High-yield fabrication of perpendicularly magnetised synthetic antiferromagnetic nanodiscs

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ABSTRACT

Synthetic antiferromagnetic (SAF) particles with perpendicular anisotropy display a number of desirable characteristics for applications in biological and other fluid environments. We present an efficient and effective method for the patterning of ultrathin Ruderman-Kittel-Kasuya-Yoshida coupled, perpendicularly magnetised SAFs using a combination of nanosphere lithography and ion milling. A Ge sacrificial layer is utilised, which provides a clean and simple lift-off process, as well as maintaining the key magnetic properties that are beneficial to target applications. We demonstrate that the method is capable of producing a particularly high yield of well-defined, thin film based nanoparticles.

KEYWORDS

nanodiscs, synthetic antiferromagnets, perpendicular anisotropy, sacrificial layers, nanofabrication

1 Introduction

Magnetic particles are widely used in a variety of biomedical applications, including cancer therapy, drug delivery and as contrast agents [1, 2]. Characteristics that are highly beneficial—and as such formulate the “ideal particle” for many biological or liquid applications—consist of a variable and high saturation magnetisation, sharp and tunable switching for efficient access to a desired saturated state, an easy axis of magnetisation with a high anisotropy to allow the efficient transduction of torque under applied field, and a zero remanence state with a low susceptibility around zero field preventing agglomeration [3]. Superparamagnetic nanoparticles (SPNPs) have so far been the major candidates in biological applications, particularly in hyperthermia for cancer therapy, however their simple magnetic properties come with limitations. Despite their zero remanence state, they exhibit considerable coercivity at higher field sweep rates. They are also prone to agglomeration via surface interactions, due to their colloidal state, without significant engineering [4–7]. Furthermore, their magnetic properties do not allow any tunability in switching field and an increase in moment can lead to a change of magnetic state, leading to ferromagnetic particles. Perpendicularly magnetised (PM) synthetic antiferromagnets (SAFs) have been shown to overcome the limitations of SPNPs and have all of the desirable properties described for an ideal particle in biological applications, offering much finer control over activation and assembly [3]. They have been demonstrated as strong candidates for the magnetomechanical destruction of cancer cells [8] and their self-assembly behaviour in liquid could be interesting in areas such as microfluidics, biosensing or soft robotics [9]. Additionally, high-aspect ratio particles, such as discs, have been shown to promote cellular endocytosis [10, 11], which is highly advantageous for in vitro applications.

So far, it has been challenging to produce a high-yield fabrication process for thin film based particles, such as SAFs, with sub-micron dimensions. Optical lithography, though a powerful and efficient method for patterning, is diffraction limited in the sub-micron regime. Furthermore, the previously used method of growing thin film stacks on top of photoresist pillars created magnetic microdiscs with significant shape defects that severely limited their aspect ratio [3]. The prevailing method, which made the production of film based nanoparticles possible, is hole-mask colloidal lithography (HMCL) [12, 13]. This method includes a multi-stage deposition and etching process, and does not lead to the production of a competitive particle yield. An alternative method for the production of such particles is nanoimprint lithography [14, 15], which has previously been used to produce in-plane SAF nanodiscs [16, 17]. However, this method suffers from similar constraints as HMCL, including the fact that deposition of a PM thin film into a nanoscale hole may have significantly negative effects on both magnetic properties and particle shape, which is due to shadowing effects during physical vapour deposition.

Here we detail an updated version of the cost and time-effective fabrication method of nanosphere lithography with ion milling [18], and make use of a Ge sacrificial layer [15], to demonstrate more optimal patterning and production of SAF nanodiscs. The method uses a streamlined protocol, with the use of polystyrene (PS) beads as a direct lithography mask, allowing access to the nanoscale. Particle yield is increased by spin coating the PS beads [19], rather than the dropcast application used in the HMCL method [13]. Additionally, the spin coating method is commercially viable, easily scalable and simple to tune for particle density. By starting with a continuous thin film and using ion milling to form the discs, we achieve excellent pattern transfer of the two-dimensional (2D) projection of the PS beads’ spherical shape onto the film.
Finally, the choice of Ge as a sacrificial layer ensures we conserve our carefully engineered magnetic properties, while facilitating ease of lift-off. These well-defined, magnetic nanodiscs are better optimised for biological applications, particularly in vitro therapies, such as the magneto-mechanical destruction of cancer cells.

