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ABSTRACT

Magneto-optical techniques are key tools for the characterization of magnetic effects at a nanoscale. Here, we present the dark-field magneto-optical Kerr effect (DFMOKE), a technique we have recently developed for the characterization of three-dimensional magnetic nanostructures. We introduce the principles of DFMOKE, based on the separation of an incident beam into multiple reflected beams when focusing on a 3D nano-geometry. We show the key modifications needed in a standard focused MOKE magnetometer to perform these measurements. Finally, we showcase the power of this method by detecting the magnetic switching of a single tilted 3D nanowire, independently from the switching of a magnetic thin film that surrounds it. We obtain independent and simultaneous switching detection of the nanowire and the film for all nanowire dimensions investigated, allowing us to estimate a magnetic sensitivity of 7×10^{-15} A m^2 for DFMOKE in the setup used. We conclude the article by providing perspectives of future avenues where DFMOKE can be a very powerful characterization tool in the future investigations of 3D magnetic nanostructures.

INTRODUCTION

Three-dimensional nanomagnetism is an emerging research field devoted to the study of new nanostructures with 3D magnetic configurations, i.e., the magnetization vector in these systems is not confined to the substrate plane but extends along the whole space.[1–3] The leap to 3D can be achieved by different strategies, including the fabrication of nonplanar objects with complex geometries and nanometric dimensions,[4–6] access to complex magnetic states that emerge in bulk systems,[7,8] or the growth of multi-layered thin film systems with a large number of layers, where magnetic interfacial and bulk energies are designed differently than in the standard heterostructures.[9] The interest behind this new field lies not only in their potential to create disruptive technologies, e.g., devices with ultrahigh storage density and massive interconnectivity but also due to their potential for scientific discoveries, including new spin textures, magnetic dynamics, and topological properties.

Moving to 3D seems like a natural evolution of nanomagnetism, as has occurred in other areas of nanotechnology.[10,11] However, the leap from 2D to 3D is challenging and requires new fabrication and characterization techniques to be developed.[24] These may be currently existing techniques, which have been used to fabricate or probe planar systems and need to be adapted, or completely new techniques that are designed from the start with 3D magnetic states and 3D geometries in mind. Here, we discuss the dark-field magneto-optical Kerr effect (DFMOKE), a magnetic characterization technique we have recently developed[12,13] to optically investigate 3D magnetic nanostructures. Being a lab-based technique, DFMOKE complements well other techniques widely used in 3D nanomagnetism, such as x-ray and electron microscopy,[5,14] available at large facilities which may be difficult to access on a regular basis. It is also a complementary method to other lab-based techniques such as magneto-electrical and spin-transport measurements.[15,16]

The magneto-optical Kerr effect (MOKE) has been an instrumental tool for the development of multiple areas in nanomagnetism,[17] thanks to a combination of assets, including ultrahigh magnetic sensitivity, ability to perform single nanoscale object magnetometry,[18] sensitivity to the three components of the magnetization vector,[19–21] ability to carry out magnetic Kerr microscopy,[22] and high time-resolution capabilities.[23] First reported by John Kerr in 1877, MOKE describes the change of the polarization state of light when reflected from a magnetic surface,[17] being today one of the most direct and versatile ways for the surface characterization of thin films, multilayers, bulk materials, and planar magnetic devices. The DFMOKE technique refers to an adaptation of MOKE to investigate 3D magnetic nanostructures with nonplanar geometries. In DFMOKE, a single focused laser beam exploits the geometry of a 3D nano-object to separate and analyze the Kerr signal resulting from specular reflections taking place at different magnetic planes of the nonplanar nanostructure.

In what follows, we present the DFMOKE technique, explain its physical basis, and show how a standard optical setup can be adapted to implement it. Then, we employ DFMOKE to perform 3D nano-magnetometry experiments, where the magnetic reversal of a thin film and a single 3D nanowire grown on top are investigated under external magnetic fields. We show how the DFMOKE technique allows to detect, independently and simultaneously, the magnetic switching of both systems. From these experiments, we also infer a lower bound for the magnetic sensitivity of the technique, which is found to be comparable to the standard focused MOKE magnetometry. These results demonstrate the power of DFMOKE for studies in 3D nanomagnetism.

