Planar array of self-assembled Ga$_x$Fe$_{4-x}$N nanocrystals in GaN: magnetic anisotropy determined via ferromagnetic resonance

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Abstract

The magnetic anisotropy of a planar array of Ga$_x$Fe$_{4-x}$N nanocrystals (NCs) embedded in a GaN host is studied by ferromagnetic resonance. X-ray diffraction and transmission electron microscopy are employed to determine the phase and distribution of the nanocrystals. The magnetic anisotropy is found to be primarily uniaxial with the hard axis normal to the NCs plane and to have a comparably weak in-plane hexagonal symmetry. The origin of the magnetic anisotropy is discussed taking into consideration the morphology of the nanocrystals, the epitaxial relations, strain effects and magnetic coupling between the NCs.

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(Some figures may appear in colour only in the online journal)

1. Introduction

Iron nitride (Fe$_x$N) compounds of various stoichiometries have lately raised much interest as suitable materials for magnetic recording applications [1–4] and have been extensively studied mostly as polycrystalline thin films and in a powder form. A particularly interesting iron nitride phase is $\gamma'$-Fe$_{4-x}$N, due to a high spin polarization of conduction electrons [10–13] which makes this material well suited for magnetic write heads and as spin injectors in semiconductors [14, 15]. On the other hand, gallium-nitride (GaN) and its compounds are not only strategic semiconductors for opto- and high-power-electronics, but—when alloyed with magnetic elements—are emerging as key systems with spintronic functionalities. Moreover, the combination of a nitride semiconducting matrix with embedded magnetic nanocrystals (NCs) is currently opening new frontiers to functional applications. Recently, the formation of Ga$_x$Fe$_{4-x}$N as secondary phases during the metalorganic vapour phase epitaxy (MOVPE) of Fe-doped GaN has been systematically studied, and control over the size, density and phase of the Fe-rich species has been obtained by varying the growth conditions and by codoping with acceptors or donors [5–8]. A further step was the growth on-demand of planar arrays of Ga$_x$Fe$_{4-x}$N NCs of a specific crystallographic phase at a defined position in the nitride host [9].

The determination of the magnetic anisotropy in hybrid semiconductor/magnetic NCs structures is essential for the design of functional devices. In this perspective we report here on the magnetic anisotropy of a planar array of Ga$_x$Fe$_{4-x}$N nanocrystals in GaN at room temperature determined by measuring ferromagnetic resonance (FMR) [16].
2. Experimental techniques

The samples studied are grown by MOVPE on c-plane sapphire substrates according to a procedure previously reported [9]. Specifically, the samples consist of the sapphire substrate, on which a low temperature (530 °C) nucleation layer is grown, followed by a 1 μm thick GaN buffer deposited at 980 °C on top of which the Fe-doped layer is grown in a (digital) δ-fasion, i.e. with the Ga flow switched on for 10 seconds and then off for 50 seconds over 30 periods at a temperature of 780 °C. The GaN:Fe layer is capped with GaN deposited at 980 °C. The precursors employed are ammonia (NH₃), trimethylgallium (TMGa) and ferrocene (Fe(C₅H₅)₂), the flow rates during the deposition of the buffer and of the capping layer are 1500 standard cubic centimetres per minute (sccm) for NH₃, and 25 sccm for TMGa. In the Fe-doped layer the NH₃ flow rate is reduced to 800 sccm, while the ferrocene flow rate is 490 sccm. When the TMGa source is enabled for the digital doping, it provides a flow rate of 5 sccm.

Transmission electron microscopy (TEM) techniques have been employed to establish the crystallographic phase, orientation and distribution of the nanocrystals in the GaN matrix [9]. Cross-section and plan-view TEM specimens are prepared by mechanical polishing, dimpling and ion milling in a Gatan Precision Ion Polishing System. Diffraction contrast experiments and high resolution TEM (HRTEM) imaging are carried out in a JEOL 2010F operating at 200 KeV.

