discussed above, but locally reduces the magnetic moment because of the tilting or disappearance of the anisotropy axis [25,30]. Indeed, between 60 and 120 min annealing time, with S_{alloy} remaining almost constant [26] and therefore with a microstructure that has attained its final equilibrium configuration, also m_s remains constant.

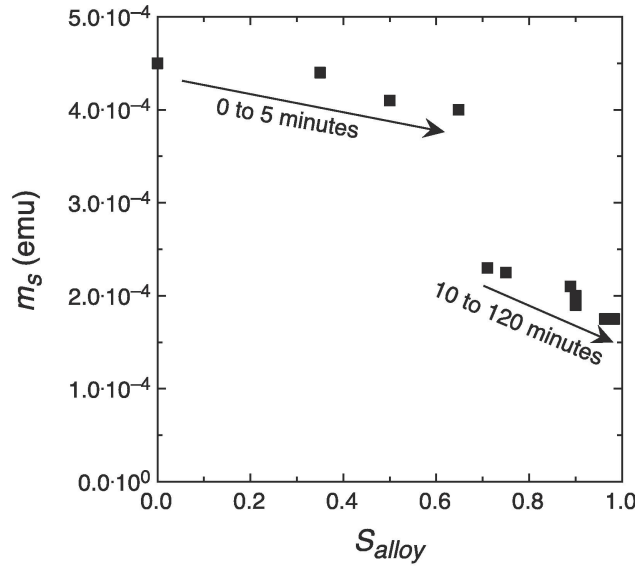


Fig. 4. Saturation magnetic moment as a function of the order parameter of the alloy for the studied annealing times.

The role of the structural defects clearly emerges also in the analysis of the remanence to saturation (m_r/m_s) ratio, that has already been quickly discussed earlier. For convenience, this ratio is reported in Fig. 5, together with the maximum energy product BH_{max} , that has the same evolution with the annealing time [31]. The second family of samples (annealing time from 1 to 5 min), is characterised by an increased m_r/m_s ratio with respect to the as-deposited sample, due to the appearance of the hard $L1_0$ phase. The maximum energy product is reached at 5 min annealing time, probably due to an optimum coupling between the hard $L1_0$ phase and the remaining soft fcc one. The third family of samples (10 through 30 min annealing time) has a reduced remanence to saturation ratio, due to the appearance of antiphase walls and twins [6] that locally give a random orientation of the anisotropy, causing a drop in the maximum energy product as well [32]. Finally, for longer annealing times, the increase of the m_r/m_s ratio can be attributed to the increase of the order parameter of the alloy S_{alloy} , associated with twin boundaries that reduce the random distribution of the uniaxial anisotropy.

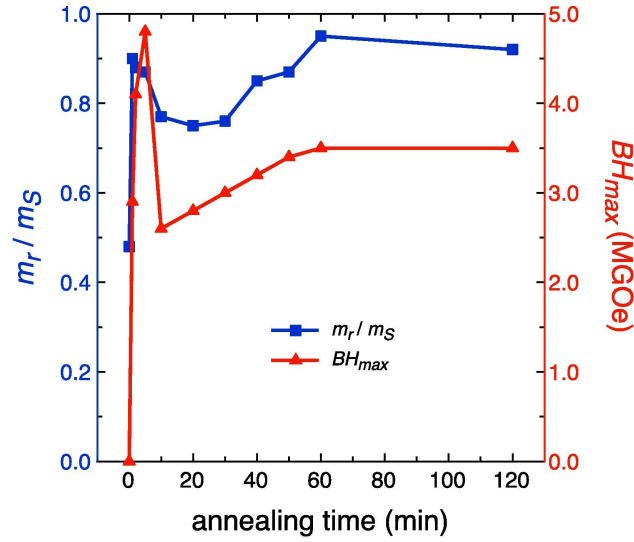


Fig. 5. Remanence ratio (blue squares) and maximum energy product (red triangles) of the Fe54Pd46 alloys as a function of the annealing time. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Representative samples of the four families have also been selected for showing their magnetic domain configuration, as imaged by magnetic force microscopy at the remanence, as illustrated in Fig. 6.

