Tailoring particle arrays by isotropic plasma etching: an approach towards percolated perpendicular media

2009 Nanotechnology 20 105304

(http://iopscience.iop.org/0957-4484/20/10/105304)

Nanotechnology **20** (2009) 105304 (5pp) doi:10.1088/0957-4484/20/10/105304

Tailoring particle arrays by isotropic plasma etching: an approach towards percolated perpendicular media

Christoph Brombacher[1,5], Marc Saitner[2,6], Christian Pfahler[2], Alfred Plettl[2], Paul Ziemann[2], Denys Makarov[3], Daniel Assmann[3], Martin H Siekman[4], Leon Abelmann[4] and Manfred Albrecht[1]

[1] Institute of Physics, Chemnitz University of Technology, D-09107 Chemnitz, Germany
[2] Institute of Solid State Physics, University of Ulm, D-89069 Ulm, Germany
[3] Department of Physics, University of Konstanz, D-78457 Konstanz, Germany
[4] MESA+ Research Institute, University of Twente, 7500 AE Enschede, The Netherlands

E-mail: christoph.brombacher@physik.tu-chemnitz.de

Received 4 December 2008, in final form 13 January 2009
Published 16 February 2009
Online at stacks.iop.org/Nano/20/105304

Abstract
Plasma etching of densely packed arrays of polystyrene particles leads to arrays of spherical nanostructures with adjustable diameters while keeping the periodicity fixed. A linear dependence between diameter of the particles and etching time was observed for particles down to sizes of sub-50 nm. Subsequent deposition of Co/Pt multilayers with perpendicular magnetic anisotropy onto these patterns leads to an exchange-decoupled, single-domain magnetic nanostructure array surrounded by a continuous magnetic film. The magnetic reversal characteristic of the film–particle system is dominated by domain nucleation and domain wall pinning at the particle locations, creating a percolated perpendicular media system.

M This article features online multimedia enhancements

The storage density in hard disk drives is rapidly increasing by downsizing in particular the magnetic grains in the storage layer. However, the superparamagnetic limit is a severe barrier for a further decrease of the grain size without requiring higher magnetic write fields and much effort is put into the search for different concepts to circumnavigate this limitation, such as the 'patterned media' [1–3] or 'percolated perpendicular media' [4–8] approach, which can make storage densities beyond 1 Tbit in^{-2} possible. To establish these approaches in magnetic data storage devices, new developments in nanotechnology are required. Several ways to create regular nanostructure arrays do exist [9–16] and combining patterned structures with the deposition of magnetic film systems with high anisotropy seems to be a promising way to achieve these new media [1, 17–21]. In this regard, a magnetic cap structure exhibiting a perpendicular anisotropy, with each cap being magnetically exchange-isolated and single-domain can be obtained by the deposition of Co/Pt or Co/Pd multilayers onto spherical particle arrays [22–24]. Alternatively, the concept of percolated perpendicular media in which densely distributed pinning sites lead to smooth transition boundaries might be realized by the deposition of a magnetic film with a perpendicular easy axis of magnetization onto non-close-packed particle arrays. In this case, each non-magnetic particle itself acts as a defect structure providing a site for nucleation and domain wall pinning.

In this paper, the method of isotropic reactive ion etching (RIE) is employed, applying various etching times to obtain particle assemblies with smaller diameters while keeping the periodicity unchanged followed by a Co/Pt multilayer deposition. The influence of physically well-separated polystyrene (PS) particles on the magnetic reversal characteristics of the surrounding film has been investigated down to particle diameters of sub-50 nm.

Common colloidal lithographic nanopatterning is done in general with anisotropic techniques like ion polishing [25, 26] or RIE in parallel plate reactors [27, 28]. The etching rates

[5] Author to whom any correspondence should be addressed.
[6] New address: Institute for Materials Research (IMO), University of Hasselt, B-3590 Diepenbeek, Belgium.

