Thomas Bublat

Hartmagnetische $L1_0$-FePt basierte großflächige Nanomuster mittels Nanoimprint-Lithografie

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Hartmagnetische $L1_0$-FePt basierte großflächige Nanomuster mittels Nanoimprint-Lithografie

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Summary

In our modern world advanced magnetic materials with excellent magnetic properties are getting more and more important. Major areas needed to be mentioned here are for example high performance permanent magnets for electric motors or magnetic data storage media. Further development of existing products or even new applications can only be possible with a continuous optimization of intrinsic and extrinsic material parameters of magnetic materials.

Looking at the temporal development of magnetic data storage devices, it is characterized by a continuous increase of the data storage density, what can be achieved by smaller bit sizes. For continuing this trend and realizing storage densities of 1 TBit/in² bit sizes are needed, which reach their physical limits. Since current magnetic storage media consist of a granular layer and one bit consists of a various number of grains, with the ongoing miniaturization of the bits the bit boundary is getting more and more blurred. This issue can be postponed by the usage of ever smaller grains. An alternative route to optimize the signal-to-noise ratio is the usage of bit patterned media, where each dot represents a single bit. Depending on the magnetic material used, however, a minimal grain size is given, since the magnetization will become thermally unstable if the grains are too small. This phenomenon is known as the superparamagnetic limit. Hence, for future magnetic storage media a material is needed having an extremely high crystalline anisotropy and hence circumventing this superparamagnetic limit.

Due to its very high crystalline anisotropy and its good corrosion resistance the $L1_0$-phase FePt is a promising candidate for future magnetic storage media. Based on this material even smallest particle sizes down to a radius of $r = 2$ nm are thermally stable, what would make a significant increase in the storage density possible. On the other hand, a high crystalline anisotropy will result in large writing fields, which cannot be provided by conventional writing heads. Thus, several methods are scope of current research with the aim to reduce writing

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fields. The issue of a good signal-to-noise ratio, thermal stability and writeability is known as the trilemma of magnetic data storage. Promising concepts for example are thermally assisted writing, microwave assisted writing, tilted writing or the usage of composite media consisting of a hard magnetic and a soft magnetic material. Especially for heat assisted writing the knowledge of temperature dependent magnetic properties of $L1_0$-FePt is of upmost importance.

Therefore, in the first part of this thesis the temperature dependent behavior of magnetic properties of insular $L1_0$-FePt nanostructures is investigated in the full ferromagnetic temperature range. Furthermore insular $L1_0$-FePt nanostructures can be seen as a first model system for regular arranged nanostructures and important results obtained from insular $L1_0$-FePt nanostructures can be used for investigations of regularly arranged $L1_0$-FePt nanostructures, which will be studied in the second part of this thesis. By investigating $L1_0$-FePt nanostructures and different kinds of $L1_0$-FePt based composite nanostructures the influence of an additional soft magnetic layer is analyzed.

Insular $L1_0$-FePt nanostructures

Since comprehensive temperature dependent investigations in the full ferromagnetic temperature range of hard magnetic insular $L1_0$-FePt nanostructures with out-of-plane texture are missing in the literature so far, in the first part of this thesis the magnetic material parameters of such $L1_0$-FePt nanostructures will be determined and analyzed by means of SQUID magnetometry. Furthermore dependencies of the order parameter, particle size, and morphology are included in the investigations, which are directly influenced and can be controlled by the layer thickness.

To prepare hard magnetic insular $L1_0$-FePt nanostructures with out-of-plane texture ultrathin films are deposited by magnetron co-sputtering on heated (800 °C) and one side polished MgO-substrates with a film growth rate of approximately 0.1 nm/s. The film thickness was chosen to be 3 nm, 5 nm, 7 nm,

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8 nm, 10 nm, 15 nm and 20 nm. To guarantee epitaxial film growth the samples were kept at a temperature of 800 °C for 1 h subsequently to sputtering.

