Magnetic and structural characteristics of ambient pressure fcc phase Ho and Tb thin films

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Abstract
We report the results of an investigation into the structural and magnetic properties of thin films of Ho and Tb sputtered on Ta-buffered Si substrates. As is often reported in thin films and nanoparticles of the rare earth (RE) metals, we observe both hcp and fcc phases where the relative fraction of each depends on the deposition conditions. The presence of a fcc RE phase at ambient conditions is generally claimed to be strain stabilised as in the bulk the fcc phase is only thermodynamically stable at elevated pressures. We find the lattice constants of the fcc phases in our films to coincide with values for the Ho- and Tb-hydrides, and analysis of the magnetic measurements shows that the Ho fcc phase is paramagnetic at ambient temperatures and antiferromagnetic at low temperatures, also a feature of the RE-hydrides. By considering both the structural and magnetic measurements on Ho and Tb films together, we demonstrate that the observed fcc phase is the antiferromagnetic rare earth hydride, which readily forms at the RE/Ta interface in the presence of the residual and outgassed hydrogen in vacuum chambers.

1. Introduction

The rare earth (RE) metals are amongst the most strongly magnetic materials known, and while their below-ambient Curie temperatures ($T_C$) have limited the industrial use of the metals to cryogenic applications, alloys of the rare earths are used in almost every application that needs a strong permanent magnet. The magnetic structure of the REs in bulk form was extensively studied starting in the 1950s [1–3] and is now well-known, if complex [4–6]. The REs are well-known to react strongly with moisture or atmospheric gases, and an array of oxides, hydrides and hydroxides with very different magnetic properties can easily form [7].

More recently attention has focused on investigating thin films of the REs, in particular for use in magnetic nanostructures for electronic data storage [8]. It has not yet been possible for thin films to achieve the saturation magnetisation values of the bulk REs, although recently Scheunert et al have achieved close to bulk saturation magnetisation ($M_S$) values for magnetron sputtered thin films of Gd, Dy and Tb [9–12].

RE single crystals exhibit a hexagonal close packed (hcp) structure. A particularly interesting difference between, on one hand, bulk and single crystal samples, and on the other thin films or nanoparticles produced by deposition in a vacuum chamber, are frequent reports of a face centred cubic (fcc) RE phase in the films and nanoparticles—seen originally by Curzon and Chlebeck in films of Er [13], but also observed in films of Gd, Tb, Dy, Ho and Tm [14–17] and in nanoparticles of Ho [18]. The presence of a fcc phase for the first ~10 s of nm in thin films, or for nanoparticles of < 30 nm diameter, has now been detected by many groups [12, 18–22]. These thin films have a reduced $M_S$ [12], and it was concluded the fcc phase in nanoparticles was paramagnetic [18, 21], although a recent report claims that a fcc Gd film is ferromagnetic with a higher $T_C$ than the hcp phase [20]. In contrast to these observations in thin films, the fcc phase in single crystal or bulk samples is stable only at high pressures; e.g. above 30 GPa for Tb and 35 GPa for Ho [23], an order of magnitude higher than pressures from structural mismatches at thin film/substrate interfaces [24–26] as suggested by some researchers [10, 12, 19].

Addressing this problem, Gasgnier et al [27] pointed out that the observed fcc thin-film lattice parameters match
that of RE hydrides [28, 29]. Later work showed that extended outgassing and gettering of residual hydrogen and water was important to allow the pure hcp RE metal films to form free of the fcc hydride phase [30].

This observed match between the lattice parameters of the fcc phase in RE thin films and the fcc RE hydrides has been ignored in all reports since 1980 [7]. In these modern reports, the fcc phase in thin films is universally attributed to a strain-stabilised fcc phase of the pure RE metal [12, 18–22]. Here we address this open question by an in-depth study of the magnetisation of thin films of Ho and Tb, along with their structural properties, as a function of the deposition conditions. The new magnetic measurements in particular demonstrate a direct match between the observed fcc phase in RE thin films, grown under typical high vacuum conditions, and the fcc RE hydride phase. Notably, we show for the first time that the observed Ho fcc phase in thin films is paramagnetic above about 10 K and antiferromagnetic at lower temperatures, exactly matching fcc HoH$_2$ [31]. We conclude that this is strong evidence that the fcc phase commonly observed in thin RE films is not, as recently claimed, a pure RE phase stabilised by strain [8, 9, 22, 32].