METHOD

The virtues of dark-field microscopy were already recognized more than a century ago,[25] when the advantages of lighting a sample off-axis were exploited to reveal objects like white blood cells, which were not visible at that time under standard imaging conditions. Drawing inspiration from dark-field microscopy, we recently reported the DFMOKE method to probe 3D magnetic nanostructures: The technique has a similar principle to the one for dark-field microscopy, but we employ, instead, a modified focused MOKE magnetometer, with the illumination field consisting of a laser beam focused on a 3D nanostructure down to a spot of a few microns in width. The reflected beam is collected by several optical paths, where the change in polarization of the light upon reflection with the magnetic object due to the Kerr effect is detected.

We illustrate the technique by using the example of a nonplanar 300 nm wide, 2 μm long, nanowire, grown at a fixed 30° angle with respect to the substrate plane [see Figs. 1(a) and 1(b)]. The 3D nanowire is covered by a 50 nm thick Permalloy film, the same as the substrate, following a two-step nanofabrication method comprising two steps: (1) focused electron beam induced deposition (3D nano-printing) of a carbonaceous material which serves as a 3D scaffold and (2) thermal evaporation of a magnetic film.[4,13] Given the different normal vector directions of a 3D nanostructure and the substrate, specular reflection of the injected light gives rise to two independent reflected beams, the *bright-field* and *dark-field* light paths [see Fig. 1(c)]. The nonplanar geometry of the 3D nanowire is thus exploited to separate a fraction of the reflected light (dark field) from most of the reflected light (bright field) coming from the substrate. In a first approximation, and for a laser spot larger than the whole nanowire, the ratio R between intensities detected by both optical paths is equal to $R = A_{NW}/(A_L - A_{NW})$, with A_{NW} being the area of the top surface of the nanowire and A_L the area of the laser beam on the substrate.

Unlike standard dark-field microscopy, which normally relies on the random scattering of light, DFMOKE of 3D nanostructures is based on an accurate matching of geometries of the sample and the optical setup, i.e., between the incoming laser direction and injecting optics, the nanostructure position and spatial orientation, and the location of the detection optics and photodetectors. A natural way to implement this method consists on implementing an extra optical path for detection, to be added to the standard one which captures the bright-field light. This extra dark-field detection

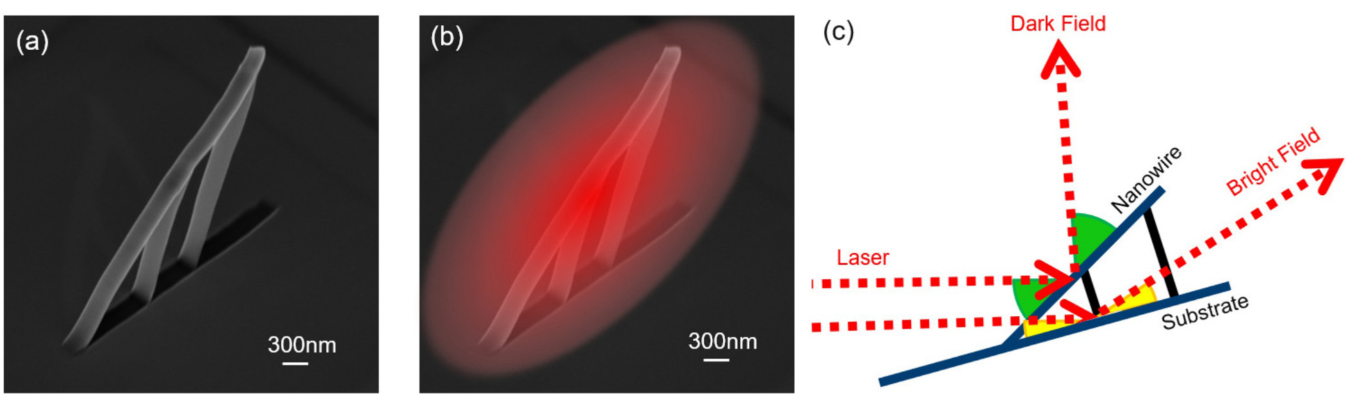

FIG. 1. DFMOKE applied to a nanowire and thin film. (a) Scanning electron microscopy micrography of the nanowire and film after evaporation of a 50 nm thick permalloy thin film. (b) Illumination of the system with a focused light beam (not to scale). (c) Principles of DFMOKE applied to this system. The focused laser beam is reflected at two different angles due to the two different planes subtended by the magnetic thin film (bright-field reflected beam) and nanowire (dark-field reflected beam). Reproduced with permission from D. Sanz-Hernández, "Fabrication and characterization of three-dimensional magnetic nanostructures," Ph.D. thesis (University of Cambridge, 2019).