The main results on insular $L1_0$-FePt nanostructures are:

1. *Morphology*
   Between thicknesses of 3 nm and 20 nm a strong dependence of the morphology and the thickness could be observed. Starting from a thickness of 3 nm the FePt film shows a clear insular growth with an average island size of 14.6 nm. With increasing thickness also the islands become bigger and differ more and more from an ideal round shape. At a layer thickness of 8 nm an average island size of 46 nm could be observed. In addition, by means of XRD measurements a dependence of the crystalline ordering and the layer thickness could be verified. For a thickness of $d = 7$ nm the ordering parameter was measured to be $S = 0.89$, whereas for thinner or thicker films reduces slightly ($S = 0.83$ for $d = 5$ nm and $S = 0.81$ for $d = 15$ nm). The reason for this was found in the total value of the lattice constants, since for a film thickness of 7 nm the lattice constant is closest to the values of bulk FePt.

2. *Hysteresis loop*
   Throughout all investigated thicknesses the magnetic hysteresis loops show a good out-of-plane texture and coercivities up to 3.97 T were measured at 300 K. Since the out-of-plane hysteresis loops are of a clear one step type, this indicates the presence of only one homogeneous phase although the fact, that the order parameter differs from the ideal value of 1. Depending on the grade of ordering for the insular films the coercivity amounts to 2.79 T for a 3 nm thick film and increases up to 3.97 T ($d = 7$ nm). For thicker films the coercivities decreases again ($\mu_0 H_C = 2.83$ T for $d = 8$ nm). When morphology changes from insular to continuous layers the coercivity drop significantly and amounts to values of 0.42 T ($d = 10$ nm) and lower.

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It is important to note that the hysteresis of the samples with various thicknesses keep their rectangular shape for all investigated temperatures ranging from 40 K up to 650 K. A continuous decrease of the coercivity and the saturation magnetization with increasing temperatures was observed.

3. *Intrinsic material parameters*

The intrinsic material parameters saturation polarization J_S, crystalline anisotropy K_1, exchange constant A, and the Curie-Temperature T_C were determined as function of the film thickness. In addition the temperature dependencies of J_S, K_1, and A were investigated. It turned out that, starting from a certain thickness the intrinsic material parameters decrease with decreasing film thicknesses. E.g. the Curie-Temperature amounts to a value of 660 K for a thickness of 8 nm and higher and decreases almost linearly for thinner films ($T_C = 585$ K for $d = 3$ nm). A very similar thickness behavior can be observed for the saturation polarization ($J_S = 1.44$ T for $d = 15$ nm and $J_S = 1.01$ T for $d = 3$ nm). The exchange constant, however, increases almost linearly in the whole thickness range ($A = 5.74$ pJ/m for $d = 15$ nm and $A = 3.62$ pJ/m for $d = 3$ nm). For the crystalline anisotropy the thickness dependence is completely different. By comparing the order parameter and the crystalline anisotropy it becomes obvious, that K_1 is directly influenced by the crystalline ordering and thus explaining the existence of a maximum of the crystalline anisotropy ($K_1 = 5.64$ MJ/m³) for a thickness of 7 nm.

4. *Magnetic reversal mechanism*

By means of the microstructural parameters α and N_{eff} the magnetic reversal mechanism has been analyzed. Due to very small values of α ($\alpha < 0.04$) for a continuous or maze-like film the coercivity field must be dominated by domain wall pinning effects. On the other hand, larger α values ($\alpha > 0.34$) indicate a nucleation hardened magnetic reversal mechanism, what is the case for the insular film.

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Regular arranged $L1_0$-FePt nanostructures

Besides the idea to enlarge the magnetic storage density by granular films having smaller grains, the usage of nanostructured hard magnetic dots is the focus of current research. In this approach each dot would represent an individual bit, hence the signal-to-noise ratio would be optimized. Nevertheless, to fabricate nanodots on a regular basis over a large area a proper nano structuring technique needs to be found. Most of the common techniques have the disadvantage, that either each dot has to be illuminated in a serial manner (e.g. electron beam lithography) and will therefore consume too much time, if a large area need to be structured, or the whole area is structured at once, but there is lack of a large range ordering (monodisperse FePt colloids). However, nanoimprint lithography can solve those issues, since by imprinting the mold into the lacquer the whole area is structured at once and the mold profile is maintained. The pattern profile can then be transferred into an underlying layer by etching.