2. Experimental Details

Thin films were deposited by DC magnetron co-sputtering using a Kurt J Lesker CMS-18 system, under similar conditions to those used in other recent studies [10, 12, 20]. Sputtering was undertaken using 2” diameter targets of Ho and Tb with elemental purities of 99.9%. The vacuum chamber base pressure was below 6 × 10$^{-6}$ Pa and the Ar pressure during deposition was 0.80 Pa and 0.93 Pa for Tb and Ho respectively. The samples consist of tri-layers of Ta/RE/Ta on 0.5 mm thick 4” diameter Si (100) substrates. Before deposition the substrates were washed in acetone followed by isopropyl alcohol and de-ionised water before drying in a flow of dry N$_2$. After loading into the deposition chamber the bare substrates underwent a 5 min RF Argon plasma etch to remove residual contaminants.

The Ta target was pre-sputtered for 2 min to remove any residual oxide, followed by a 10 nm Ta buffer layer deposited at ambient temperature and at 0.09 nm s$^{-1}$. 100 nm$^{-1}$ RE films were deposited on to the 10 nm Ta buffer layer at a nominal substrate temperature of 350 °C and then capped with 100 nm Ta deposited at ambient temperature before removal from vacuum. Following preliminary depositions, and guidance from the literature [8, 9, 20, 32], three RE deposition rates were used: 0.1, 0.2, 0.3 nm s$^{-1}$. These rates were selected as they resulted in films exhibiting a useful range of fcc to hcp ratios. All growth rates were determined from separate calibration films grown under the same sputtering conditions, using a stylus profiler and SEM to measure the thickness to an accuracy of 5%.

The crystal structures were investigated using a Bruker D8 Advance X-ray diffraction system with Co K$_\alpha$ radiation at a wavelength of 1.79 Å. The EDAX capability of a FEI Quanta SEM found negligible oxygen contamination. Magnetisation was measured with a Quantum Design MPMS-7 SQUID magnetometer between room temperature and 2 K and to fields of 7 T.

3. Results and discussion

3.1. Structural characterization
The $\theta$–$2\theta$ and grazing incident X-ray diffractograms for films deposited at 350 °C are shown in figure 1. We note that the $\theta$–$2\theta$ measurements of figures 1(a) and 1(c) are relatively complex patterns exhibiting numerous lines from both hcp and fcc phases, along with lines from the Ta cap and buffer layers and lines from the Si substrate (marked with an “*”). First, we consider the lines related to the Ho in figure 1(a). The only line visible from the well-known hcp structure is the (002), appearing only for the film grown at the highest deposition rate of 0.3 nm s$^{-1}$. This implies that only at the highest growth rate is there a significant fraction of the hcp phase in the Ho films and in this case, it is highly textured. By contrast, for all Ho films in figure 1(a) there is a strong line present at 34.6 °C–34.9 °C previously identified as fcc (111) [12, 14, 18], and in the highest deposition rate film the second order fcc (222) line also appears at ~73.5 °C underneath a sharper Si substrate line.

The $\theta$–$2\theta$ diffractogram for the Tb films in figure 1(c) shows similar characteristics to those of the Ho films. In the case of Tb, the only hcp line is again the (002), though this time the line is visible for films grown at all deposition rates, with the line being stronger at higher deposition rates. The fcc (111) line is present for all Tb films.

By contrast to the complex $\theta$–$2\theta$ patterns, the grazing incidence (3°) measurements of figures 1(b) and 1(d) are much simpler, and almost all of the RE-related lines that are present in the $\theta$–$2\theta$ configuration are missing in these patterns. This shows that the RE phases are highly textured, since, in the grazing incidence configuration, crystal planes with scattering vectors aligned normal to the surface will not satisfy the Bragg condition. Notably, there is no sign of the fcc phase except in the lowest growth rate Ho film. This implies that the fcc phase is concentrated near the bottom Ta/RE interface, and the hcp phase is more prominent in films with higher...
deposition rates, with the strengthening (002) peak indicating an improved out-of-plane c-axis alignment. Our observations are consistent with those of Scheunert et al\cite{12}, where their film grown at 350 °C has a fcc phase initially forming on top of the Ta buffer, then an increasing fraction of the hcp phase as films grow thicker than ∼ 10–30 nm.