In order to get further insight into the structure and orientation of the nanocrystals, the samples have also been investigated by high resolution X-ray diffraction (HRXRD). The measurements are performed in a Panalytical X’Pert Pro Material Research Diffractometer equipped with a hybrid monochromator with a 1/4° divergence slit. The diffracted beam is measured with a PixCel detector used as single channel detector with an active length of 0.5 mm, and a 5.0 mm anti-scatter slit.

The magnetic resonance measurements have been performed with a Bruker Elexys E580 electron paramagnetic resonance spectrometer capable of static magnetic fields up to 1.5 T and equipped with a continuous flow cryostat. An X-band microwave cavity is employed and the samples are carried out at microwave frequencies between 9.4 and 9.5 GHz. The static magnetic field has been modulated with an amplitude of 0.5 mT at 100 kHz to allow lock-in detection. The samples are cut into 4 mm² square specimens necessary to fit the sample space in the spectrometer for in-plane experiments. The out-of-plane measurements have been carried out by recording an FMR spectrum every 10° for one full rotation. In-plane measurements have been performed every 2° for half a circle. The microwave power has been adjusted to 2 mW for the in-plane measurements, while due to larger peak width 20 mW have been employed for the out-of-plane experiments.

3. Experimental results

In the TEM micrograph of a cross-section specimen reported in figure 1(a), it is shown that the NCs distribute on a plane perpendicular to the c growth direction, over a thickness of ~40 nm. Moreover, as estimated from a statistically significant number of plan-view images similar to figure 1(b), the average diameter of the NCs is (24.7 ± 5.2) nm. A similar analysis of 14 cross section TEM images yields an average size of (16.9 ± 2.2) nm along the c-direction of GaN and of (20.1 ± 3.9) nm perpendicular to it. The nanocrystals are estimated to occupy (4.8 ± 0.2)% of a basal plane.

The crystalline phase of the NCs is determined from long radial XRD scans collected along the c-axis of GaN as reported in [9] and established to be γ-Ga₅Fe₇N₃. Their orientation is determined from the position of the (111) asymmetric peak. The epitaxial relationships between the NCs and the host GaN have been identified as [001]NC∥[001]GaN and (001)[110]NC∥(0001)[1120]GaN, giving 12 equivalent in-plane orientations of the NCs, while their basal plane keeps a parallel orientation with respect to that of GaN. Moreover, reciprocal space maps of the NCs, (111) asymmetric peak have been collected at different azimuths. A displacement of the (111)NC from an azimuth aligned with the (1010)GaN to an azimuth rotated by 30° would indicate a uniaxial strain of the NCs, breaking the 12-fold symmetry. The reciprocal space map of the (111)NC collected at four different azimuths taken every 30° and reported in figure 2 does not show any modifications of the peak position in-plane, ruling out a substantial in-plane strain component in the NCs. Uniaxial strain along the [001] direction is not clearly detected, yet cannot be excluded throughout. Furthermore, the XRD measurements indicate that the minority of the nanocrystals is incorporated with the epitaxial relation (111)BC∥[0001]GaN. Their number cannot be quantified since their main diffraction peak overlaps with the (006) diffraction of the sapphire substrate.

Temperature dependent FMR measurements have been carried out, and the signal clearly visible at room temperature (RT) broadens with decreasing temperatures, quenching around 40 K. Therefore, the anisotropy measurements reported here are restricted to RT. In this context, the effects of thermal broadening may be considered [17] and a similar
temperature-dependent behaviour was reported by Bardeleben et al. for Co precipitates in ZnO and attributed to the nano-crystalline structure of the films [18].

Due to the equivalent in-plane orientations of the nanocrystals analyzed here, one would expect to detect three FMR lines, which coincide when the magnetic field is perpendicular to the sample surface ($\theta = 0^\circ$). Nevertheless, only one line can be observed, which shows an uniaxial dependence ($\cos^2(\theta)$) when varying the out-of-plane angle $\theta$, while measurements with the magnetic field in-plane ($\phi = 0^\circ$ and $\phi = 30^\circ$, respectively).

