



# Structural modifications of thin magnetic Permalloy films induced by ion implantation and thermal annealing: A comparison

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## Abstract

We report the structural properties of thin magnetic Permalloy films treated by two different methods: broad beam Ga<sup>+</sup> ion implantation at an energy of 30 keV as well as annealing at different temperatures under ultrahigh vacuum. Transmission electron microscopy imaging and X-ray diffraction measurements have demonstrated that both ion implantation and annealing (above 300 °C) lead to further material crystallization and crystallite growth. Whereas annealing (above 400 °C) leads to a strain-free state with an almost constant lattice parameter and to a further enhancement of the initial (111) texture, ion beam implantation boosts the growth of small, arbitrarily oriented crystallites and leads to a linear increase in the lattice parameter, introducing microstrain into the sample. The observed decrease in the saturation magnetization for the implanted samples is mainly attributed to the presence of the non-magnetic Ga atoms incorporated in the Permalloy film itself. The increase in the saturation magnetization for the samples annealed at temperatures above 500 °C is explained by an arising dewetting effect since no ordered Ni<sub>3</sub>Fe phase was detected with anomalous X-ray diffraction. © 2014 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

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## 1. Introduction

In the last decades ion beam implantation became an important tool for structural modifications of a broad range of different materials. Of particular interest is the production of magnetic nanostructures widely used for magnetic recording media [1–4], magnetoresistive sensors [1,5,6], magnetic random access memories [7–9] and other applications [10,11]. For nanoscale patterning, focused ion beam (FIB) technology has proven to be an efficient tool compared to standard broad-beam ion implantation. The number of technological steps is dramatically

decreased, since there is no longer any need for implantation masks [12–18]. Many investigations of the influence of FIB Ga<sup>+</sup> ion implantation on the structural and magnetic properties of thin magnetic Permalloy films have been reported [19–23]. The choice of Ga is based on its liquid state at room temperature, and is, therefore, mainly used in commercial FIB systems. Recent publications show that FIB implantation leads to a significant crystallite growth [20–23], texturing towards the (111)-direction [23] and to an increase in the lattice parameter [21,23]. Furthermore, out-of-plane magneto-optic Kerr effect (MOKE) measurements demonstrate a reduction in the saturation magnetization with increasing ion fluence [20,22,23], which is explained by the incorporation of non-magnetic Ga atoms at the lattice sites of the Permalloy film [19,23]. It was always assumed that even though there is a difference in the beam current densities of 7 orders of magnitude, FIB implantation is equivalent to broad-beam ion implantation

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and no relevant annealing effects, which can additionally modify the material, occur. This assumption can be justified by MOKE measurements [24], which are demonstrated in Fig. 1(a). It can be clearly seen that the saturation magnetization decreases with increasing ion fluences for the samples implanted with a broad-beam ion implanter (black) as well as for the samples implanted with a FIB system (red) according to the same linear law. The results for the samples implanted with a FIB system are taken from Roshchupkina et al. [23], and broad-beam implantation details can be found in the experimental details of this manuscript. For comparison, the dependence of the saturation magnetization ( $M_S$ ) on the annealing temperature is demonstrated in Fig. 1(b) and it rises at temperatures above 500 °C. Therefore, a strong temperature annealing effect influencing crystalline structure and/or morphology during FIB implantation can be completely excluded. In several previous works it was demonstrated that annealing also induces a grain growth [25–32], and a material texturing towards the (111)-direction [29]. The rise of the saturation magnetization was then explained by the change of the surface morphology [27,33], neglecting a possible ordering of the system during heat treatment. The aim of this work is to study the difference in structural changes induced by broad-beam  $\text{Ga}^+$  ion implantation and by annealing in a ultrahigh-vacuum (UHV) chamber. Broad-beam ion implantation was used instead of FIB implantation since samples implanted with broad beams are larger and easier to prepare than those produced by a FIB. In general, many investigations have been reported of the influence of implantation using different ion types [34,35] and of annealing [25–33] on the structural and magnetic properties of Permalloy thin films. However, no systematic study with comparable samples has been performed (i.e. using Permalloy films with the same film thickness and produced by the same technique under the same conditions). Moreover, possible structural phase changes of Permalloy at high annealing temperatures need to be accurately investigated.

## 2. Experimental details

A 50 nm thick Permalloy ( $\text{Ni}_{81}\text{Fe}_{19}$ ) film was grown on a 1  $\mu\text{m}$   $\text{SiO}_2$  buffer layer based on a (100)-oriented Si wafer

using magnetron sputtering at room temperature. The broad-beam ion implantation, i.e. standard ion implantation, parameters were chosen as close as possible to the  $\text{Ga}^+$  ion beam parameters of the FIB system (as in Ref. [23]). The samples were loaded into a DANFYSIK 1050 Low Energy Ion Implanter with a base pressure of  $1 \times 10^{-6}$  mbar [36,37]. Ion implantation was then performed at room temperature using a 30 keV  $\text{Ga}^+$  ion beam with varying ion fluences, and a beam direction perpendicular to the sample surface. To achieve homogeneous ion implantation the beam was swept over the entire sample at two different frequencies of around 1 kHz in the  $x$  and  $y$  directions using an electrostatic sweeping system. With a beam diameter of 3–5 mm the beam current was in the range 2.5–3  $\mu\text{A}$  [37]. For the second part of the study a series of Permalloy films was annealed in an UHV chamber at temperatures up to 800 °C and at a base pressure below  $1 \times 10^{-6}$  mbar. The X-ray diffraction (XRD) measurements were carried out using laboratory setups with  $\text{Cu } K_\alpha$  radiation. A Bruker AXS Vantec position-sensitive detector (PSD) or a point detector were used to record the scattered intensity. For both detectors the detected energy range was suitably chosen to suppress the fluorescence signal from the iron, resulting in a low background signal. Additionally, the structure of the Permalloy films was investigated using high-resolution cross-sectional transmission electron microscopy (XTEM) with a FEI Titan 80–300 microscope equipped with a CEOS image corrector. Possible phase transformations were studied using anomalous scattering measurements, which were performed at the Rossendorf Beamline (ROBL) at ESRF (BM20) with an energy resolution better than 2 eV [37–39]. The magnetic properties were characterized using MOKE magnetometry in polar geometry.

## 3. Results and discussion

Fig. 2(a)–(c) show bright-field XTEM images of an as-deposited sample, an ion-implanted sample using an ion fluence of  $3.75 \times 10^{16}$  ions  $\text{cm}^{-2}$ , as well as a sample annealed at 700 °C, respectively. In addition to a crystallite growth induced by ion implantation and by annealing, an increased surface roughness can be clearly observed. In contrast to the as-deposited film (Fig. 2(d)), the diffraction

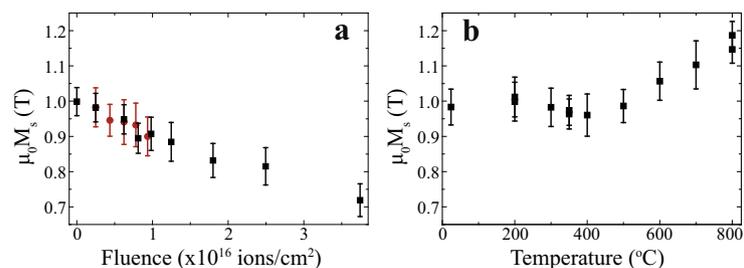


Fig. 1. Saturation magnetization dependence on the ion fluence (a). Red points represent results taken from Ref. [23] for the samples implanted with a FIB system. Saturation magnetization dependence on the annealing temperature (b). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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