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Characterization of microscopic ferromagnetic defects in thin films using magnetic microscope based on Nitrogen-Vacancy centres

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Abstract. In this work we present results acquired by applying magnetic field imaging technique based on Nitrogen-Vacancy centres in diamond crystal for characterization of magnetic thin films defects. We used the constructed wide-field magnetic microscope for measurements of two kinds of magnetic defects in thin films. One family of defects under study was a result of non-optimal thin film growth conditions. The magnetic field maps of several regions of the thin films created under very similar conditions to previously published research revealed microscopic impurity islands of ferromagnetic defects, that potentially could disturb the magnetic properties of the surface. The second part of the measurements was dedicated to defects created post deposition - mechanical defects introduced in ferromagnetic thin films. In both cases, the measurements identify the magnetic field amplitude and distribution of the magnetic defects. In addition, the magnetic field maps were correlated with the corresponding optical images. As this method has great potential for quality control of different stages of magnetic thin film manufacturing process and it can rival other widely used measurement techniques, we also propose solutions for the optimization of the device in the perspective of high throughput.

Keywords: Wide-field magnetic microscopy, Ferromagnetic thin film, Surface defect characterization, Optically detected magnetic resonance, Nitrogen-Vacancy centres in diamond

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1. Introduction

The Nitrogen-Vacancy (NV) centres in diamond crystal have potential for a variety of applications, from magnetometry [1], thermometry [2] and measurements of electric field [3, 4] to strain analysis [5]. An ensemble of NV centres that is fixed in concrete place in a diamond crystal can also be used to acquire 2D maps of the aforementioned physical properties.

Using NV centres for magnetic field imaging has a number of advantages: possibility to measure relatively wide area of sample simultaneously, while maintaining diffraction limited spatial resolution. A diamond crystal itself is a convenient base for variety of measurement conditions, as it can be brought into close proximity to the sample, as the diamond matrix is chemically and mechanically durable, as well as non-toxic. Furthermore, measurements can be made over a range of temperatures from cryogenic to several hundreds of degrees Celsius [6, 7, 8]. The combination of these properties allows to investigate magnetic properties of thin films and magnetic structures on a microscopic scale [9, 10], potentially allowing to monitor the manufacturing process of various structures as well as quality control of the final product.

The magnetic imaging could be useful for a number of thin film and microstructure applications [11] as it is based on standard microscopy techniques and therefore allows to combine optical images with magnetic field images. Examples for this are estimation of magnetic moments of microscopic magnetic particles [12], magnetic domain topology [13], ferromagnetic thin film growth [14, 15], visualization of magnetic leakage fields [16] and orientation of liquid crystals [17].

In this research we focused on imaging of ferromagnetic structures for several reasons: structural defects in ferromagnetic thin films tend to create magnetic defects with complicated patterns, ferromagnetic materials exhibit complicated properties even at low external magnetic fields and at room temperature, and ferromagnetic thin films have large variety of practical applications. For example, research of magnetic phases and their dependence of material thickness [18], magnetic property research by material contents and processing [19], wide range of perovskite structures [20, 21, 22], magnetic shape memory thin films [23, 24, 25] as well as resistive random access memory [26], to name a few.

To demonstrate the capabilities of the magnetic field imaging technique we applied it to observe magnetic defects on a thin film produced under conditions very similar to previously published research. We found out that ferromagnetic signal comes from impurity defects condensed in microscopic islands as a result of non-optimal thin film growth process. Another group of measurements were done in ferromagnetic thin films which were exposed to mechanical interaction.

2. Methods used

To measure the properties of ferromagnetic thin films we used an optically detected magnetic resonance (ODMR) method. In FIG. 1 one can see the NV energy scheme which provides the basis for the measurement of ODMR signals. A frequency doubled Nd:YAG laser light (532 nm) is used to optically excite the NV centers. After light absorption with absorption rate $\Gamma_p$ and rapid relaxation in the phonon band the population of the excited state magnetic sublevels can either decay back to the ground state with equal rates $\Gamma_0$ and radiate light in the red part of the spectrum or undergo non-radiative transitions to the singlet level $^1A_1$. These non-radiative transitions occur approximately five times more frequently from the excited state electron spin sublevels $m_S = \pm 1$ compared to $m_S = 0$. After the first step the singlet–singlet transition $^1A_1 \rightarrow ^1E$ takes place with almost all the energy being transferred in a non-radiative way with a small fraction radiated in form of IR radiation. Finally, the population undergoes non-radiative transitions from $^1E$ to the ground triplet state with approximately equal transition probabilities to all three electron spin projection components of the $^3A_2$ level. Although the literature data is inconsistent and the relaxation rates vary in rather wide range [27, 28, 29], in all cases the differences of the non-radiative transition rates in the excited triplet state $^3E$ leads to the situation that after several excitation–relaxation cycles the population of the NV centers in the ground triplet state will be transferred to the magnetic sublevel $m_S = 0$ and the electron spin angular momentum will be strongly polarized [30]. In our measurements we used continuous laser excitation.