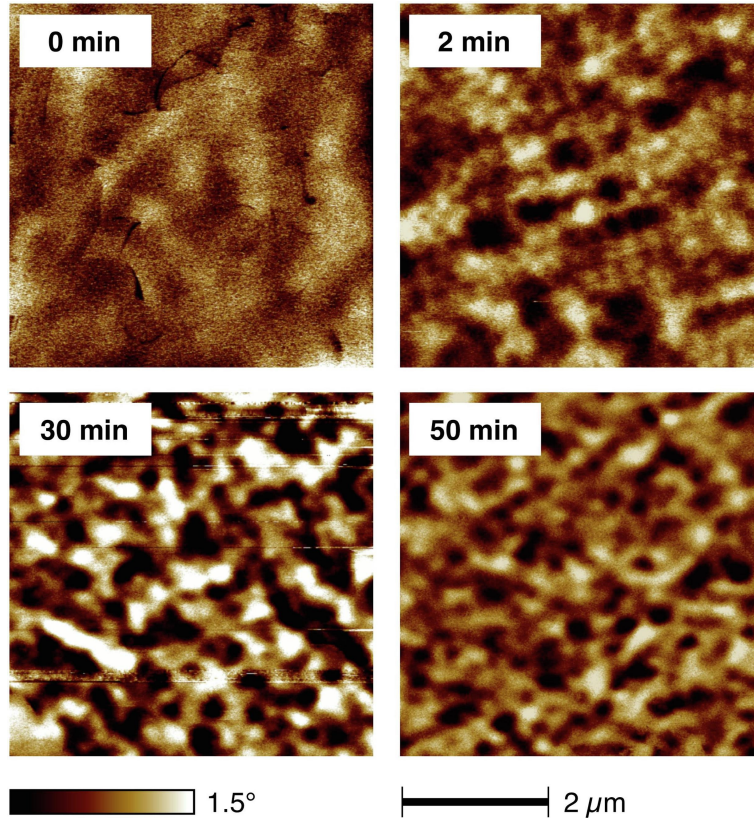


Fig. 6. Magnetic force microscopy images taken at the in-plane magnetic remanence on representative samples of Fe54Pd46 alloys annealed for selected times.

The images correspond to the same areas reported, for the same samples, in Fig. 2. The as-deposited sample is characterised by a confused and not very contrasted bright and dark configuration, typical of soft magnetic thin films with disordered microstructure whose local anisotropy slightly competes with the shape anisotropy, inducing magnetisation fluctuations off the film plane. This is compatible with the shape of the hysteresis loop (Fig. 3), that besides a small coercivity displays a shape that is not rectangular, with a certain component of moments rotation toward the applied magnetic field direction to reach saturation. The sample

annealed for 2 min, representative of the second family, is instead characterised by a MFM image displaying bright and dark regions of rather circular shape and different sizes, dispersed in areas where a neutral colour indicates a much weaker contribution of the magnetisation to the magnetic field fringing out of the sample surface. This indicates a still irregular and incomplete development of the $L1_0$ phase, in uncoupled grains. The sample annealed for 30 min, representative of the third family, is instead characterised by bright and dark regions of irregular shape covering most of the field of view [33]; in this sample the development and the ordering of the $L1_0$ phase are complete, and twins are already developing. Finally, the sample annealed for 50 min, and representative of the fourth family, has a magnetic configuration similar to the sample annealed for 30 min, as one would expect since this family only undergoes a refinement of the microstructure with progressive ordering of the alloy (S_{alloy} approaching unity [26]), but with a weaker magnetic contrast possibly due to the full development of twin boundaries and polytwins [34].

5. Conclusions

The effects on the magnetic properties of the phase transformation from disordered fcc to ordered $L1_0$ has been studied as a function of annealing time in the $\text{Fe}_{56}\text{Pd}_{44}$ alloy system in thin films form submitted to thermal treatments at 550 °C in vacuum. The detailed microstructure evolution [6] has been exploited to interpret a rich magnetic behaviour. Besides the expected increase of the coercive field, due to the development of the hard $L1_0$ phase, the non-monotonous behaviour of the remanence to saturation ratio, with the associated maximum energy product, has been explained in terms of the ordering of the $L1_0$ phase first, and of the alloy as a whole thereafter, when upon completion of the fcc to $L1_0$ phase transformation the microstructure defects (twinned domains, twin boundaries, antiphase boundaries and polytwins) acted in a complex way, by locally altering the magnetic anisotropy. This behaviour appears in the evolution of the hysteresis loops shape as a function of annealing time and in the magnetic domains patterns observed by magnetic force microscopy, that have been interpreted in the light of the detailed knowledge of the microstructure evolution and of the development of its defects.

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