4. Discussion

In order to shed light on the origin of this uniaxial angular dependence, we consider the shape of the nanocrystals as determined by HRTEM, since any non-symmetric uniformly magnetized particle will show uniaxial shape anisotropy. By approximating the nanocrystals to oblate spheroids [19] and using the nanocrystal’s dimensions mentioned above, the anisotropy tensor in diagonal form has the following components:

$$
N_{xx} = N_{yy} = 0.31 \pm 0.03 \quad \text{and} \quad N_{zz} = 0.38 \pm 0.06.
$$

The error bars of the shape anisotropy do not rule out that on average the NC shape could be isotropic. In this case, strain induced by the GaN host crystal on the cubic Ga$_x$Fe$_{4-x}$N NCs can serve as an explanation for the observed uniaxial anisotropy, since it would generate uniaxial terms in the free energy, which, assuming a high enough prefactor, could dominate over the cubic terms from the iron nitride crystal lattice. Alternatively, a sizeable magnetic coupling between the nanocrystals supported by the planar arrangement would as well lead to uniaxial anisotropy [18]. The coverage required to have coupling energies comparable to the thermal energy at RT can be estimated using the results in [20] and correcting the energies reported by the square of the ratio of the saturation magnetization of pure Fe ($1.76 \times 10^6$ A m$^{-1}$) and Fe$_{4-x}$N (whose value was experimentally established to be $1.42 \times 10^6$ A m$^{-1}$ in thin films [22], and

![Figure 2. XRD of Ga$_x$Fe$_{4-x}$N nanocrystals in the GaN matrix: (a) $\theta - 2\theta$ spectra, showing the alignment of the (002) of the Ga$_x$Fe$_{4-x}$N nanocrystals with the (002) of GaN; (b) and (c) reciprocal space maps measured around the (111) peak of Ga$_x$Fe$_{4-x}$N in the (x0z) and (x0x) planes of the GaN reciprocal space ($\phi = 0^\circ$ and $\phi = 30^\circ$, respectively).](image)

![Figure 3. Angular dependence of the FMR signal (points), plotted together with the fitting results of the models (i)-(v) described in the text. Solid line: phenomenological crystal field anisotropy; dashed line: fits with cubic nanocrystals with their [111] axis along the [001] axis of GaN, having the uniaxial out-of-plane anisotropy induced by shape anisotropy or coupling, both yielding the same fit quality and line-shape; point-dashed line: fits accounting for the phenomenological hexagonal in-plane anisotropy and having the out-of-plane uniaxial anisotropy induced by shape or coupling, both giving commensurate fit quality and line-shape. (a) Out-of-plane FMR measurement: the in-plane angle is fixed at $\phi = 0^\circ$, and the out-of-plane angle $\theta$ has been varied between 0 and $2\pi$; (b) in-plane data-points; here the out-of-plane angle is kept at $\phi = 0^\circ$, and the in-plane angle $\theta$ is varied between 0 and $\pi$.](image)
Table 1. Material parameters obtained by fitting the angular dependence of the FMR (details are provided in the Additional Material). In the models (i), (ii) and (iii) the nanocrystals are treated as isolated magnetic moments. The model (i) is purely phenomenological and includes a uniaxial/hexagonal anisotropy, where $K_1$ and $K_2$ are the prefactors of the second and fourth order uniaxial term, and $K_3$ is the prefactor of the sixth order hexagonal term; in (ii) the prefactors of the uniaxial crystal strain are set to zero, while the model includes the shape information from HRTEM for shape anisotropy; for (iii) the shape information from HRTEM is employed and the nanocrystals are modeled with a cubic anisotropy and with their [111] direction parallel to the [001] direction of GaN.

| Individual Crystal/Strain | Shape + Hex | Shape + Cube | Hexagonal | Cubic |
|---------------------------|-------------|-------------|-----------|-------|
| $g$                       | 2.069       | 2.069       | 2.065     | 2.069 |
| $m_{sat}$ [kA m$^{-1}$]   | 1443±12     | 695±5       | 589±8     | 49±0.4 |
| $K_1$ [J m$^{-1}$]        | −40492      | set 0       | 729       | set 0 |
| $K_2$ [J m$^{-1}$]        | ±495        | ±170        | ±7768     | ±551  |
| $K_3$ [J m$^{-1}$]        | ±251        | ±1000       | ±1000     | ±71   |
| Fit Error                 | 57.46       | 91          | 165       | 91    |

1.51×10$^9$ A m$^{-1}$ in powders [3, 23, 24]. Based on these figures, the coverage of (4.8 ± 0.2)% obtained by (HR)TEM for the samples considered in this work represents a border limit for the observation of coupling between the NCs at room temperature.