The optical polarisation can be used to measure magnetic field in the following way. The transitions from the ground state $m_S = \pm 1$ states due to the larger probability of population relaxation to the non-radiative transitions results in lower luminescence signal. If the system starts with an optically polarized ground state (population in $m_S = 0$) and we add microwave frequency scan that at some point energetically connects the $m_S = 0$ and $m_S = -1$ or $m_S = +1$ levels, we will observe a drop in luminescence signal at the exact resonance frequency. This in order gives the distance between the ground state levels, and can be used for magnetic field measurements as the $m_S = \pm 1$ split in the external magnetic field. If the external magnetic field is aligned along one of the NV axes the $m_S = \pm 1$ states of the corresponding NV...
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3. Data processing

The acquired data (full ODMR shape for each pixel) was processed in the following way. The ODMR signal was fitted with a combination of three Lorentzian profiles (corresponding to each of the three hyperfine components) for each pixel, where each experimental point represents a narrow MW field value. This is done to account for the asymmetry in the line-profile (see Figure 2) due to polarization of the nitrogen nucleus forming the NV center [30, 32]. The obtained fit values for the transition frequencies (minimum value of the fit curve) were plotted as a 2D heat-map revealing the spatial distribution of the magnetic field.

Determining the minimum value from the fitted curve lessens the imperfections in data quality as noise and relatively sparse data points at the tip of the resonance shape. In this case seemingly the nuclear spin polarisation plays small role in the shape of the ODMR profile, but it has to be noted that the shape and FWHM of the resonance is this wide due to the specifics of the thin film samples - the local magnetic field changes rapidly and even one pixel of the camera detects NV centres with different resonance frequencies. For comparison the same diamond crystal and set-up with small modifications were used to measure other type of samples [33], and gave notably narrower FWHM and much more pronounced signature of the nuclear spin polarisation. The MW power and magnetic bias field used in the measurements were roughly the same, but the laser power in this research was two times smaller.

4. Experimental Device

The experimental scheme for magnetic field imaging device is depicted in Figure 3. We used a Coherent Verdi V-18 laser to excite the NV centres. Laser power at the sample was around 40 mW. We used a dynamic transmissive speckle reducer (Optotune LSR-3005) to suppress interference artifacts (very pronounced in cases with well reflecting samples) originating from the thin air gap between the diamond and the sample. A lens system was used to optimise the lighting over the field of view. In our case the field-of-view was a 110x110 µm² square. We used an epifluorescent set-up where the excitation and luminescence detection are done through the same optical path and the red luminescence was separated by dichroic mirror (see Fig. 3). A 40x infinity-corrected objective with an NA of 0.65 was used, giving a theoretical resolution limit of 650 nm at the average value of luminescence wavelength of 700 nm (note that in this case the resolution applies to the...
Figure 3. Experimental setup. The laser light was coupled into an optical fiber that led it to the experimental system, in which a dichroic mirror (Thorlabs DMLP567R) reflects the green light, which, in turn, was directed to the sample via optical system that insured smooth distribution of exciting radiation across the field of view. The luminescence from the sample was collected with a 40x infinity-corrected objective with an NA of 0.65, and, after passing through the dichroic mirror and a long-pass filter (Thorlabs FEL0600), it was focused onto the sCMOS matrix of the camera (Andor NEO 5.5) or a photodiode (Thorlabs PDA36A-EC).

Figure 4. SRIM simulation of the $^{14}\text{N}$ ion distribution after 3 successive implantations at 60, 35 and 10 keV (cumulative dose of $5.5 \cdot 10^{12} \text{ ions/cm}^2$).