In the second part of this thesis a process is developed to structure continuous hard magnetic FePt films in regular arrange nanodots having sizes of 40 nm, 60 nm, 80 nm, and 100 nm. This makes it possible to investigate the dot size dependence of the magnetic properties. Because the dots are additionally structured over a large area of 2 mm x 2 mm the accumulated moment of those dots is large enough to be measured by SQUID magnetometry, hence making it possible to determine magnetic properties of individual dots using macroscopic measurements. Furthermore the influence of the etching process is studied in detail in combination with AFM/MFM and TEM measurements.

The main results on regular arranged $L1_0$-FePt nanostructures are:

1. *Nanoimprint process*

   Continuous $L1_0$-FePt films with a thickness of 40 nm were structured into a regular arrangement of nanodots having a diameter of 40 nm, 60 nm, 80 nm, and 100 nm over a total area of 2 mm x 2 mm. For the imprint process a combination of IPS® and STU® imprinting has been used, for the etching ICP-

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RIE was chosen. Process parameters like imprint pressure, time, temperature, and etching time were optimized resulting in a maximum FePt dot height of 30 nm. The dot array has been verified to be very homogenous over the total area after etching and only very few dots are missing at the outer edges of the structured area. Those dots represent the worldwide first hard magnetic nanostructures, which are structures on a regular basis over a large area.

2. *As-etched nanostructures*

   Directly after structuring the coercivity significantly increases up to 1.66 T. It has been shown, that the dots are of a single domain type and have a strong out-of-plane crystalline anisotropy. Due to the etching process the dots are marked by a rough surface and the crystalline ordering is strongly disturbed near the surface. TEM investigations have proven that in the top few nm the far less hard magnetic A1-FePt phase is present, whereas the main volume of the dots consists of hard magnetic $L1_0$-FePt. This two phase system is thus representing a phase graded exchange coupled two layer nanocomposite dot. The magnetization is reversed by generating an incomplete domain wall, which is pinned at the hard/soft magnetic boundary. The coercivity of as-etched dots has a weak temperature dependence ($2.05\ \mathrm{T} < \mu_0 H_C < 1.55\ \mathrm{T}$) in a temperature range of 40 K up to 350 K.

3. *Post-annealed nanostructures*

   It could be shown, that due a subsequent annealing procedure in Ar-atmosphere the crystalline defects caused by the etching process are cured almost completely. As a consequence the whole volume consists of the hard magnetic $L1_0$-FePt phase and the coercivity increases up to 4.56 T. The dots are still single domain particles and the crystalline anisotropy is further increased. In addition the morphology changes slightly since after annealing the dots are of a very round shape and smooth surface. The coercivity of post-annealed dots shows a strong temperature dependence ($6.44\ \mathrm{T} >$

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$\mu_0 H_C > 3.79$ T) in a temperature range of 40 K up to 350 K. The magnetic reversal mechanism of post-annealed nanostructures is dominated by nucleation.

4. *Coercivity mechanism*

   By means of angular dependent measurements and determination of the microstructural parameters α and N_{eff} the magnetic reversal mechanism has been investigated and a significant difference between the as-etched state ($\alpha = 0.14$) and the post-annealed state ($\alpha = 0.80$) of the 60 nm nanostructures could be observed. As a consequence of the large α value nucleation of inverse magnetization must be origin of magnetic reversal for the post-annealed $L1_0$-FePt nanostructures. Angular dependent measurement have verified this due to a good compliance of experimental results and theoretical calculations of α_ψ.