path probes the light reflected from a 3D nanostructure, which would be otherwise lost. Besides being precisely matched to the reflection planes of the nanostructure under study, the dark-field detection path is identical to the detection path employed in a standard MOKE magnetometer, facilitating independent detection and polarization analysis. The tensorial dependence between electric field and magnetization is, thus, fully valid and remains unaltered from a 2D case, although care must be taken when considering what type (longitudinal, transverse, polar, or a mixture of them) of the Kerr effect is probed at each detector. The sensitivity will, in general, differ from each of the scattered planes considered due to the angular dependence of different Kerr effects for each material.[22] In the examples explained in this article, we have used Permalloy, where the magnetic states are normally dominated by shape anisotropy, avoiding unnecessary complications in the interpretation of the results which could result from other magnetic energies playing a role in the magnetic state of the system. Polarization analysis of signals at different angles has also been employed with success for films with varying refractive index[26] in transmission configuration (i.e., Faraday effect) and in diffraction-MOKE of nano-element arrays,[27] where different diffraction orders emerge at particular observation angles.

Due to the 3D geometrical character of the technique, an in-depth geometrical characterization of the sample in the form of, e.g., scanning electron microscopy before the DFMOKE experiments, is necessary. This is key to accurately match the optical setup and the sample geometry. A high reproducibility in the sample 3D geometry production is also imperative to avoid incompatibilities with the optics arrangement. In our case, this becomes possible, thanks to our recent advances in the 3D nano-printing methods based on focused electron beam induced deposition (FEBID).[4] Further studies are required in order to determine the exact degree of precision required for the matching of the nanostructure angle and the optical path, but preliminary evaluations indicate that deviations up to 10° can be tolerated without the need for major realignment.[12] For the case of curved structures like a nanocap, it is expected that a DF MOKE signal will only be collected from those surfaces of the structure forming the correct angle to the optical path.

Now we show how to adapt a focused laser MOKE magnetometer setup for this purpose. We specifically describe the adaptation of a NanoMOKE2 system by Durham MagnetoOptics, a setup normally used for the characterization of thin films and 2D nanostructures. The standard system for planar optical magnetometry and the adapted version for DFMOKE measurements are schematically shown in Figs. 2(a) and 2(b), respectively. In both systems, a linearly polarized laser is focused on the sample (5 μm spot width), and changes in polarization upon reflection are detected by