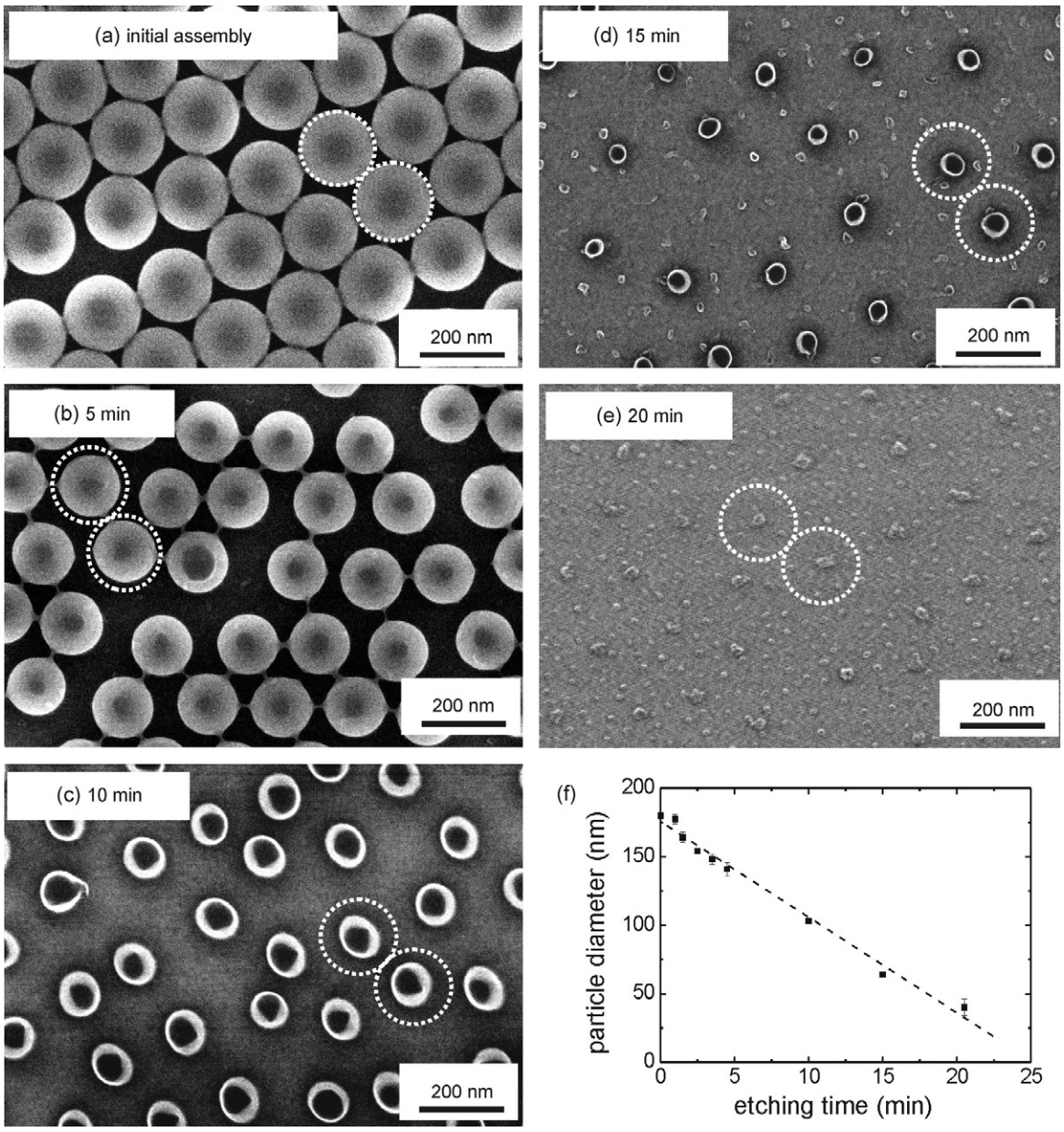

Figure 1. SEM images of (a) a densely packed monolayer of 180 nm PS spheres and (b)–(e) particle arrays that have been exposed to RIE applying different etching times. (f) Relationship between particle size and etching time.

