Selective etching of epitaxial MnAs films on GaAs(001): Influence of structure and strain

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Strain in epitaxial MnAs thin films on GaAs(001) substrates plays an important role in the coupled magnetostructural phase transition. As a result of strain, the phase transition from the ferromagnetic α phase to the paramagnetic β phase proceeds over a wide temperature range and the coexisting phases form a periodic stripe array. Employing suitable wet chemical etchants, the two MnAs phases can be etched selectively. Perpendicular to the α-β-stripe structure, the built-up strain relaxes in the course of the etching process by the formation of cracks. The combination of both strain relaxation mechanisms allows for the defined patterning of two-dimensional arrays of nanomagnets. Through micromagnetic investigations, it is possible to identify the location of α- and β-MnAs which helps to clarify two major aspects of the etching process. First, it is possible to determine the etch rates of α- and β-MnAs and follow the complex interplay of strain and phase composition during the etching process. Second, as strain reflects itself in a shifted phase-transition temperature, temperature-dependent micromagnetic studies allow to determine the strain environment of the cracks. © *2005 American Institute of Physics*. [DOI: 10.1063/1.1954888]

I. INTRODUCTION

Epitaxially grown thin-film heterostructures, in which there is a lattice mismatch between the substrate and the film, are commonly used for semiconductor devices. The resulting strain, often a problem for heteroepitaxy, can be the basis for self-organized nanofabrication.[1] Recently, we demonstrated how strain in a ferromagnet-semiconductor hybrid system—MnAs on GaAs(001)—can be the basis of self-organized patterning and deep etching.[2–5]

Here, we present a detailed investigation of the etching process. As the etch rates for α- and β-MnAs turn out to be remarkably different, and as the phase composition in the coexistence regime is a function of the temperature, the temperature dependence of the etching process was investigated in detail. Furthermore, the strain does not only reflect itself in the formation of coexisting phases (that have different etch rates), it also leads to the formation of cracks. The formation of cracks and their influence on the etching process will be discussed. As strain affects directly the magnetic properties of MnAs, useful information can be obtained by studying the micromagnetic properties in the vicinity of cracks.

II. EPITAXIAL MNAS FILMS ON GAAS(001)

A. Sample preparation and properties

MnAs is a ferromagnetic metal which has proven to be of interest for spin injection[6] and magnetologic[7] applications. MnAs is ferromagnetic in its hexagonal α phase with alternating hexagonal planes of Mn and As atoms (cf. Fig. 1). It undergoes a first-order magnetostructural phase transition to the paramagnetic β phase (orthorhombic) at about 40 °C.[8–10] This transformation involves a discontinuous change of the volume of the unit cell of about 2%.[11]

MnAs films can be grown epitaxially on GaAs at low temperatures of about 250 °C.[12–14] On GaAs(001), MnAs grows with the MnAs($\bar{1}100$) surface parallel to the GaAs(001) surface, and MnAs[0001] parallel to GaAs[$1\bar{1}0$], i.e., the hexagonal prism plane is parallel to the cubic plane. This break of symmetry at the interface results in a misfit that strongly depends on the in-plane lattice direction and leads to an unusual misfit accommodation process[15,16] that reduces the natural lattice misfit along GaAs[110] to about 5%.[17] The lattice mismatch for the GaAs[$\bar{1}10$] direction, i.e., between MnAs{$1\bar{1}20$} and GaAs{110} planes, reaches only about 7.5%.

As a result of strain in MnAs on GaAs(001) in the GaAs[110] direction both the ferromagnetic α phase and the

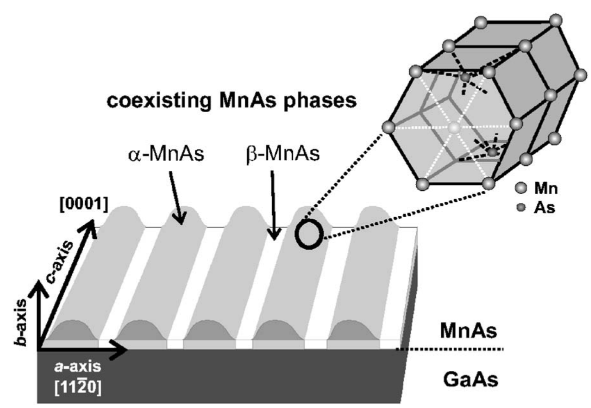

FIG. 1. Sketch of the stripe structure of the strain-stabilized coexisting α- and β-MnAs phases on GaAs(001) in the temperature range from 10 to 40 °C. The in-plane a axis is the easy axis of magnetization and the magnetic hard axis is along the in-plane c-axis direction (MnAs[0001]). On the top right, a detailed sketch of the hexagonal α-MnAs cell is shown.