In order to elucidate the origin of the observed in-plane magnetic anisotropy it is mandatory to take into consideration a number of factors. Shape anisotropy can be ruled out, since for single domain particles only uniaxial like shape anisotropies are enabled. While the phenomenological theory of magnetic crystal or strain anisotropy does not exclude the appearance of high order terms, which would not cancel out for the given strain geometry, microscopic arguments speak against strain as a possible explanation, since if a quadratic lattice is isotropically stressed along three directions—with an angle offset of 120° between them—the resulting lattice will still be a square one. Also, due to the small size of the nanocrystals considered, relaxation of strain is not to be expected; therefore a local change in magnetic anisotropy within a NC is unlikely. On the other hand the observed 6-fold anisotropy is compatible with the arrangement of a minority of the nanocrystals with their [111] direction aligned parallel to the [001] direction of GaN. Such NCs, together with the uniaxial anisotropy induced by their shape, yield an angular dependence close to the one observed. The angle dependence of the sum of the three peaks due to the other crystallographic orientations nearly averages out (as it can be seen by plotting the line-shape as a function of the angle), while the 6-fold anisotropy of the minority of crystals dominates. Although, as observed by HRTEM, the interface between the NCs and the GaN matrix is atomically flat, intermixing cannot be excluded. The hexagonal crystal structure of the intermixing region pseudomorphic with wurtzite GaN in the proximity of the NCs would generate an antiferromagnetic shell surrounding the NCs and give rise to a hexagonal magnetic crystal anisotropy. For the nanocrystals considered here, the ratio between the atoms at the surface and those in the NC bulk is of the order of 10, thus even a thin antiferromagnetic layer surrounding the nanocrystals is likely to couple to them and to induce a sizeable effect.

The presence of non-interacting paramagnetic Fe$^{3+}$ ions within the GaN host does not show in plane magnetic anisotropy [25] and can be ruled out as source of the measured effect. On the other hand, the aforementioned possibility of magnetic coupling between nanocrystals even at room temperature would play a role in producing magnetic anisotropy. As stated, high order terms of uniaxial strain anisotropy are possible in the phenomenological description, and if the nanocrystals—singularly strained along one of the equivalent directions in the host crystal plane—couple rigidly (i.e. with a fixed phase relation of the precessional motion of their magnetization), the overall free energy will contain terms that do not cancel out and that give rise to a hexagonal in-plane anisotropy. However, the HRXRD data does not provide evidence of a significant in-plane strain. In combination with magnetic coupling, the cubic crystal anisotropy of NCs with their [111] direction parallel to the [001] of GaN can also describe the observed angular dependence of the FMR signal.

To fit the measured data, five different models have been tested, specifically: (i) fit of a hexagonal crystal anisotropy, without information on the NC’s morphology from HRTEM, and yielding apparent anisotropy constants; (ii) an individual treatment of the nanocrystals, including the average dimensions as obtained from HRTEM for the shape anisotropy and adding a phenomenological hexagonal in-plane anisotropy term; (iii) a model of individual nanocrystals of cubic crystal anisotropy with their [111] direction along the [001] of the GaN host and taking into account the shape information from HRTEM; (iv) nanocrystals as a rigidly coupled system, i.e. with the shape anisotropy of an infinitely extended thin layer, yet keeping the phenomenological 6-fold symmetry term; (v) a model treating the whole planar array of nanocrystals as a rigidly coupled system of cubic NCs with the [111] direction along the [001] of the GaN host. The formalism employed for the different models is reported in the Additional Material. In order to determine the resonance frequency, the method described by Smit and Beljers [26] is employed, which allows us to calculate the frequency $\omega$ for FMR conditions from the free energy $F$, written as a sum over the different anisotropy
contributions and with $\gamma$ as gyromagnetic ratio:

$$\left(\frac{\omega}{\gamma}\right)^2 = \frac{1 + \alpha^2}{M^2 \sin^2 (\theta)^2} \left(\frac{\partial^2 F}{\partial \theta^2} \frac{\partial^2 F}{\partial \phi^2} - \left(\frac{\partial^2 F}{\partial \phi \partial \theta}\right)^2\right).$$  \hspace{1cm} (1)