Figure 2(a) summarises the deposition rate dependent c-axis lattice constants extracted from the hcp (002) reflections in figures 1(a) and 1(c). The Ho c-axis hcp lattice constant at the highest growth rate coincides with reported single crystal values \cite{33} and is close to literature values for thin films \cite{14}. In our Tb films, the c-axis hcp lattice constant contracts systematically with increasing deposition rate, consistent with literature values \cite{12, 14} and an improving crystallinity and reduction in lattice strain with increasing deposition rate.
Figure 2 illustrates the lattice constants derived from the fcc \((111)\) reflections in figures 1(a) and 1(c), which are close to previously reported values in thin films \([12, 14]\). For both REs the fcc lattice constant decreases with increasing deposition rate. Also shown are the lattice parameters for the fcc RE hydrides HoH\(_2\) and TbH\(_2\), which are known to be stable under ambient temperature and pressure conditions \([29]\). It is striking that the lattice parameters of the fcc lines in our RE films match very closely literature values for RE hydrides. This outcome is consistent with inferences made by Gasgnier \([7, 27]\) and Singh et al \([35]\) in the 1970s, based on TEM and X-ray diffraction studies respectively, that the observed fcc phase in RE thin films was likely to be a RE hydride. This claim has been ignored since. In the next section we discuss a careful study of the magnetisation of Ho and Tb thin films prepared under the same deposition conditions as the films analysed by X-ray diffraction to provide a direct link between the observed fcc phase in thin RE films and the RE hydride.

3.2. Magnetic characterization

In order to confirm the nature of the fcc phase we have measured the magnetisation of our films. Figure 3(a) displays the temperature dependent magnetic induction, \(\mu_0 M(T)\), for the Ho films, and figure 3(b) shows the corresponding 10 K field loops. For Ho at a deposition rate of 0.3 nm s\(^{-1}\) there is a hysteresis at 10 K, indicating ferromagnetic behaviour. The temperature-dependent magnetisation starts to rise more steeply below \(\sim 150\) K before flattening off at low temperatures, a sign that the film undergoes a ferromagnetic-like transition \([36]\) as expected for hcp Ho. However, in the Ho films grown at 0.1 nm s\(^{-1}\) (and at 0.2 nm s\(^{-1}\), not shown) the magnetisation does not exhibit any ferromagnetic behaviour, notably no hysteresis. This demonstrates that there is no ferromagnetic hcp phase in these low growth rate films, and so the only phase is the fcc one as identified in the XRD results discussed above.

In contrast to the Ho, the magnetisation of the Tb films, as displayed in figures 3(c)–3(d), is consistent with the presence of ferromagnetic hcp Tb in all films deposited at 0.1, 0.2 (not shown) and 0.3 nm s\(^{-1}\), as demonstrated by the hysteresis at 10 K. The temperature dependence of \(\mu_0 M(T)\) in figure 3(c) becomes less steep near 220 K (see inset in figure 3(c)), which is consistent with the magnetic transition temperature for bulk hcp phase Tb. This is also consistent with the Tb XRD patterns, that exhibit a hcp phase at all deposition rates. We note that the ferromagnetic transition in figure 3(c) is significantly broadened due to the strong 7 T magnetic field employed, so that the transition temperature does not appear as a sharp change in slope.
In the bottom inset of figure 4, we show how we have estimated the saturation magnetisation, \( \mu_0 M_s \), for the ferromagnetic films, from an extrapolation of a M versus 1/H plot to 1/H = 0 [37]. In table 1 we report the measured \( \mu_0 M_s \) values. For Ho and Tb at the highest growth rate, \( \mu_0 M_s = 2.76 \) T and \( \mu_0 M_s = 2.56 \) T respectively. Table 1 also compares these values with bulk \( \mu_0 M_s \) values from the literature [1, 3, 38]. The measured values are considerably lower than those of bulk values for Ho (\( \mu_0 M_s = 3.87 \) T) and Tb (\( \mu_0 M_s = 3.40 \) T). X-ray diffraction measurements however, show that our films are a mix of hcp and fcc phases, with the fcc phase dominating near the initial growth interface, particularly in the case of Ho, and above we have shown that the fcc phase has different magnetic properties, as have others in nanoparticles [18, 21]. In table 1 we also display the adjusted \( \mu_0 M_s \) of the films that show the fcc phase in XRD. We assume that, in films that show a mix of fcc and hcp phases, the fcc phase is present in equal proportions in these films, is equivalent to 20 nm thickness, and does not contribute to \( \mu_0 M_s \). 20 nm was selected as being the middle of the often-quoted thickness range of 10–30 nm where the fcc has been observed in thin RE films [12, 20, 22]. The adjusted \( \mu_0 M_s \) values now reach ~90% of the reference hcp phase bulk values; 3.43 T for Ho films compared to 3.87 T for Ho bulk and 3.2 T for Tb films compared to 3.4 T for Tb bulk. Although this is a significant assumption, our analysis supports the existence of a non-ferromagnetic fcc phase fraction in all our Ho and Tb films and a growing ferromagnetic hcp phase fraction with increasing deposition rate.