are therefore rather high, typically of the order of 1 nm s^{-1}. Accordingly, the particles lose their spherical shape, their smooth surface and also the substrate surface is roughened. Thus, the application of these techniques is limited to particle sizes of some 100 nm or larger. For size reduction of particles with good conservation of the spherical shape, especially of the upper half of the particle, we developed an isotropic etching process using a commercial RIE–ICP system (Oxford Plasma Technology Type 80 Plus) with independent RIE and ICP (inductively coupled plasma) sources. Starting the plasma with the RIE source (at 25 W, with 100 W ICP and 50 sccm oxygen) and switching gradually within 30 s to an almost perfect isotropic ICP mode (with 60 W ICP, and for high stability of the plasma with 1 W RIE and 15 sccm oxygen) at a constant pressure of 55 mTorr and room temperature yields etching rates of 5–8 nm min^{-1}, depending on the PS particles used. During the first 2–3 s, the DC bias amounts to about −79 and −7 V under stable isotropic conditions. Figure 1(a) shows a scanning electron microscopy (SEM, Hitachi S-5200) image of the initial 180 nm particle assembly which was prepared on a naturally oxidized Si(001) substrate by a slow evaporation process under ambient conditions [29]. The evolution of the particle assembly with increasing etching time is demonstrated by the series of SEM images of figures 1(b)–(e). A linear dependence between the particle diameter and the etching time down to 40 nm after an etching time of 20 min has been observed, as summarized in figure 1(f). The specific particle morphology has been investigated by high resolution SEM, revealing a good preservation of the spherical shape of the upper cap of the particle down to about half the starting

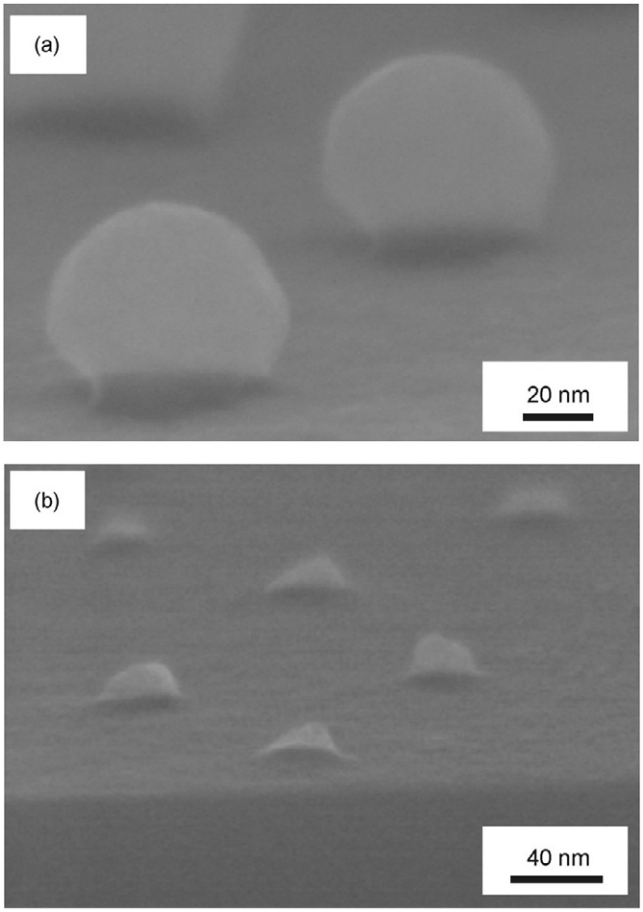

Figure 2. Cross-section high resolution SEM images after an etching time of (a) 15 min and (b) 20 min.