The diamond sample we used (bought from Element 6) was an electronics grade type IIa diamond with a (100) surface polish. The physical dimensions of the crystal are $3 \text{ mm} \times 3 \text{ mm} \times 0.1 \text{ mm}$. The crystal was irradiated with $^{14}\text{N}$ ions at three separate energies - 10 keV, 35 keV and 60 keV (cumulative dose of $5.5 \cdot 10^{12} \text{ ions/cm}^2$) - at the facilities of CuttingEdge Ions, LLC. The diamond was then annealed at 800 °C under vacuum. This increases the vacancy mobility such that they could migrate and be capture by substitutial nitrogen defects form NV centres. The activation energy of nitrogen is higher than that of the vacancies [34] and they can be considered immobile. Since the only source of substitutional nitrogen in an electronics grade diamond is the implanted ions, the ion distribution determines the final NV distribution. A Stopping Range of Ions in Matter or SRIM simulation [35] was performed to determine the NV distribution and the result is shown in Fig. 4. While the final distribution is multi-modal, both the optical resolution and characteristic feature size under investigation are significantly larger than the NV layer thickness and the inhomogeneity in the NV distribution does not play a significant role in the performance of the magnetic microscope.

5. Results and discussion

To demonstrate defect detection technique using NV centres and ODMR approach we used two types of ferromagnetic thin films, one being a thin film with ferromagnetic impurities resulting from non-optimal growth conditions and the second being a ferromagnetic films with ex situ introduced mechanical defects. All of the measurements were performed at room temperature.

In the first case, nanolaminated (atomically lay-
ered) thin films of (Cr$_{0.5}$Mn$_{0.5}$)$_2$GaC were chosen, whose properties and deposition conditions are described elsewhere [36]. This thin film was of specific interest, as similar thin films were measured before using vibrating sample magnetometry, ferromagnetic resonance and SQUID magnetometry [36, 37]. However, even grown under optimal or near-optimal conditions, produced films can contain a small amount of surface impurities, which in turn can produce a ferromagnetic signal in addition to the signal arising from main phase present in the thin film. The magnetic field imaging technique based on NV centres allows to pinpoint the existence and origin of these signals, as well as to identify the magnetic properties of these structures.

The first set of measurements (Figures 5 and 6) are depicting two separate locations on the surface of a single thin film sample (field of view 110x110 $\mu$m$^2$): the left-side panels present optical images acquired by illuminating the sample with a white light source, and the right-side panels present the ODMR maps (magnetic field images), where the color scale represent a relative shift of the resonance frequency. In both cases there are a very distinct structures with varying shapes that create difference in relative magnetic field shifts approaching 1 mT. These shapes are most likely attributed to impurities consisting of tetragonal Mn$_5$Ga$_2$, forming islands on the main thin film, as they were clearly seen in X-ray diffraction along with Mn$_5$Ga$_8$ impurities in other films during early stages of film optimization (not published). The choice of Mn$_5$Ga$_2$ being the impurity that is seen in magnetic imaging over Mn$_5$Ga$_8$ is motivated by the magnetic transition temperature being $T_C$=743 K for Mn$_5$Ga$_2$ and $T_C$=210 K for Mn$_5$Ga$_8$ [38], while all measurements represented here are done at room temperature. Although these shapes resemble grains on the surface, these structures are relatively flat as they are in the optical focus at the same time as the surface of the main body of the thin film is. The non-magnetic structure, that can be seen in both optical images is pure gallium (lighter shade covering relatively large surface areas). Interestingly, the islands (prominently seen in optical image of Fig. 5) seem to strip away some part of the gallium from the surface to form the island. We deduce that the element on the surface of the thin film is gallium, as it has been demonstrated in similar nanolaminated thin films [39, 40] that the A element of the $M_{n+1}AX_n$ phases [41] tend to segregate to the surface of the film.

Although at first glance the magnetic dipoles formed by the islands might seem to be oriented in the same direction, upon closer inspection it can be clearly seen that the orientation follows some general direction, but the deviations from this direction can be rather large. For example, in Fig. 6 the orientation of the dipoles on the right side and the left side of the image differs by some 15°. Similarly, in Fig. 5 the orientation of the dipoles differs by some 20°. It is known that there exists a certain in-plane epitaxial relationship between single-crystal MgO substrate and (Cr$_{0.5}$Mn$_{0.5}$)$_2$GaC thin films, as described in [36], thus there most likely also exists an epitaxial relationship between crystalline Mn$_5$Ga$_2$ impurities and (Cr$_{0.5}$Mn$_{0.5}$)$_2$GaC thin films, meaning that the impurities would tend to follow a certain in-plane growth direction on the film and these impurity crystals would also magnetize more easily along the easy axis of the material. The difference between magnetization direction between these different crystals is most likely attributed to some variance in growth direction. And by applying a small magnetic field during the measurement, these different crystals magnetize along the easy axis of the material.