Recalling that the 0.1 nm s\(^{-1}\) Ho film shows no sign of ferromagnetism, and is predominantly fcc (figure 1(a)), we further analyse the magnetisation behaviour of this film to identify the magnetic properties of the ambient-pressure fcc phase of Ho. The results of this film in figures 3(a) and 3(b) resemble paramagnetism, so we apply the Curie-Weiss (CW) law in the top inset of figure 4, \( \chi_m = C_m / (T - T_C) \), where \( \chi_m \) is the molar magnetic susceptibility, \( C_m \) is the molar Curie constant that depends on the effective moment, \( p_{eff} \), and \( T_C \) is the Curie temperature [39]. The estimated effective paramagnetic moment, \( p_{eff} = 10.8 \pm 0.1 \) \( \mu_B \) is close to the expected free Ho\(^{3+}\) ion moment \( p_{eff} = 10.6 \) [40], with a small negative temperature axis intercept of the fitted line.

In addition, in the main part of figure 4 we apply a Brillouin function fit to the field loop data taken at 10 K, as appropriate for non-interacting moments in a paramagnet [39].

Table 1. Saturation magnetic induction values derived from an extrapolation of a plot of M versus 1/H to 1/H = 0 [37], with and without correction for a 20 nm layer of non-ferromagnetic fcc phase material. Bulk values are taken from [1, 3, 38].

| Growth rate (nm s\(^{-1}\)) | Total film thickness (nm) | \( \mu_0 M_s \) (T) | \( \mu_0 M_s \) (20% fcc phase correction) | Bulk | \( \mu_0 M_s \) (T) |
|-----------------------------|--------------------------|----------------------|------------------------------------------|------|----------------------|
| Ho 0.3                      | 100                      | 2.76                 | 3.43                                     | Ho   | 3.87                 |
| Tb 0.2                      | 100                      | 2.28                 | 2.84                                     | Tb   | 3.40                 |
| Tb 0.1                      | 100                      | 2.16                 | 2.68                                     | Tb   | 3.40                 |
| Tb 0.3                      | 100                      | 2.56                 | 3.2                                      | Tb   | 3.40                 |
\[ M = N g J g B J(B, T) \]
\[ B J(B, T) = \frac{2J + 1}{2J} \coth \left( \frac{2J + 1}{2J} \frac{B}{J g B} \right) - \frac{1}{2J} \coth \left( \frac{1}{2J} \frac{B}{J g B} \right) \]
\[ y = \frac{J g B}{k_B T}, \]
(1)

where \( N \) is the number of magnetic moments per unit volume, \( g \) is the Landé g-factor, \( J \) is the total angular momentum, \( B \) is the magnetic field, \( T \) is the temperature and \( k_B \) is Boltzmann’s constant. Free paramagnetic Ho\(^{3+}\) ions have the [Xe]4f\(^{10}\) electron configuration with the \(^5I_8\) ground state and a \( J \) value of \( \sim 8 \) [40], however the fit results in a substantially lower effective \( J \) of 3. Our analysis implies that, while at higher temperatures the film acts as expected for largely non-interacting paramagnetic Ho\(^{3+}\) ions, there is a different magnetic state at low temperatures that is not clearly seen in the temperature-dependent measurements of figure 3. The value of \( J \) from the Brillouin fit reflects the steepness of the field loop at 10 K—the low fitted \( J \) value indicates that the moments of the Ho\(^{3+}\) ions require stronger magnetic fields to align them than if there was no exchange interaction between the moments. This implies antiferromagnetic interactions between the moments, that prevent them from aligning in a field, consistent with the small negative temperature axis intercept in the top inset of figure 4. Our results demonstrate the fcc phase in Ho thin films is paramagnetic at high temperatures, but with signs of antiferromagnetism near 10 K. These characteristics resemble those of fcc-HoH\(_2\), which is known to undergo several antiferromagnetic transitions below \( \sim 10 \) K [31]. We thus conclude that the magnetic data, when combined with the XRD data, demonstrates that the ambient pressure fcc phase behaviour is consistent with the presence of HoH\(_2\) in our films, and indicates that the fcc phase in the Tb films is similarly likely to be TbH\(_2\). The hydrogen stoichiometry of RE metals is well known to vary up to the tri-hydride. With increasing hydrogen content, the antiferromagnetic transition temperature \( T_N \) drops, becoming less than 4 K for the tri-hydride [41]. The magnetic data indicates that the hydride phase in our samples has a stoichiometry of at least 2 and perhaps higher.