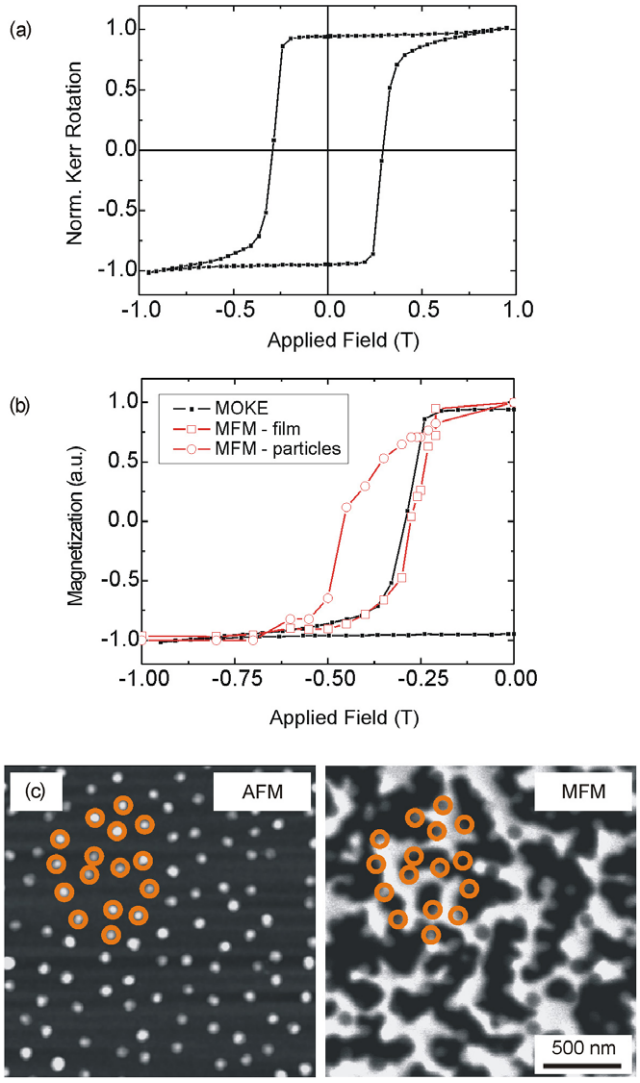

Figure 3. (a) Polar MOKE hysteresis loop of Co/Pt multilayers deposited onto an etched particle array with particle diameters of 40 nm (20 min etching time) and (b) plot of the magnetization versus applied field of film and particle caps obtained from in-field MFM in comparison with the measured P-MOKE loop. (c) Corresponding AFM and MFM image in remanence after demagnetizing the sample. Some particle locations are indicated by circles.

diameter and a flattening for longer etching times as shown in figure 2. The roughness of the particle surface and that of the surrounding substrate remains very small. However, it should be noted that there is additional material in between the particles present, probably due to impurities in the colloidal suspension. This leads to an additional surface roughness of the surrounding substrate before the film deposition.

All templates were introduced into a molecular beam epitaxy chamber with a base pressure of 2×10^{-10} mbar. A film stack of Pt(5 nm)/[Co(0.3 nm)/Pt(0.8 nm)] $\times$ 12/Pt(1 nm) with an effective perpendicular magnetic anisotropy $K_{\mathrm{eff}} = 0.3$ MJ m^{-3} [30] was deposited by alternating evaporation from two independent e-beam sources. All samples were prepared at room temperature in one single evaporation process to ensure identical growth conditions.

Polar magneto-optical Kerr effect (P-MOKE) magnetometry was utilized to investigate the magnetic properties. In this set-up, a focused laser beam with a spot diameter of about 100 μm and a wavelength of 670 nm was used. The following experimental results are focused on the sample exhibiting the smallest particle diameter of 40 nm. Note that, for these etched templates, the relative ratio of the magnetic contributions to the MOKE signal from the surrounding film and the particle caps is determined by the particle filling factor. For the smallest particle sizes, the reduced filling factor and the stronger scattering of the reflected laser beam off the particle caps reduces this relative ratio substantially, resulting in an almost negligible cap contribution to the MOKE signal. A hysteresis loop of the Co/Pt multilayer deposited onto an etched template with a particle diameter of 40 nm is shown in figure 3(a). A sharp switching behavior of the continuous film with a coercivity of $H_{\mathrm{c}} = 290$ mT and a remanence of 1 (normalized to the saturation value) is observed, indicating a reversal process dominated by the nucleation and subsequent propagation of domain walls [23].