Another aspect describing the magnetic islands is their visible correlation of size to the strength of the magnetic field. For example, in Fig. 5 it can be clearly seen that a small island located near the centre of image creates relatively small deviations from the central frequency of the magnetic image, whilst the increasingly larger islands create increasingly larger deviations from the central frequency of the magnetic image. This hints to crystalline structure of the islands with a defined magnetic domain orientation.

It has to be noted that at the edges of the magnetic images one can see some magnetic structures, that can not be identified in the optical images - these patterns occur due to defects outside field-of-view, but as the created magnetic field extends beyond the physical dimensions of magnetic structures, one can see them in magnetic images.

It should be noted that the analysis performed in publications [36] and [37] was done on thin films grown under optimal or near optimal conditions, whereas the thin films analysed in present work have been synthesized under very similar, but slightly differing conditions, thus leading to surface impurities, which were present on some parts of the film surface. Another aspect that must be stressed here is the magnetic properties of interest. While the two papers [36] and [37] concentrated on the magnetic properties of the thin film itself (prominently revealed at cryogenic temperatures), we focused on the surface properties that might be relevant for some applications.

In the upper central part of the ODMR map of Fig. 6, denoted with a dashed circle, there is a defect that is not related to the magnetic properties of the thin film, but rather to a local defect within the diamond crystal lattice, this is a well known phenomenon [5, 42, 43].

The second type of magnetic films that were
analysed are samples with ex situ mechanical defects on the 500 nm thick iron thin film, whose magnetic images are shown in Fig. 7 (needle punch) and Fig. 8 (defect introduced by assembly knife, consisting of multiple lines). In both cases the magnetic signature of the thin film defect is clearly visible in contrast to the spots where the thin film is intact. As expected ferromagnetic thin film creates strong variations of local magnetic field (variations reaching 1 mT difference) and spatially rapidly changing magnetic pattern at the spot where the integrity of the thin film is broken.

To better understand the magnetic signals and their origins we simulated the structure that can be seen in Fig. 7. The magnetic field was simulated with COMSOL, a finite-element-modelling software. The source of the magnetic field was a homogeneously magnetized 500 nm thick film with a cut-out (left panel of Fig. 9) shaped similarly as the defect in the left panel of Fig. 7. The magnetization direction was assumed to be colinear with the applied magnetic bias field and thus also the NV axis. The magnitude of the magnetization was chosen based on the observed signal amplitude and set equal to 7.5 \cdot 10^4 A/m. The plot (the right panel of Fig. 9) depicts the projection of the magnetic field on the NV axis 10 \mu m under the surface of the homogeneously magnetized layer. As can be seen, the result of the simulation in Fig. 9 qualitatively gives the same magnetic field shape as the measured defect in Fig. 7. Interestingly, the simulations showed that the 10 \mu m gap is responsible for the blurriness of the magnetic field structures, as smaller gaps (e.g., 1 \mu m) gave much sharper depiction of the shapes.

In the case of surface defects introduced by an assembly knife (Fig. 8) it is visible that the defect structure is relatively rich with scratches differing in width and depth that are not clearly distinguishable in the magnetic image. Similarly to the case of the needle punch, the blurriness of the shapes could be explained by an air gap between the NV layer and the sample. As in the previous case with the ferromagnetic impurity islands, the magnetic field creates a significantly larger magnetic pattern around the defect than its physical dimensions. This can be a useful effect if the magnetic field imaging technique is used for a quality control of thin film parameters (for example magnetic shape memory thin films and resistive random access memory, mentioned in the Introduction), as it helps to magnify the the defect size, thus even a defect with dimensions well below the optical resolution could be clearly seen.