### 3.3. Origin of the hydrogen

We note it is well known that hydrogen is the dominant residual gas at the low pressures used in modern sputtering systems due to the relative inefficiency of hydrogen pumping compared to other gases, and from outgassing from stainless-steel vacuum chambers, their components, and the heated source [42, 43]. Moreover, RE metals rapidly getter hydrogen [29], and RE evaporation has been known to reduce the hydrogen partial pressure in vacuum systems much more dramatically than evaporation of Ti [44]. While the fcc phase is present in XRD patterns for all our films, it is more prominent in thin films formed at lower growth rates. Under these conditions the high temperature growths will ensure hydrogen outgassing from the surfaces of the vacuum chamber, and there is a lower flux of RE to getter the outgassed hydrogen. To explain our results, we propose the following growth process for sputtered RE films in vacuum: at the start of the RE film deposition, the residual and outgassed hydrogen in the chamber reacts with the sputtered RE to form fcc RE-dihydride on the substrate. As deposition continues, sputtered RE progressively getters the residual hydrogen and also coats the surfaces of the vacuum chamber, hindering further outgassing. Eventually, the RE material arriving at the substrate encounters too little residual hydrogen to form the fcc hydride, and instead the RE hcp phase starts to appear. At higher growth rates, the enhanced gettering of hydrogen, faster coating of the chamber walls and more rapid RE film deposition quickly reduces the hydrogen level below that needed to form the hydride, such that the hcp RE phase dominates in these films earlier in the growth.

Our hydrogen-gettering hypothesis is supported by observing that the base pressure of the growth chamber drops considerably after deposition of the RE films compared to after deposition of transition metal Mo. Starting from a base of \( 4.3 \times 10^{-6} \) Pa, the pressure drops by a factor of 1.2 to \( 3.7 \times 10^{-6} \) Pa 12 h after room temperature growth of a film of Mo. By contrast, starting from a higher base pressure of \( 2.3 \times 10^{-5} \) Pa, the pressure drops by a factor of 30 to \( 7.9 \times 10^{-7} \) Pa only 5 h after the RE film deposition, due to the enhanced hydrogen gettering from the RE-coated surfaces. The pressure reached after RE deposition was much lower than that achieved after the Mo deposition, which clearly demonstrates the RE gettering effect. The gettering continues even after growth of the 100 nm Ta capping layers, whereby the pressure drops from \( 1.5 \times 10^{-5} \) Pa immediately after the Ta deposition to a minimum of \( 4.7 \times 10^{-7} \) Pa 5 h later.

### 4. Summary and conclusions

In summary, we have reported the results of an investigation into the structural and magnetic properties of thin films of Ho and Tb sputtered on Ta-buffered substrates. XRD studies clearly indicate the presence of both hcp and fcc phases where the relative fraction of each depends on the deposition conditions. It is well-established that in the bulk the fcc phase is only thermodynamically stable at elevated pressures, and we have found in our
films that the c-axis lattice constant for the fcc phase coincides with values for the Ho- and Tb-hydrides. Careful analysis of the magnetic measurements shows that the fcc phase is paramagnetic at ambient temperatures and antiferromagnetic at low temperatures. Combining the structural and magnetic measurements directly demonstrates that the observed fcc phase is the antiferromagnetic RE hydride, which forms at the RE/Ta interface in the presence of residual hydrogen in vacuum chambers due to the strong hydrogen gettering nature of the REs.

In conclusion we have shown that by carefully removing the hydrogen from the deposition system before the RE evaporation it is possible to prepare ferromagnetic RE thin films with bulk saturation values.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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