2 Results and discussion

2.1 Thin films and sacrificial layers

The typical motif of a PM SAF multilayer thin film is Ta/Pt/CoFeB/Pt/Ru/Pt/CoFeB/Pt, as shown in Fig. 1(a). The easy axis response of such a film is shown in the magneto-optical Kerr effect (MOKE) loop in Fig. 1(b), with the arrows representing the magnetisation direction of the CoFeB layers. The PM state of the system is created by spin-orbit interactions between the Pt and CoFeB [20]. This state has been demonstrated as key to effective transduction of torque [8], which is highly beneficial in applications that involve mechanical actuation. The antiparallel state at remanence and low magnetic fields is induced by Ruderman– Kittel– Kasuya–Yoshida (RKKY) coupling [22–24], provided by a Ru interlayer with a thickness that lies at an antiferromagnetic coupling peak [25]. This produces a zero remanence state and low susceptibility, which prevents particle agglomeration [3, 26, 27]. The hysteresis loop in Fig. 1(b) has an apparent non-zero magnetisation due to the depth dependence of the MOKE signal. The Pt interlayers either side of the Ru not only stabilise the PM state [21], but also yield a means of tuning the sharp switch to the saturated parallel state [28]. This allows a film to be tailored to a specific application [3].

As the magnetic state of our PM SAF films is governed by interfacial interactions, the RKKY coupling and spin-orbit coupling, it is very sensitive to changes in growth conditions. Hence, the choice of sacrificial layer in a thin film based particle fabrication method must be made based on two key criteria: ease of removal, and therefore the ability to release particles into solution, and the effect of the underlayer on the magnetic properties of a thin film grown on top. Two key features to conserve, which we noted as desirable for magnetic particles to be used in biological applications, are the sharp magnetic transitions and zero remanence state.

Conventionally, photoresist is implemented as a sacrificial layer as it provides simplicity in both application and removal. However, exposing photoresist to ion milling commonly causes it to crosslink, making it significantly challenging to dissolve. Thus, Ge and Al were chosen as suitable candidates: They are amenable to the same deposition methods as the thin film, therefore simplifying the process requirements. Additionally, ease of lift-off is not compromised, as they readily dissolve in hydrogen peroxide (Ge) and basic solvents (Al), thus eliminating the use of the potentially toxic, organic solvents that are required for crosslinked photoresist. Here, Ge has an advantage over Al as a sacrificial layer, as hydrogen peroxide naturally degrades to water: This eliminates extra washing steps of lifted off particles, limiting the loss of yield.

To investigate the effect that each underlayer has on the hysteresis of PM CoFeB, sputtered magnetic stacks were grown on evaporated Ge, sputtered Al, and on Si to compare. PM SAF stacks were grown in the form Ta(2)/Pt(4)/CoFeB(0.9)/Pt(tθ)/Ru(0.9)/Pt(tθ)/CoFeB(0.9)/Pt(4)/Ta(2) (thicknesses in nm). Pt thickness, tθ, was varied from 0.41–0.69 nm to create a series of SAFs with a range of coupling strengths [21, 28]. Single layers of 0.9 nm PM CoFeB were also grown on each substrate to further analyse the effect of the underlayers on the magnetic transitions, the data for these samples is presented in Fig. S2 in the Electronic Supplementary Material (ESM).

2.2 Effect of underlayer on SAF hysteresis

Figures 2(a)–2(c) show the easy axis MOKE loops of the PM SAF series. We see a clear, drastic effect on the behaviour of the films grown on Al. The sharpness of the transitions is completely lost and the coercivity has increased. This leads to a loss of the zero remanence state in the more weakly coupled films. In comparison, the Ge underlayer has a much smaller effect on the hysteresis loops. We observe a small decrease in saturation field across the series, however, there is a relatively small change in coercivity and the sharpness of the transitions is conserved. The MOKE data indicates that Ge is a good option for a sacrificial layer, as it appears to ensure the robustness of magnetic properties and also maintains the access to a large range of coupled films with zero remanence and different switching fields.