With examples that clearly demonstrate the capabilities of the magnetic field imaging technique let us discuss the optimisation perspectives of similar devices. To use magnetic field imaging for quality control of different steps of magnetic thin film fabrication, there can be various aspects that could be easily modified to upgrade performance of the device. The first thing is the measurement time - to acquire smooth magnetic field images, as shown in figures 5, 6, 7 and 8 the measurement time was around 20 minutes per one ODMR map. To potentially identify the shift from the expected value, the measurement time could be reduced by many orders of magnitude. In our measurements the MW frequency was scanned across 40 MHz range (yielding 40 data points) to obtain full information about the ODMR shape. This parameter can be reduced to 3 concrete MW frequencies if the direction of the frequency shift is important, or to a two MW frequency mode if only deviation from concrete magnetic field value is monitored (one frequency to monitor signal level, other to monitor changes in contrast). Another factor determining the throughput of similar devices is the contrast of the ODMR signals. In this case we used very modest laser power, as our set-up was built to correlate the optical image with the magnetic image, thus the laser light reached the magnetic sample itself. If the surface of the diamond crystal would be covered in a reflective thin film, or the laser excitation was provided in the direction parallel to the surface of the magnetic thin films, one could use much larger laser power, insuring better contrast of ODMR signals. This would result in smaller number of required averages, that in order could easily reduce the measurement time by two orders of magnitude.

One more aspect that needs to be considered is the properties of the possible defects to be studied. The advantage of magnetic detection of defects in ferromagnetic thin films lies within the fact, that in contrary to defects studied in this work, structural imperfections can occur within the volume of a thin film, with no visible surface deformation. In such case the magnetic structure will still be detectable, while the visual surface monitoring methods will fail to detect the defect.

6. Conclusions

We have demonstrated the experimental results that clearly identify the different magnetic properties of structures on the surface of thin films caused by two types of surface defects: surface impurities formed during the thin film growth and defects created by mechanical interaction with the thin film. The studied samples show the strengths and advantages of wide field magnetic microscopy with high spatial resolution in detection of local magnetic defects. This was clearly demonstrated by pinpointing and describing the presence of ferromagnetic impurities, attributing the ferromagnetic behavior to microscopic
islands on the surface of the thin film. The mechanical defects on a solid iron thin film were also described successfully, showing the complex magnetic structure created by damaged integrity of the thin films. We have also discussed the first steps of optimization of similar systems towards higher throughput. In comparison to widely used vibrating sample magnetometry the magnetic field imaging method based on NV centres delivers sub-micrometer scale spatial resolution, opening up new possibilities in thin film research and quality control. In comparison to scanning tip magnetometry performed by atomic force microscopy devices, superconducting quantum interference devices (SQUID) or single NV probes [44] the method presented here delivers a wide-field magnetic microscopy (whole field of view is measured simultaneously), that cannot compete in sensitivity, but is superior in measurement speed and potential throughput as well as is not disturbed by rapid changes in the height and depth of the measured structures. Additionally, the flat surface of the diamond surfaces, usually used in imaging experiments, somewhat lessens the probability of local mechanical interaction with the sample under study.

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Figure 6. Measurements of (Cr$_{0.5}$Mn$_{0.5}$)$_2$GaC thin film surface defects created due to non-optimal thin film growth process. **Left panel:** optical image taken using the same optical system but with white light illumination. The lighter material prominently covering right half of the field of view is Gallium (nonmagnetic). The islands forming the local magnetic field defects are attributed to impurities consisting of tetragonal Mn$_3$Ga. **Right panel:** The ODMR map representing the magnetic field changes over the field of view. The defect in the dashed circle is related to inner strain of the diamond crystal, and not to the properties of the thin film. In both panels the field of view is 110x110 $\mu$m$^2$.

Figure 7. Measurements of 500 nm thick Fe thin film with a point like surface defect introduced by a needle punch. **Left panel:** optical image taken using the same optical system but with white light illumination. **Right panel:** The ODMR map representing the magnetic field changes over the field of view. In both panels the field of view is 110x110 $\mu$m$^2$. 
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Figure 8. Measurements of 500 nm thick Fe thin film with line defects on the surface introduced by an assembly knife. **Left panel:** optical image taken using the same optical system but with white light illumination. **Right panel:** The ODMR map representing the magnetic field changes over the field of view. In both panels the field of view is 110x110 µm².

Figure 9. Simulation representing a defect that is shaped similarly as the one in Fig. 7. **Left panel:** the initial shape of the simulated defect - the dark part is the cut-out, but the white part represents undamaged thin film. **Right panel:** the plot depicts the projection of the magnetic field on the NV axis 10 µm under the surface of the homogeneously magnetized layer. The magnetization direction was assumed to be colinear with the applied magnetic bias field and thus also the NV axis (there is an angle between the bias magnetic field and horizontal direction of the thin film).
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