   For the as-etched state of the 60 nm $L1_0$-FePt/A1-FePt nanostructures determination of α alone is not sufficient to ascertain the type the magnetic reversal mechanism, since in principle nucleation and pinning would be possible due to the relative low α value. However, angular dependent measurements have proven the absence of a pure nucleation or pure domain wall pinning. As a consequence of the negative N_{eff} an incomplete domain wall will be generated at the surface tips of the dots and will then move through the dot. This represents a superposition of nucleation and pinning.

5. *Dot size dependence*

   Nanostructures having a size of 40 nm, 60 nm, 80 nm, and 100 nm have been prepared on a large area to investigate the dot size dependence of the magnetic properties and the magnetic reversal mechanism. However, within this dot size range no significant change of the coercivity or microstructural parameters could be observed for both the as-etched the post-annealed nanostructures.

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Regularly arranged $L1_0$-FePt/Fe nanostructures

As reported small hard magnetic $L1_0$-FePt nanostructures can have an extremely high coercive field, which is much too high for conventional writing heads in magnetic data storage devices. Thus, it is very important to provide technologies making it possible to reduce writing field in a controlled and a reliable manner. Especially for bit patterned media composite materials could be appropriate.

Large area composite nanostructures will therefore be the subject of the last part of this thesis distinguishing between two different types of composite nanostructures. In the first approach nanostructures are produced from a single $L1_0$-FePt layer. After annealing those dots are covered with an additional soft magnetic cover layer, thus building composite nanostructures. The second type of nanostructures are directly structured from a bilayer composite consisting of $L1_0$-FePt and Fe. At both approaches the magnetic behavior is investigated. In particular the coupling between the hard and soft magnetic layers and the effects on the coercivity mechanism are analyzed.

The main results on regular arranged composite nanostructures are:

1. *Subsequently with Fe covered nanostructures*

   Composite nanostructures were prepared by covering $L1_0$-FePt nanostructures with Fe having a thickness of 1 nm, 3 nm, 5 nm, and 10 nm. Since sputtered at room temperature the Fe forms a continuous layer and hence the magnetic moment points into the Fe layer due to strong form anisotropy. For all investigated Fe layer thicknesses the soft magnetic part and the hard magnetic $L1_0$-FePt part seems to be fully decoupled.

2. *Nanostructures from $L1_0$-FePt/Fe bilayers*

   A bilayer having a $L1_0$-FePt thickness of 20 nm and a Fe thickness of 7 nm has been structured into 60 nm dots to investigate the coupling of composite nanostructures prepared from a bilayer system. It could be

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shown, that the hard magnetic part and the soft magnetic part couple almost completely and the coercivity can be effectively reduced from a value of 0.86 T to 0.46 T.

Finally one can summarize, that the temperature dependent investigations on insular $L1_0$-FePt covering the whole ferromagnetic temperature range contribute primarily important results on the temperature dependence of magnetic properties for thermal assisted writing. Furthermore insular $L1_0$-FePt nanostructures can be taken as first model systems for bit patterned media.

Within the framework of this thesis the worldwide first hard magnetic nanostructures having a size between 40 nm and 100 nm are structured by nanoimprint lithography over a total area of 2 x 2 mm². This allows a determination of magnetic properties of individual dots by macroscopic measurements. It has been found out, that using the developed nanostructuring process continuous $L1_0$-FePt can be directly turned into $L1_0$-FePt/A1-FePt composite nanostructures having a reduced coercivity due to A1-FePt content. Simultaneously the thermal stability is given by the $L1_0$-FePt portion. Those represent first prototype nanostructures for future bit patterned media for magnetic data storage.
It could be shown, that $L1_0$-FePt/A1-FePt composite nanostructures can be transformed into $L1_0$-FePt nanostructures by a subsequent annealing procedure having a very large coercive field. In combination with thermal or microwave assisted writing those nanostructures might be used for magnetic data storage.
Although the nanopatterns produced in this thesis do not exceed the storage destiny of modern hard discs, with small modification the developed nanostructuring process it should be possible to produce smaller nanostructures having a structure size of less than 20 nm, hence in future very interesting research results are to be expected.

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