In order to shed more light onto this reversal behavior, atomic and magnetic force microscopy (AFM/MFM) investigations were carried out in a MultiMode SPM from Digital Instruments and a home-built in-field MFM. Figure 3(c) shows both the topography (left) and the corresponding magnetic

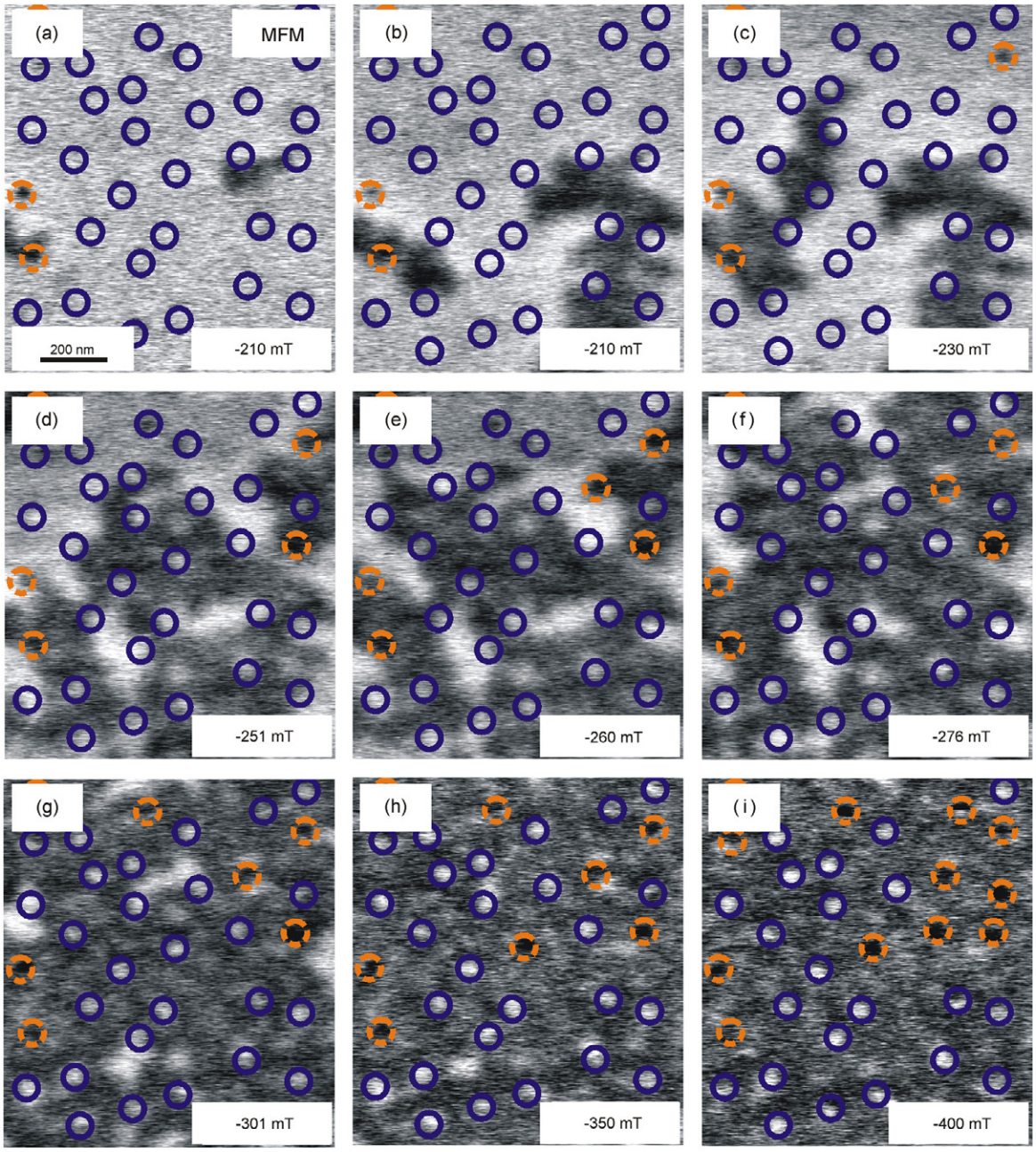

Figure 4. In-field MFM images taken after saturating the sample at +1.6 T and applying reverse fields from (a) −210 mT to (i) −400 mT. Reversed caps are marked by dashed circles while continuous circles indicate caps that have not switched. Note that (a) and (b) are the first and second scans of the same area applying a constant field, indicating a thermally activated reversal process or tip-induced switching.