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paramagnetic β phase coexist over a temperature range of 10–40 °C.[18–20] The two phases exhibit a self-organized array of stripes elongated along the c axis of MnAs ([0001] direction),[21–24] as illustrated in Fig. 1. Magnetic measurements reveal that MnAs films on GaAs(001) exhibit a strong uniaxial anisotropy with the in-plane a axis being the easy axis of magnetization and the in-plane c axis the hard axis of magnetization.[25] A sketch of the coexisting α and β phases of MnAs on GaAs(001) and the crystallographic orientation of α–MnAs are shown in Fig. 1.

B. Strain in MnAs/GaAs(001)

MnAs films on GaAs(001) are found to be anisotropically strained. Strain in this system may have three interdependent origins, namely, (I) the crystallographic lattice mismatch between MnAs and GaAs as discussed above, (II) the thermal-expansion mismatch between the two crystals, and (III) the first-order phase transition with the associated volume change. (II) Above the ferromagnetic-to-paramagnetic phase-transition temperature, the thermal-expansion coefficients of MnAs in the a-axis and c-axis directions are similar and about one order of magnitude larger than those of GaAs.[11,26] Below the phase-transition temperature, the thermal-expansion coefficient along the a axis changes sign, meaning that the lattice expands upon cooling.[11,26] Therefore, cooling from the growth temperature to room temperature gives rise to tensile strain in the MnAs film along the c-axis direction. In the perpendicular a-axis direction, the strain is tensile above the phase-transition temperature and compressive below the phase-transition temperature. (III) From the bulk it is known that the lattice spacing along the c axis is not affected by the phase transition.[11] This is also the case for MnAs films, where the stress evolution along the c axis is solely reflecting the thermal-expansion properties.[26] In the basal plane, on the other hand, bulk studies revealed that the a lattice spacing increases during the transformation from β-MnAs to α-MnAs.[11] In the film, the in-plane a axis is clamped to the GaAs substrate due to epitaxial constrictions. Quite obviously, the MnAs film changes its volume by uniaxial expansion in the growth direction. The resulting modulation of the topography of the α-β stripe structure is about 1.7% [studied by atomic force microscopy (AFM), and cf. Fig. 2] and compares well with the 1.9% calculated for a clamped bulk crystal.[27]

In summary, MnAs films on GaAs(001) in the α-β-phase coexistence regime relax the built-up strain along the a-axis direction by forming an ordered array of α-MnAs ridges, separated by β-MnAs valleys.[18–20] The ordered array of stripes stretching along the c-axis direction can be easily identified in the AFM image in Fig. 2. The period measured along a line marked (a) is 125 μm with a peak-to-valley height modulation of roughly 3 nm. Perpendicular to this structure, the remaining strain along the c-axis direction also results in a weaker film height modulation. The parallel dotted lines on the right-hand side of the figure help to identify the second stripe array. The periodicity of this modulation, measured along the a axis and indicated by the line (b), is 1.28 μm and exhibits a height modulation of around 1 nm. This kind of structures reduce the stress energy by increasing the surface area and are often observed as wrinkles or periodic structures at the boundary of clamped elastic sheets.[28]

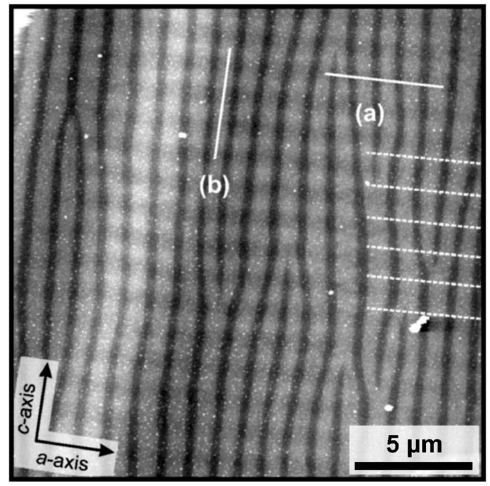

FIG. 2. Two-dimensional strain modulation in MnAs films grown on GaAs(001) (AFM micrograph, film thickness of 215 nm). The array of α-MnAs stripes (bright) is oriented parallel to the c axis and separated by β-MnAs. The period, measured along the line indicated by (a), is 1.25 μm and the peak-to-valley height modulation is roughly 3 nm. Perpendicular to this array along the a-axis direction, a second array is visible that is indicated by the dotted white lines. Its period is 1.28 μm and the height modulation is around 1 nm [measured along line (b)].

