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Diamond-Based Magnetic Widefield-Microscopy of Domain Patterns in Electrical Steel

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Summary
In this work, we demonstrate magnetic stray field imaging on a grain-oriented FeSi electrical steel micro sample after cyclic loading, using a home-built NV widefield microscope. The image is compared with a corresponding magnetic domain image of the same sample area acquired via Magneto Optical Kerr Effect (MOKE) microscopy.

Keywords: NV center, Widefield Microscopy, Magnetic Imaging, Non-Destructive Testing, Quantum Magnetometry

Introduction

Microscopic testing of magnetic materials has been utilized for the measurement of a variety of relevant properties for material sciences and engineering, some of which being residual stress, microstructure, or hardness. Recently, it has been shown [1] that measuring the stray field induced from the the inverse magneto-strictive, or Villari effect [2] can potentially be exploited to retrieve a magnetic signature of fatigue and early-stage crack initiation in micro-mechanical samples.

To find a magnetic signature in these measurements, it is essential to understand the evolution of the magnetic state in the sample, based on the structural and mechanical behavior of the material under different loading conditions. One route towards this, is by imaging the magnetic domain patterns, as can be done by Magneto-Optical Kerr Effect (MOKE) microscopy. Despite being an established method, it has the drawback of only providing information on the magnetization but not on the magnetic stray field [3].

Magnetic sensing techniques that utilize the nitrogen-vacancy (NV) center in diamond have been established in a wide variety of research, ranging from Bio- and solid-state magnetism over electronics to chemical analysis for magnetic, electric, thermal and strain sensing [4]. NV-center-based magnetic widefield microscopy, involves a dense layer of NV-centers rather than an individual one. In this way, a magnetic stray field distribution can be imaged over a wide area of hundreds of micrometers in only a few minutes of time, while still maintaining a diffraction optics spatial resolution. In this work, we demonstrate magnetic stray field imaging on a FeSi electrical steel micro sample using a home-built NV widefield microscope and compare the results with a corresponding magnetic domain image acquired prior and in-situ during cyclic mechanical loading of the sample via MOKE microscopy.

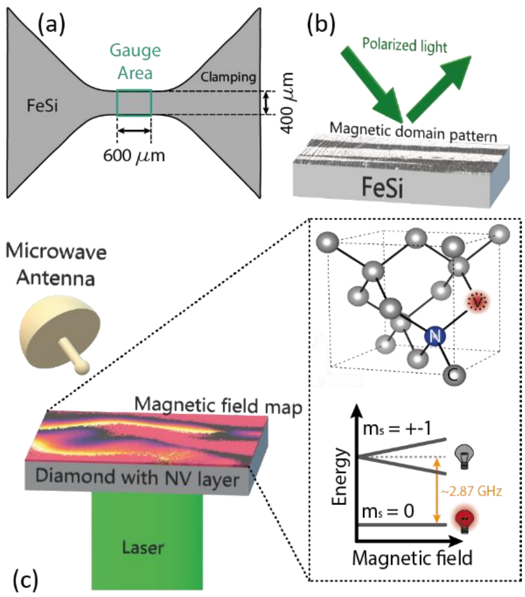

Fig. 1. (a) Schematic of a micro sample. The gauge area is highlighted (b) Schematic of a MOKE measurement. (c) Schematic of the widefield-NV setup. The inset shows the crystal structure and ground state energy levels of a single NV.

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Experiment

The micro samples were cut from a single grain of grain oriented FeSi electrical steel using wire erosion to minimize heat-affected zones along the edges. The specimens have a thickness of 220 µm, with a gauge section of 600x400 µm² which widens on both ends into a clamping area. A schematic of a micro sample with the geometry used here is shown in Fig. 1 (a). For the in-situ MOKE measurements, as schematically described in Fig. 1 (b), a micro-tensile apparatus, integrated into a Kerr microscope, was used. On the sample shown here, we performed a fatigue test in the elastic regime with a stress amplitude of a_S = 195 MPa and a load ratio of R = 0.1 at a frequency of f = 30 Hz. After that, the sample showed severe plastic deformation and the image in Fig. 2 (c) was taken at 0 MPa after 1651 cycles.

Wide-field NV microscopy was performed utilizing a dense layer of nitrogen-vacancy (NV) centers. The nitrogen doped diamond was home-grown by chemical vapor deposition (CVD) (Fig. 1 (c)). The sample was later electron irradiated and finally the NV-centers were formed in a thermal annealing procedure. Initialization and readout of the of the NV-center's spin state is performed optically with a green laser at λ = 532 nm. The magnetic resonance is obtained by tuning the frequency of a microwave irradiation from a nearby antenna. The red fluorescence is detected with a CMOS camera. This, together with the spin-dependent photo luminescence of the NV center allows for the measurement of the magnetic field from the field-dependent spin splitting of the $m_s = \pm 1$ states (see inset in Fig. 1 (c) and Fig. 2 (a)) via optically detected magnetic resonance (ODMR) spectroscopy.

Results

Fig. 2 (a) shows an averaged ODMR spectrum of multiple NV centers, as recorded for each pixel of the acquired image. The splitting of the dips is directly proportional to the magnetic field and is plotted for each pixel in (b). Fig. 2 (b) and (c) show the same area of a plastically deformed FeSi micro sample. The map of the ODMR splitting in (b) has a resolution of around 1 µm and took around 20 minutes recording time for an image size of 250x250 µm². The splitting is proportional to the distribution of the magnetic field generated due to the effective magnetic anisotropy of the sample, which is mainly a superposition of the crystal anisotropy of the material and the shape of the sample in the investigated area. The fatigue process and the plastic deformation of the material induce defects like dislocations,

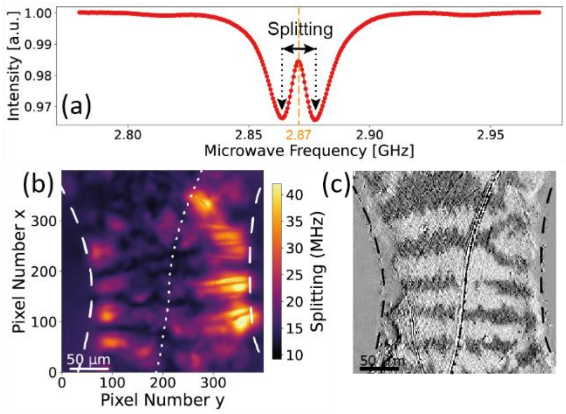

Fig. 2. (a) Averaged ODMR spectrum of the NV layer at one pixel. (b) Map of the magnetic field dependent splitting of the ODMR signal. (c) MOKE image of the same area as in (b).

slip and shear bands, inclusions, or precipitations, which can act as pinning cites for the magnetic domains [5]. This potentially causes the complex domain pattern displayed in (c), as recorded by MOKE microscopy. Comparison of the two images shows, that the magnetic field distribution in (b) and the domain pattern in (c) can be correlated reasonably well with each other. The lines in the images serve as guides to the eye and highlight characteristic features of the sample, such as the edges (dashed) and a scratch in the center (dotted), which can clearly be identified in both images. In conclusion, we have shown that NV-center based wide-field magnetometry as a quantum technology, can potentially be used in materials testing for imaging of the magnetic field distribution in micro samples. The NV map is clearly related to the underlying domain pattern imaged by MOKE microscopy but adds additional information about the magnetic stray field. This could be of advantage to find a magnetic fingerprint for early state fatigue damage in ferromagnetic materials.

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