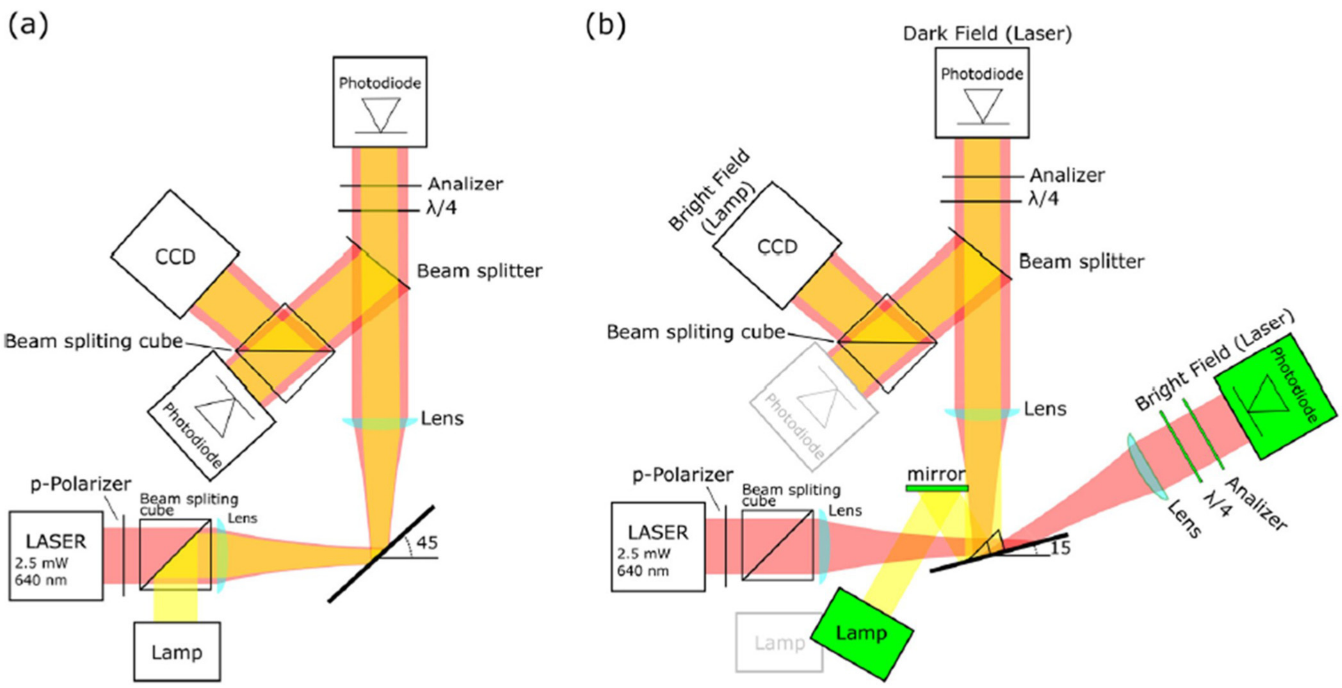

FIG. 2. A focused MOKE magnetometer and its adaption to perform DFMOKE measurements. (a) Standard MOKE with incoming and reflected laser (red) at 45° with respect to the sample. Incident light is p-polarized and analyzed by a combination of a quarter-wave plate and an analyzer. White light (green) is introduced in the optical path to image the sample and find nanostructures on its surface. (b) Modified MOKE to perform DFMOKE. The main changes (green) are (i) a new substrate angle, modified to take into account the geometry of the nanostructure, (ii) an extra optical path to capture and analyze the reflected light from the substrate, and (iii) a modified light source including a mirror, to obtain a clear image of the sample and the 3D nanostructure prior to magnetometry measurements. Reproduced with permission from Sanz-Hernández *et al.*, ACS Nano **11**, 11066–11073 (2017). Copyright 2017 Author(s), licensed under a Creative Commons Attribution (CC BY) license.

employing a combination of a retardation plate and an analyzing polarizer. In addition, for sample alignment and nanostructure location, white light (yellow color in Fig. 2) is injected into the laser path and projected onto a CCD camera. A two-axes motor stage to perform reflectivity maps is also available.[18] This type of system is capable of performing highly sensitive magnetometry measurements in planar systems. In particular, provided that enough distance exists between neighbor nanostructures, magnetometry of single nanowires has been demonstrated,[18] something widely exploited, e.g., for studies in domain wall devices.[28]

Figure 2(b) shows three key modifications applied to the optical setup in order to perform DFMOKE magnetometry of 3D nanostructures (highlighted using green color in Fig. 2). First, the angle formed by the sample plane and the incoming laser beam was originally set to 45° to provide good sensitivity to both longitudinal and polar signals.[22] Instead, in the DFMOKE setup, this is changed to 15°, given that the 3D nanostructure angle formed with the substrate is 30°. In this new configuration, the 3D nanostructure reflects a small fraction of the laser into the original detection path, which now becomes the dark-field path. A second modification consists of adding an extra detection path at 15° with respect to the substrate plane. This set of optics is used to capture and analyze the main laser reflection coming from the substrate, becoming the bright-field detection optical path [see detector marked in green in Fig. 2(b)].

Third, to perform proper sample alignment and locate the 3D nanostructures, the light injected into the optical path must be reflected from the substrate to reach the CCD. This could be achieved by either moving the camera to the bright-field path or keeping it in the dark-field path and modifying the light source. Here, we choose the second option, where we add an extra mirror to match the injected light angle with the substrate plane and the CCD dark-field path.