Domain imaging of the thin films, using polar Kerr microscopy, reveals the nucleation and propagation dynamics that drive the MOKE loop transitions of the PM SAFs. The significantly sloped transitions seen in the loops of the samples grown on Al underlayers are the result of nucleation dominant switching, where the domain formations are too small to observe with the polar MOKE imaging. Conversely, the sharp transitions seen for the films grown on Si and Ge translate into clear domain images with fast moving domain walls. Figures 2(d) and 2(e) show domain images of the tθ = 0.5 nm SAF, on both Si and Ge, during the antiparallel to parallel (AP-P) transition. These images show a number of nucleation sites, creating small domains that merge together. This is consistent with previous findings on the domain dynamics of coupled CoFeB: The AP-P transition involves a higher density of nucleation sites for more highly coupled samples [29]. We observe rapid domain wall propagation, which results in the sharp transitions in the corresponding MOKE loops.

Atomic force microscopy (AFM) measurements offer further explanation of the effects that the sacrificial layers have on the magnetic stacks. Figure 3(a) shows that the roughness of Al is considerably higher than that of Si or Ge. Additionally, when we look at the radial point spectral density function (PSDF) profiles in Fig. 3(b), there is a significant magnitude of roughness in the Al sample in the 10–20 nm range, which is the length-scale associated with domain wall dynamics in PM thin films [30]. This leads to a significant effect on the nucleation and pinning of domain walls during magnetic reversal, and hence more sloped transitions in the hysteresis loops. Ge does not exhibit significant roughness on the same length-scale, with values comparable to that of Si, therefore it has a much smaller effect on the hysteresis loops of these thin films. These findings

Figure 1  (a) A schematic of the typical PM SAF thin film multilayer stack. (b) An easy axis MOKE hysteresis loop of a PM SAF thin film.
Figure 3  (a) AFM topography images of the substrates, Si/Ge/Al, and of the top Ta layer of a SAF bilayer ($t_{Pt} = 0.69$ nm) grown on top of each substrate. The root mean squared roughness ($R_{rms}$) is noted below each image. (b) The radial PSDF profiles taken from each of the substrate scans in (a), where $r$ is radius and $W_r$ is the radial PSDF, representing roughness at different length-scales.

support Ge as a suitable material for the sacrificial layer in the production of thin film based nanoparticles.

2.3 Fabrication of nanodisks

A schematic of the nanodisc fabrication process is displayed in Fig. 4. Corresponding scanning electronic microscopy (SEM) images of three of the key stages are included in Fig. S3 in the ESM. The process consists of four main stages: material deposition of the sacrificial layer, SAF stack and a capping layer; nanosphere lithography [19] of PS beads to form a mask; ion milling to pattern the thin film into discs; and finally, liquid etches to lift the magnetic nanodiscs into solution. First, (1) Ge (~ 50 nm) is evaporated onto a Si substrate, forming a sacrificial layer that will later allow the nanodiscs to be lifted into solution. (2) The PM SAF stack is sputtered on top, in the form Ta/Pt/CoFeB/Pt/Ru/Pt/CoFeB/Pt/Ta. (3) A capping layer of Al, deposited by either sputtering or evaporation, is added. This promotes the adhesion of PS beads and also provides a secondary sacrificial layer, which aids the removal of material that is redeposited during ion milling. (4) PS beads (Polybead® microspheres, diameter of 100 nm–1 μm), which have been diluted in deionised water as necessary (see Table 1), are spin coated across the Al surface to create a dense monolayer. (5) Oxygen plasma ashing is used to partially shrink the PS...
beads. The timing of the ashing is carefully tuned to minimise the size reduction and prevent bead deformation, while creating sufficient spacing between beads for subsequent film patterning into individual discs (see Table 1). (6) The PS beads are used as a lithography mask during the Ar ion milling of the full thin film stack, transferring the 2D projection of the beads onto the film beneath. (7) The PS beads are removed by sonication in water. (8) The sample is briefly soaked (~ 30 s) in photoresist developer (a tetramethylammonium hydroxide based solvent), which etches away the Al cap and leaves behind just the SAF discs on the substrate. Finally, (9) the sample is immersed in a 35% H2O2 solution for at least 10 min, which etches away the Ge sacrificial layer. It is then submerged in water, diluting away the H2O2, and sonicated to lift-off the discs into solution. This process leaves behind a clean, reusable Si wafer, which is highly beneficial from the perspectives of reducing production costs or limiting consumption of resources.