In the fit, the equilibrium magnetization direction ($\theta, \phi$) has been determined by minimizing the free energy for each computation of the resonance frequency, while the damping $\alpha$ has been neglected. All five models describe the observed data well, but the best agreement is nevertheless obtained with the purely phenomenological hexagonal crystal anisotropy model. The $g$-factor, the saturation magnetization and the anisotropy constants of the crystal anisotropy have been fitted and the values are reported in Table 1.

Since the uniaxial anisotropy generated by the NC shape, the saturation magnetization and the uniaxial crystal field anisotropy term are mutually dependent, the obtained quality of the fit is commensurate for the models including the phenomenological hexagonal term but without the fourth order uniaxial term, and for the two models assuming the cubic anisotropy. The variation is restricted to the fitted anisotropy constant values.

While the obtained $g$-factor differs from the value reported for Fe$_4$N thin films [27], in the frame of the hexagonal model, which does not include the shape anisotropy, the fitted saturation magnetization of $1.44 \times 10^6$ A m$^{-1}$ is very close to the literature values $1.42 \times 10^6$ A m$^{-1}$ [22] and $1.51 \times 10^6$ A m$^{-1}$ [3, 23, 24]. When taking into account the shape anisotropy using the average NC aspect ratio as determined by HRTEM, the saturation magnetization given by the fits is lower than the literature value, yet one has to keep in mind the relatively large error bars for the shape anisotropy tensor components, which allow a broad range of fitted saturation magnetization values without changing the quality of the fit. In order to obtain the literature value for the saturation magnetization of Fe$_4$N, one would have to set an aspect-ratio of 1.08, which is within the error bar of the size information from HRTEM. Also, since the lattice parameters of Fe$_4$N and GaFe$_2$N are comparable, the incorporation of Ga into the NCs, which would lead to a decreased saturation magnetization, cannot be ruled out. Regarding the models assuming rigidly coupled nanocrystals, the saturation magnetization is much weaker than the one reported for thin films. This may be attributed to the fact that in the assumed arrangement of coupled NCs a substantial fraction of the volume is actually represented by paramagnetic dilute (Ga,Fe)N, as proven by SQUID magnetometry measurements [28].

In summary, measurements of room temperature FMR of a planar array of $\gamma$-Ga$_x$Fe$_{4-x}$N nanocrystals in a GaN host reveal a strong uniaxial magnetic anisotropy with three easy axes in the plane normal to the $c$-axis of GaN. The uniaxial anisotropy can be described by the shape of the nanocrystals, and the hexagonal in-plane anisotropy can be understood by considering the presence of a minority of cubic nanocrystals with their [111] axis aligned along the [001] direction of the GaN host, whose presence is indicated by XRD. The data can also be described within a conventional hexagonal magnetic crystal anisotropy model, and a significant agreement is obtained for a layer of coupled crystals.

A broad range of applications for metallic and magnetic nanocrystals in a semiconductor matrix is envisaged and among the most thrilling prospects one can mention the option of exploiting them as spin current injectors into the semiconductor host crystal [13], possibly via FMR spin pumping [14, 15] and spin current detection by inverse spin Hall effect [35]. A further application is directed at electric flash memory-like data storage [36, 37]. For magnetic storage in-plane anisotropy is required, and it can be expected to be induced by e.g. elongated nanocrystals obtained by modulating the growth conditions [38]. Moreover, the control of the magnetic coupling between nanocrystals makes the hybrid semiconductor/NCs system an exciting candidate with which to study frustrated magnetic systems and spin glass behaviours [20].
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