Complementing the three modifications explained above, a fourth main modification consists of extending the directions to which a controlled magnetic field can be applied. Here, we achieve a three-axis electromagnet by combining a quadrupole and a single-pole electromagnet that is oriented perpendicularly to the plane of the quadrupole. 3D magnetic Permalloy nanostructures

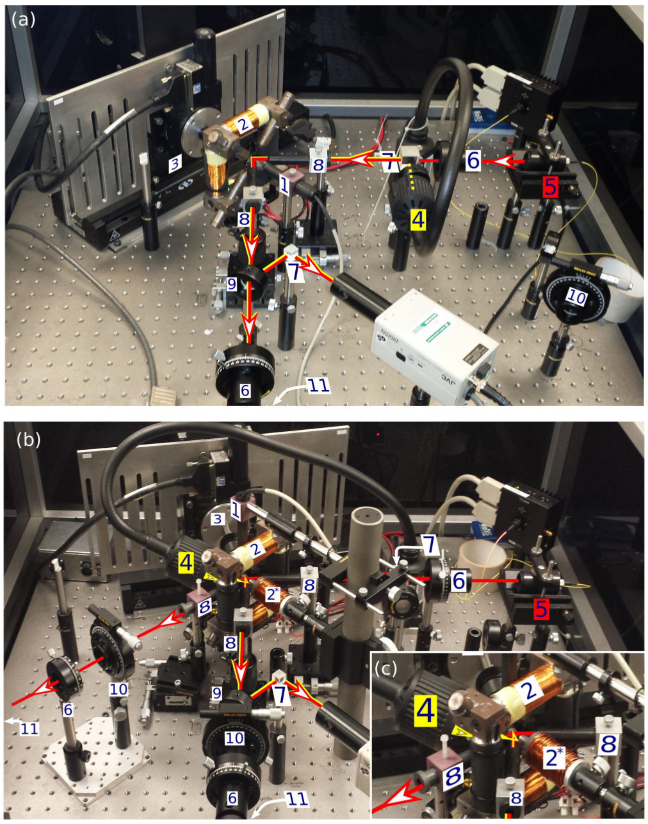

FIG. 3. Adaptation of a focused MOKE setup to perform DFMOKE experiments. (a) Standard MOKE setup, including one reflected optical path and a quadrupole electromagnet. The experiment of planar systems is performed at 45° incident and reflected angles. (b) Modified DFMOKE setup, with two reflection optical paths (bright field at 30° and dark field at 45°), and an additional coil for the application of the 3D magnetic fields. (c) The region around the sample, where the tubes holding the lenses, quadrupole, additional coil, and sample holder are observed. The components are labeled as (1) Hall probe, (2), electromagnet, (3) translational stage, (4) lamp, (5) laser, (6) polarizer, (7) beam splitter cube, (8) lens tube, (9) beam splitter, (10) waveplate, and (11) photodetector. Reproduced with permission from D. Sanz-Hernández, "Fabrication and characterization of three-dimensional magnetic nanostructures," Ph.D. thesis (University of Cambridge, 2019).

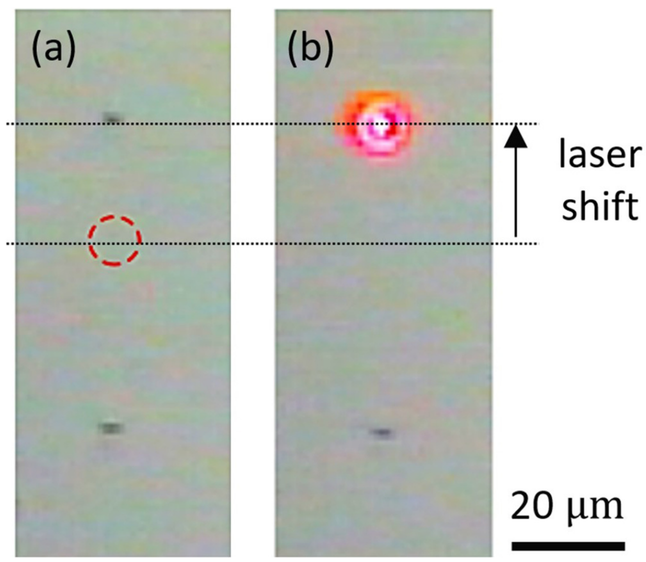

FIG. 4. Optical image of two 3D nanowires grown on top of the substrate. (a) The laser spot is located in between the two nanowires, resulting in no reflection from the beam in the CCD located on the dark-field optical path. (b) The laser beam is located on top of the 3D nanowire, producing a clear reflection in the image. Reproduced with permission from Sanz-Hernández *et al.*, ACS Nano **11**, 11066–11073 (2017). Copyright 2017 Author(s), licensed under a Creative Commons Attribution (CC BY) license.