### 2.4 Characterisation of nanodiscs

This fabrication technique has so far been demonstrated using PS beads of diameters between 100 nm and 1 μm, as shown in Fig. 5. In these images we see that the use of plasma ashing, to separate out the PS beads prior to milling, produces individual discs with diameters slightly under that of the original beads. It is noted that a small point is observed at the centre of some of the nanodiscs. This small feature originates from the fabrication process, as detailed in Fig. S4 in the ESM.

Yield calculations show percentages of coverage by area that are significantly higher than those previously reported, for all disc sizes: with the 100 nm beads we achieved 31% coverage (~ 80 × 10^6 discs/mm^2), with 200 nm beads 41% coverage (~ 18 × 10^6 discs/mm^2), with 500 nm beads 55% coverage (~ 4 × 10^6 discs/mm^2) and with 1 μm beads 50% coverage (~ 8 × 10^5 discs/mm^2). For comparison, previous HMCL of ~ 100 nm discs achieved around 10% coverage, yielding just a third of the number produced with this new method [13]. Furthermore, the optical lithography based, photoresist pillar method for making 2 μm discs gives ~ 30% coverage. Therefore, our nanosphere lithography is highly competitive with the traditional optical lithography technique in coverage. As our sacrificial layer is amenable to the same deposition methods as our magnetic stack, there is additional scope for a three-dimensional (3D) increase in particle density through the insertion of Ge buffers between multiple SAF layers.

500 nm discs fabricated on Si and Ge were compared. The thin film stack grown for patterning is described by: Ta(2)/Pt(2)/CoFeB(0.9)/Pt(0.3)/Ru(0.9)/Pt(0.3)/CoFeB(0.9)/Pt(2)/Ta(2) (thicknesses in nm). MOKE loops of each size of particle, taken before lift-off, are displayed alongside each of the SEM images in Fig. 5. There is a decrease in coupling and an increase in coercivity for all of the disc samples compared with an equivalent thin film, however, there is no significant difference in these parameters between the different disc sizes.

To test the robustness of the new fabrication method, a set of 1 μm discs (see Fig. 6(a)) were created from a more complex thin film of the form: Ta(2)/Pt(t)/CoFeB(0.9)/Pt(t)/Ru(0.9)/Pt(t)/CoFeB(0.9)/Pt(2)/Ta(2) (thicknesses in nm) where t = 0.45 and 0.3 nm. Figures 6(b)–6(d) present vibrating sample magnetometry (VSM) measurements of the original film, and the discs before and after lift-off into liquid. The distinct series of magnetic transitions is maintained from the thin film into the discs on chip. Additionally, we can still distinguish between the independent switches in the liquid response. The ability to resolve the separate transitions after both patterning and lift-off strongly supports the use of the

### Table 1 Dilution (beads:water) and plasma ashing time criteria for the PS beads of different diameters

| Diameter (nm) | 100  | 200  | 500  | 1,000 |
|---------------|------|------|------|-------|
| Dilution      | 2:1  | 1:1  | 1:0  | 1:0   |
| Ash time (s)  | 30   | 60   | 240  | 300   |

![Figure 5](image-url) SEM images of SAF discs made with PS beads of diameters (a) 100 nm, (b) 200 nm, (c) 500 nm and (d) 1 μm. All scale bars are 1 μm. Alongside are major (black) and minor (red) MOKE loops taken of discs of each size.

![Figure 6](image-url) (a) An SEM image of 1 μm discs patterned from a double SAF thin film with a 1 μm scale bar. VSM measurements of (b) the film, and of the discs (c) before and (d) after lift-off into water.
new fabrication method for making particles, including for more complex magnetic systems. Furthermore, the particle behaviours here show that we can maintain access to multiple switches in a single sample, and indicate that this would also be possible for a particle mixture, which could be beneficial in a variety of applications such as microfluidic devices and cell sorting.

3 Conclusions

We have presented an efficient and effective method for the fabrication of thin film based nanodiscs. A Ge sacrificial layer has been shown to ensure the robustness of a PM SAF film, without compromising ease of lift-off. PS beads have been used in the updated combination of nanosphere lithography with ion milling, which is capable of producing a particularly high yield of high-aspect ratio nanodiscs. This method is widely applicable to the production of nanoparticles from other relevant thin films, such as magnetic vortices or in-plane SAFs. The production of these types of particles is interesting for liquid applications, particularly in biomedical research areas, and provides a nanoscale alternative to the SAF microdiscs that have previously been implemented in magneto-mechanical destruction cancer therapy.

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