contrast (right) in remanence after demagnetizing the sample in a perpendicular field. The image reveals, that all cap structures are in a single-domain state and strongly exchange-decoupled from the surrounding film exhibiting magnetic domain structures in the range of 200 nm. It is apparent that the domain wall boundaries are preferentially located along the particle locations, which is energetically the most favorable state. The switching behavior was further monitored with in-field MFM [31], operated in constant-height mode, shown in figures 4(a)–(i) and in the movie (available at stacks.iop.org/Nano/20/105304). All images are taken in-field. By reducing the scan height, topographic cross-talk was increased and particle positions could be determined. Correct particle positions are marked by dashed orange (particle cap has switched) and continuous purple (cap has not switched) circles in the magnetic images. After saturating the sample at +1.6 T, domains start to nucleate at reverse fields of −210 mT (figure 4(a)) next to the particle locations. Note that also at this rather low field a few magnetic caps start already reversing their magnetization orientation. The further switching of the film is then dominated by domain wall propagation, but it can be observed that the domain wall propagation is preferentially stopped at particle locations. This is a clear indication of domain wall pinning, which can be used to limit the domain

size when exposed to a spatially localized magnetic field as provided by a magnetic recording head. While there is no strong indication of exchange interaction between particle caps and surrounding film, magnetostatic interactions do play a role, leading to a broadening of the switching field distribution (SFD) of the magnetic caps and an increased coercivity. The latter is based on the fact that caps which remained in their initial magnetization state after the surrounding film has switched are further stabilized by demagnetization fields of the surrounding film system. However, the dominant contribution to the SFD is the specific nucleation field of the dots, which strongly depends on the magnetic film properties of the deposited film, such as magnetic anisotropy, and on the size and geometrical shape of the caps. From the in-field MFM images, hysteresis loops of the particle caps and the surrounding film were constructed by simply evaluating the number of reversed caps and by measuring the areas of black and white contrast as a function of applied field, respectively. These results are included in figure 3(b), revealing a coercivity of 450 mT and a rather broad SFD of about 500 mT for the particle cap array. The extracted hysteresis loop of the film shows rather good agreement with the hysteresis loop obtained by P-MOKE, confirming the assumption of a negligible contribution of the particle caps to the MOKE signal.

In conclusion, carefully controlled RIE etching of densely packed PS particle assemblies leads to well-separated spherical nanostructures. A linear dependence of the particle size with etching time has been observed down to particle sizes of 40 nm. These particle arrays have been used as templates for the deposition of Co/Pt multilayers. Such a film/particle system allows tailoring the reversal behavior of the continuous magnetic film, where domains start to nucleate at particle locations and propagate until the domain walls are pinned preferentially at particle-induced defects. This behavior can be used to limit the domain size when exposed to a spatially localized magnetic field as provided by a magnetic recording head. Considering that this approach can be extended towards particle arrays with smaller periodicity to increase the pinning site density and by using hard magnetic alloys to ensure sufficient domain wall pinning for smaller defect structures, a route to realize the concept of 'percolated perpendicular media' is provided.

Acknowledgments

The authors thank F Enderle and P Walther (University of Ulm) for their assistance in plasma etching and in SEM measurements. The financial support provided by the Landesstiftung Baden-Württemberg is gratefully acknowledged.