are expected to be susceptible to moderate magnetic fields along any direction of space. The introduction of a vector electromagnet that is able to apply fields in any arbitrary direction is thus important to fully understand all possible magnetic reversal mechanisms of the system and exploit its 3D nature.[1,12]

Real-life images of these two experimental setups, for standard MOKE and DFMOKE, are shown in Figs. 3(a) and 3(b), respectively. The main components in both cases are labeled and explained in the figure caption. Figure 3(c) shows a close-up view of the final electromagnet assembly and the high focusing power lenses used to focus the laser on the sample and collect the two specular reflections. The lenses are held at the end of the tubes to position them at the correct operational distance to the sample. The pair of images in Fig. 3 illustrates the additional complexity of the DFMOKE setup, where an extra optical path and a 3D electromagnet are added, which requires the integration of many components within a small volume around the sample. For DFMOKE, the setup alignment is also significantly more challenging, since the

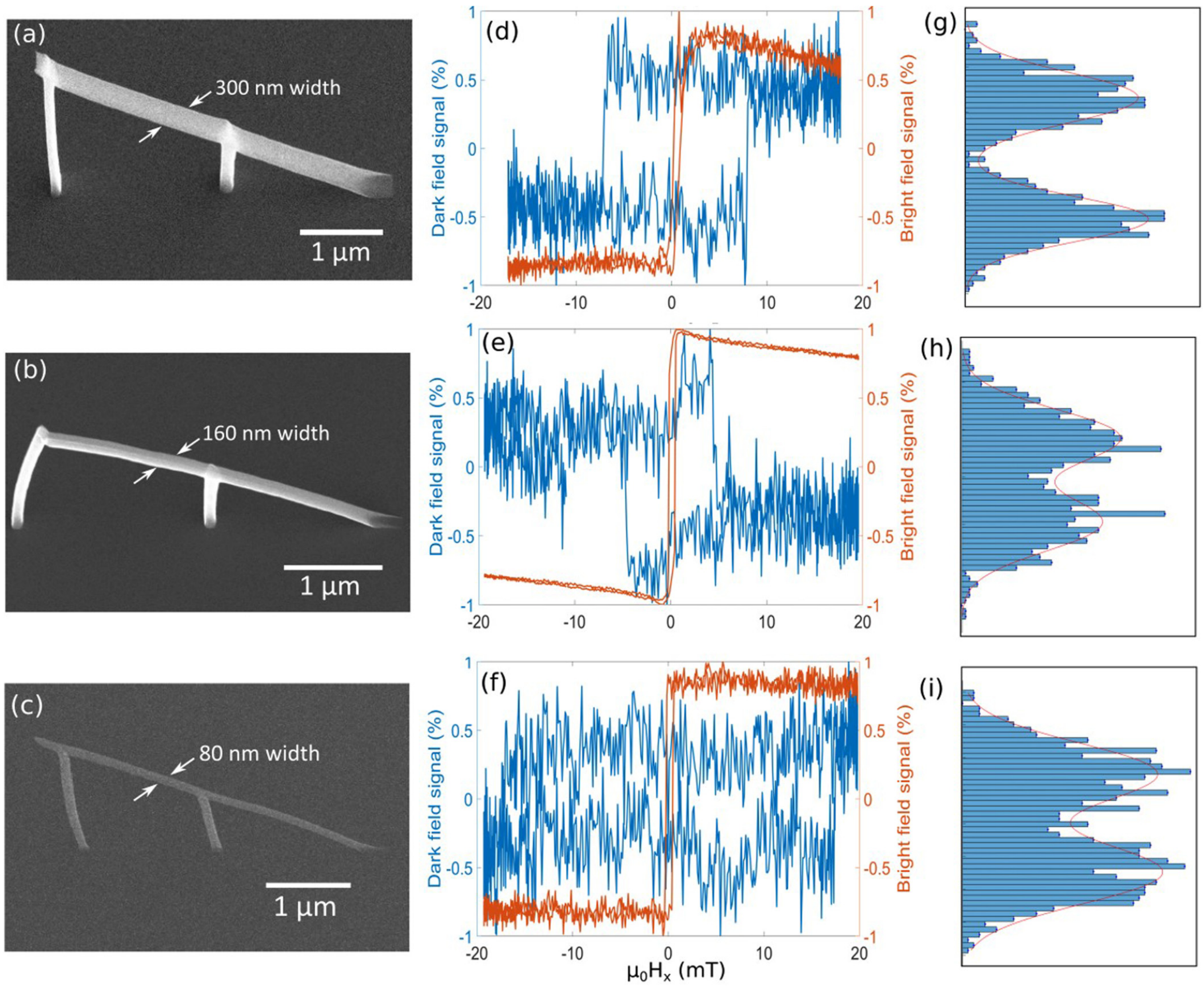

FIG. 5. DFMOKE magnetometry experiments of a single 3D nanowire on top of a thin film. (a)–(c) SEM micrographs at 45° observation angle of the nanostructures under investigation, all forming 30° with respect to the substrate and with different widths: 300 (a), 150 (b), and 80 nm(c). (d)–(f) MOKE signals of the thin film and 3D nanowire as a function of the magnetic field applied along the long axis of the nanowire, showing independent switching for both systems. The two signals are simultaneously collected using bright field (film) and dark field (nanowire) optical paths and detectors. (g)–(i) Histogram of dark-field signals illustrating signal-to-noise ratio for the different nanostructure widths.

three optical paths need to match the position of the nanostructure with better accuracy than the laser spot width.

After describing the optical setup used for DFMOKE, we show in the next section, the adequacy of the technique to probe 3D nanostructures.

DISCUSSION