In general, the period of the α-β-stripe structure was found to be proportional to the film thickness.[6] By analyzing a multitude of samples, an empirical expression for the period p was found as $p=4.8t$, with t being the thickness. Upon changing the temperature of a film with a given thickness, the period stays the same, while the phase content ϕ varies in the range between the purely α-phase film ($\phi=1$) at the onset of the striped phase and the purely β-phase film ($\phi=0$) at the phase-transition temperature $T^\star$. This tunable periodic two-dimensional structure can be employed in the sense of a latent image since a phase-selective etchant will be able to transfer the pattern permanently into the sample. Details of the etching process will be presented in Sec. III.

III. ETCHING OF MNAS: PROCEDURE

Etching has been widely used in semiconductor research as a classic tool to make defects visible.[29] By employing suitable etchants, wet chemistry can be the basis for the formation of self-organized magnetic island structures in MnAs thin films.[2] It was found that virtually all GaAs etchants do also react with MnAs. The selective etching of α- and β-MnAs was demonstrated with both hydrochloric-acid- and sulphuric-acid-based solutions.[3] Before etching, defined areas of the film were protected with polymethylmethacrylate (PMMA, patterned by electron-beam lithography), as indicated by black squares in the checkerboard pattern top of Fig. 3. The resulting regular island structure is visible in the etched areas [cf., e.g., the white circle in Fig. 3(a)]. The dotted square in the checkerboardlike pattern indicates the position where the AFM micrograph [cf. Fig. 3(a)] was recorded.

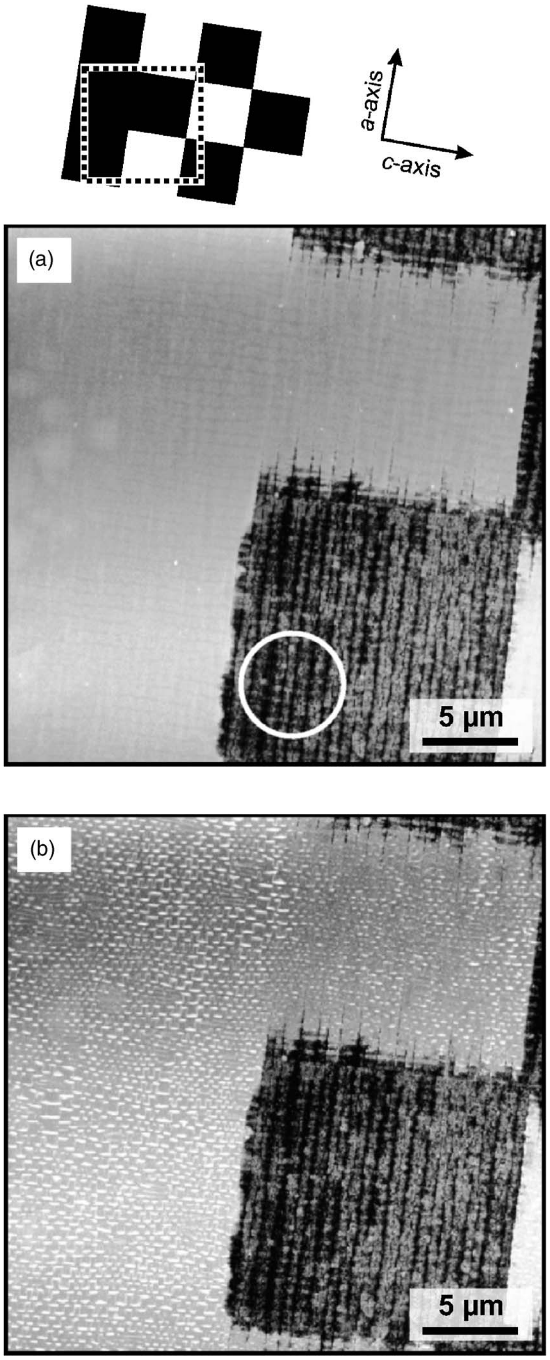

FIG. 3. Atomic force microscopy (AFM) (a) and magnetic force microscopy (MFM) (b) images of a partially wet-etched MnAs film on GaAs. The checkerboardlike pattern above shows the areas (black) that were protected by resist during the wet etching performed at room temperature. Self-organized magnetic islands are found in the etched areas (cf. white circle). Moreover, the etch solution creeps under the protective resist layer during the etching leading to irregular MnAs edges. This effect is more pronounced for *c*-axis oriented edges than for *a*-axis oriented edges. Note also the formation of cracks along the *a*-axis direction that penetrate deep under the resist mask. The MFM image shows a regular domain pattern of antiparallely magnetized domains on the ferromagnetic stripes.