References

[1] Terris B D and Thomson T 2005 *J. Phys. D: Appl. Phys.* **38** R199
[2] Moser A, Takano K, Margulies D T, Albrecht M, Sonobe Y, Ikeda Y, Sun S and Fullerton E E 2002 *J. Phys. D: Appl. Phys.* **35** R157
[3] Albrecht M, Moser A, Rettner C T, Anders S, Thomson T and Terris B D 2002 *Appl. Phys. Lett.* **80** 3409
[4] Zhu J-G and Tang Y 2006 *J. Appl. Phys.* **99** 08Q903
[5] Suess D, Fidler J, Porath K, Schrefl T and Weller D 2006 *J. Appl. Phys.* **99** 08G905
[6] Rahman M T, Lai C-H, Vokoun D and Shams N N 2007 *IEEE Trans. Magn.* **43** 2133
[7] Laughlin D E, Peng Y, Qin Y–L, Lin M and Zhu J-G 2007 *IEEE Trans. Magn.* **43** 693
[8] Makarov D, Bermúdez-Ureña E, Schmidt O G, Liscio F, Maret M, Brombacher C, Schulze S, Hietschold M and Albrecht M 2008 *Appl. Phys. Lett.* **93** 153112
[9] Stewart M E, Motala M J, Yao J, Thompson L B and Nuzzo R G 2007 *Proc. Inst. Mech. Eng.* N **220** 81
[10] Zhu M, Li Y, Meng T, Zhan P, Sun J, Wu J, Wang Z, Zhu S and Ming N 2006 *Langmuir* **22** 7248
[11] Cheng J Y, Ross C A, Thomas E L, Smith H I and Vancso G J 2002 *Appl. Phys. Lett.* **81** 3657
[12] Facsko S, Dekorsy T, Koerdt C, Trappe C, Kurz H, Vogt A and Hartnagel H L 1999 *Science* **285** 1551
[13] Weekes S M, Ogrin F Y, Murray W A and Keatley P S 2007 *Langmuir* **23** 1057
[14] Aranda P and Garcia J M 2002 *J. Magn. Magn. Mater.* **249** 214
[15] Sun S, Murray C B, Weller D, Folks L and Moser A 2000 *Science* **287** 1989
[16] Nielsch K, Wehrspon R B, Barthel J, Kirschner J, Fischer S F, Kronmüller H, Schweinböck T, Weiss D and Gösele U 2002 *J. Magn. Magn. Mater.* **249** 234
[17] Lodder J C 2004 *J. Magn. Magn. Mater.* **272–276** 1692
[18] Terris B D, Albrecht M, Hu G, Thomson T and Rettner C T 2005 *IEEE Trans. Magn.* **41** 2822
[19] Makarov D, Baraban L, Guhr I L, Boneberg J, Schift H, Gobrecht J, Schatz G, Leiderer P and Albrecht M 2007 *Appl. Phys. Lett.* **90** 093117
[20] Kim C, Loedding T, Jang S, Zeng H, Li Z, Sui Y and Sellmyer D J 2007 *Appl. Phys. Lett.* **91** 172508
[21] Wang T *et al* 2008 *Nanotechnology* **19** 455703
[22] Albrecht M, Hu G, Guhr I L, Ulbrich T C, Boneberg J, Leiderer P and Schatz G 2005 *Nat. Mater.* **4** 203
[23] Ulbrich T C, Makarov D, Hu G, Guhr I L, Suess D, Schrefl T and Albrecht M 2006 *Phys. Rev. Lett.* **96** 077202
[24] Guhr I L, Hellwig O, Brombacher C and Albrecht M 2007 *Phys. Rev.* B **76** 064434
[25] Zheng Y B, Wang S J, Huan A C H and Wang Y H 2006 *J. Appl. Phys.* **99** 034308
[26] Yan L, Wang K, Wu J and Ye L 2006 *J. Phys. Chem.* B **110** 11241
[27] Haginoya C, Ishibashi M and Koike K 1997 *Appl. Phys. Lett.* **71** 2934
[28] Huang Z, Fang H and Zhu J 2007 *Adv. Mater.* **19** 744
[29] Micheletto R, Fukuda H and Ohtsu M 1995 *Langmuir* **11** 3333
[30] Ulbrich T C, Assmann D and Albrecht M 2008 *J. Appl. Phys.* **104** 084311
[31] Murillo Vallejo R, Siekman M H, Bolhuis T, Abelmann L and Lodder J C 2007 *Microsyst. Technol.* **13** 177