Using the optical setup described above, we show now the DFMOKE experiments devoted to the characterization of a 3D nanowire such as the one in Fig. 1, forming a 30° angle with respect to a continuous magnetic thin film. This is a particularly challenging system to be characterized magnetically due to several reasons. First, the effective area of a nanowire probed by focused light is orders of magnitude smaller than for a thin film and results in a tiny fraction of light reflected with respect to the film, given approximately by the ratio R define above. For example, for the $2\,\mu$m long, 300 nm wide, and 50 nm thick nanowire of Fig. 1, and this setup with a beam laser diameter of $5\,\mu$m, R = 0.03. Second, there is no lateral spatial separation between the two, with the magnetic thin film surrounding the 3D wire. Finally, the magnetic moment of the nanowire under investigation is small enough (2×10^{-14} Am^2) to require the use of a very sensitive magnetometry technique to detect it. From these considerations, it is evident that either bulk magnetometry techniques or standard focused MOKE magnetometry are not suitable since the total detected signal would be fully dominated by the film. However, with the wire and film forming two different well-defined magnetic planes, they constitute a perfect example to test the capabilities of DFMOKE.

Before starting with magnetometry measurements, the first step consists of accurately positioning the laser on top of the 3D nanostructure, by making use of the CCD camera positioned at the dark-field optical path. Figure 4 shows two optical images acquired, where the beam has been attenuated with a neutral density filter to avoid saturating the camera, which reveals two single 3D nanowires far apart from each other (see black small regions along the vertical direction). In Fig. 4(a), the laser beam is positioned far away from any of the two nanowires, at a location marked by the red dashed circle. Since the laser beam is reflected by the substrate only on that location, no light reflected from the nanowire is observed in the camera. This contrasts with Fig. 4(b), where the stage has been positioned at the location where the laser beam is located on top of one of the 3D nanowires, which results in a clear image of the laser reflection. This experiment constitutes an excellent visual confirmation of the successful implementation of the dark-field technique since no laser intensity is detected on the CCD that is located at the dark-field path, unless a structure with the correct 3D geometry is present to deflect the incoming light.