A. Etching selectivity of MnAs phases

MnAs is markedly chemically reactive. Any wet etchants used for GaAs (to the extent we have investigated so far) also etches MnAs. The exposure of MnAs samples to acids, alkalis, and even water, results to a certain degree in a chemical reaction. On the other hand, MnAs does not react with alcohols, thus allowing a number of standard lithography processes for the film patterning. A hydrochloric-acid-based solution, HCl: H_2O_2: H_2O with a ratio of 4:1:12, yields a high MnAs-phase selectivity and a manageable etch rate (tens of nanometers per minute).[2] The etching process does not proceed in a layer-by-layer manner, but the etch solution selectively removes Mn atoms, as no Mn could be detected in the etched areas by energy dispersive x-ray analysis.[3] Subsequent Raman measurements revealed a broad peak at $200-230\ \mathrm{cm}^{-1}$ that is associated with amorphous As left behind after the initial stage of the etching process.[3] Further exposure to the etch solution then results in a slow dissolution of the amorphous As. It has to be noted that, in contrast with the fast etching properties of MnAs in wet etchants, MnAs withstands reactive ion etching (RIE) for extended times. In this way, MnAs was used as a RIE etch mask for GaAs.[2]

Before exposing the MnAs film to the etch solution, the surface corrugation due to the two coexisting phases of MnAs can be easily measured by, e.g., AFM. During the etching process, the sole information about the evolution of the surface corrugation does not give a sufficient information about the etching rates of the two phases. The reason for this is that if the phase that occupies a larger volume (in this case α-MnAs) etches faster than the other phase, the corrugation will first vanish and then invert with respect to the initial corrugation. Thus, we have to make use of the fact that the two phases have distinct magnetic properties. Employing a micromagnetic scanning probe technique, namely, magnetic force microscopy (MFM), in conjunction with AFM, enables us to follow precisely the etch selectivity of the MnAs phases.

The dominant micromagnetic contrast of a demagnetized MnAs sample in the α-β-stripe phase stems from oppositely magnetized domains (along the easy *a*-axis direction) that line up along the ferromagnetic stripes (oriented along the *c*-axis direction). In MFM, a ferromagnetic tip is magnetized along its axis and thus interacts with the out-of-plane moments of the magnetic stray field of the sample. At the ends of the bar-magnet-like domains, the cantilever encounters an attractive (repulsive) additional interaction, when the tip probes a stray field that points into (out of) the sample plane. The individual domains are separated by 180° Bloch walls. Figure 3(b) presents the MFM contrast obtained for an etched film (as described above). It predominantly shows alternatingly magnetized domains across the etch-protected parts of the film. Two areas of this film are of particular interest and will be discussed in more detail. The first is located close to the *a*-axis-oriented edge of the etch mask. This area allows to get a deeper insight into the etch rates of the two phases. The other is located close to the *c*-axis-oriented edge of the etch mask and illuminates the formation of the cracks and the influence of strain on the micromagnetic properties.

Taking a closer look at the *a*-axis-oriented edge, shown in Fig. 4, allows to determine the etch selectivity towards α- and β-phase MnAs. On the left-hand side (lhs), the MFM image of the unetched area clearly shows the typical micro-

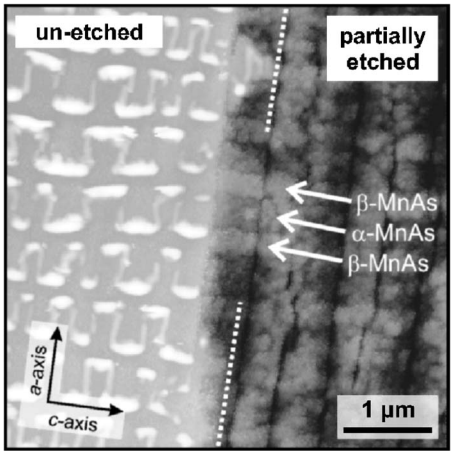

FIG. 4. Phase-selective etching of a 215-nm-thick MnAs film showing that the etch rate for α-MnAs is larger than for β-MnAs. The MFM image was recorded close to the boundary between an etched and a nominally unetched region (indicated by a dotted line). In the vicinity of the boundary, the etchant tends to underetch the protective resist mask which allows to visualize the etching selectivity. The white arrows mark the partially etched MnAs film. By correlating their position with the magnetic contrast of the α stripes on the unetched film, it can be concluded that α-MnAs etches faster than β-MnAs.