Once the laser beam is accurately positioned on top of a single 3D nanowire, the laser attenuation filter is removed, the white light is switched off, and an oscillating magnetic field parallel to the long axis of the nanowire is applied. Under these conditions, the MOKE signal from both reflected beams is obtained by analyzing the change of ellipticity of light, using a combination of quarter waveplate and analyzing polarizer. Figure 5 shows examples of hysteresis loops measured at the same time for both dark-field and bright-field detectors. Permalloy is a soft magnetic material with an in-plane magnetic configuration. The signal collected by this optical arrangement is, thus, dominated by the longitudinal Kerr effect in both cases. Results for measurements on single 3D nanowires with three different widths (300, 160, and 80 nm) are included. SEM side view images of the 3D nanowires accompany the MOKE results. In all cases shown here, very distinct loops are obtained for both detectors: In the bright-field detector, a low coercivity loop corresponding to the planar thin film is observed. A much higher coercivity is observed for the signal recorded in the dark-field detector, where the 3D nanowire is measured. These results, in agreement with our previous works,[12,13] show how the two recorded signals do not show common switching field values. This constitutes a proof of how DFMOKE can independently and simultaneously probe different planes of a 3D nanomagnetic system, in this case, a thin film and a 3D nanowire with a constant slope grown on top of it. Despite dealing with 3D nanoscale geometries, this result also demonstrates how DFMOKE using focused MOKE magnetometers is sensitive to very low magnetic moments. In this case, the magnetic moment probed corresponding to the narrowest wire is estimated to be equal to 7×10^{-15} Am^2, comparable to the best values previously reported for focused MOKE of planar objects.[18] In fact, the dark-field detection channel may benefit from a reduction in background noise from light reflected from the substrate, an important factor affecting MOKE sensitivity in planar nanostructures, particularly, when these are positioned close to patterned pads. We also attempted to measure narrower nanowires with a width approaching 40 nm (not shown here) with no conclusive results, indicating a sensitivity not better than 5×10^{-15} Am^2 for the DFMOKE technique in this setup.

CONCLUSIONS

We have presented a detailed description of dark-field MOKE magnetometry, a powerful technique for the investigation of magnetic nanostructures with 3D geometries. We show how the technique can be implemented by appropriate modifications of a standard focused MOKE magnetometer. We show how the technique can be applied to perform single-nanostructure 3D magnetometry, by measuring the reversal of 3D nanowires with different widths, fabricated at a constant slope of 30° with respect to a thin film. Future studies aiming to develop the technique further might focus on determining the minimum relative angle between magnetic planes necessary to obtain independent switching in different detectors, and its relationship with the numerical aperture of the lenses and with diffuse scattering taking place in nonplanar objects.[29] This could be key, for instance, in studies devoted to probe the magnetic switching of complex 3D magnetic lattices such as 3D artificial spin ice elements.[30,31]

Another important aspect of the setup presented is its ability to apply complex magnetic field sequences along any spatial direction. We have previously reported how systematic studies as a function of 3D field scans can be used to determine the nucleation, propagation, and depinning fields of 3D domain wall conduits, allowing us to perform advanced experiments where domain walls were controllably injected from the substrate plane into nonplanar conduits by different reversal mechanisms. This type of advanced magnetic programming of the 3D magnetic nanostructures under complex magnetic fields can be of great use for applications in vector magnetic sensing[32–34] and nanorobotics.

In this work, we also show a very high magnetic sensitivity of the technique, capable of probing single 3D nanostructures with magnetic moments below 10^{-14} Am^2. Further developments may include the exploitation of diffraction-limited spatial resolution of Kerr microscopy[22] for the magnetic imaging of the 3D nanostructures, and the high performance of time-resolved MOKE setups[23,35] to probe magnetic dynamics in 3D.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Dédalo Sanz-Hernández: Conceptualization (equal); Data curation (equal); Formal analysis (equal); Investigation (equal); Methodology (equal); Software (equal); Supervision (equal); Validation (equal); Visualization (equal); Writing – review & editing (equal). **Luka Skoric:** Data curation (equal); Formal analysis (equal); Investigation (equal); Methodology (equal); Software (equal); Validation (equal); Visualization (equal); Writing – review & editing (equal). **Miguel Ángel Cascales-Sandoval:** Investigation (equal); Methodology (equal); Software (equal); Validation (equal); Visualization (equal); Writing – review & editing (equal). **Amalio Fernández-Pacheco:** Conceptualization (equal); Formal analysis (equal); Funding acquisition (lead); Investigation (equal); Methodology (equal); Project administration (lead); Resources (lead); Supervision (equal); Writing – original draft (lead); Writing – review & editing (equal).

DATA AVAILABILITY

The details shown in this work provide the necessary information to implement a focused MOKE system for DFMOKE magnetometry. The data that support the findings of this study are openly available in Digital.CSIC at https://digital.csic.es/, Ref. 36.

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