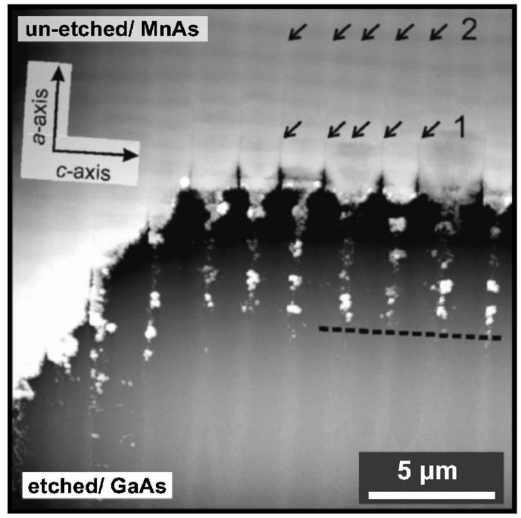

FIG. 5. AFM image at the border between the etched and unetched areas of a 215-nm-thick MnAs film. The film was etched at room temperature, i.e., in the α-β-stripe phase, again showing a large underetching. The unetched parts of the film show the coexisting phases oriented along the c axis and, perpendicular to them, regularly spaced bright stripes (see arrows labeled '2') that originate from cracks at the etch front (see arrows labeled '1').

magnetic contrast on the ferromagnetic α stripes discussed above. The dotted white line indicates the edge of the protective etch mask that was removed before the MFM scan. It is obvious that the etchant creeps underneath the mask and slowly etches from the side into the film. The underetching stops at a crack that runs along the a-axis direction slightly to the left from the dotted line. The unprotected area shows no magnetic contrast and the GaAs substrate is still covered with amorphous As, as confirmed by Raman spectroscopy. Extending now the ferromagnetic stripes found on the lhs of the image into the underetched area clearly proves that the α phase etches faster than the β phase (cf. arrows indicating the two phases).

One further remark has to be made about the etching process in terms of the dependence of the stripe phase period on film thickness. If we assume a layer-by-layer etching process, the period gets narrower in the course of the etching as the film thickness decreases. This could be a problem in terms of phase selectivity, as the initially α-phase stripes transform into β-phase stripes periodically during the etching process. For example, assuming an initial film thickness of 100 nm associated with a stripe period of roughly 480 nm, a removal of 33 nm will lead to a stripe period of 320 nm and thus a strain-induced local phase inversion. The fact that the period does hardly change during the etching process (roughly 10%, see Ref. 2) leads to the conclusion that the initial etching quickly destroys the strain balance and leaves the initial α-β period, as determined by the etching temperature, intact.

B. Etch-related strain release: Crack formation

While the Mn species are rapidly removed from the MnAs layer, the formation of regularly spaced cracks running along the a-axis direction takes place. This phenomenon can be observed in areas located close to the c-axis-oriented edge of the etch mask in Fig. 5. In general, the formation of cracks is observed for film thicknesses above 90 nm. Epitaxially grown films above a thickness of 500 nm exhibit cracks already in the as-grown state.[30] Since the MnAs layers employed in the present experiment are much thinner than the critical layer thickness for cracking, there is no reason for crack formation as the etching further reduces the film thickness. The generation of cracks is a result of the accumulated strain along the c-axis direction. Whereas the strain along the a-axis direction due to lattice mismatch between MnAs and GaAs and large difference of the thermal-expansion coefficients is relieved by the formation of the α-β-stripe structure, no such relaxation mechanism exists along the c-axis direction. The stress balance is assumed to break down during the etching, possibly due to the out-of-plane component of the stress originating from the selective etching and the wrinkles in the layers revealed in Fig. 2.

Figure 5 shows an AFM micrograph recorded at the edge of the etch window oriented along the c-axis direction. In the lower part of the figure, all MnAs is removed from the sample and the blank GaAs substrate becomes visible. Closer to the etch front, i.e., already underneath the protective etch mask (position indicated by the dashed black line) that was removed before taking the AFM scan, amorphous As islands are found. Right at the etch front, open cracks are visible that penetrate deep into the etch-protected film area, indicated by arrows labeled "1." The cracks exhibit a defined periodicity. Closed cracks (indicated by arrows labeled "2"), i.e., cracks that do not penetrate to the surface, are found further away from the edge of the protective etch mask and they exhibit the same spacing as in the two-dimensional strain modulation pattern, shown in Fig. 2. It is very likely that the etchant penetrates deep into the film directly at the MnAs–GaAs interface, leading to closed cracks that are found even 80 μm away from the edge. The local delamination that is typical for strained layers then allows the etchant to slowly open the cracks up the surface.

IV. TEMPERATURE DEPENDENCE OF THE ETCHING PROCESS

In order to clarify the origin of the crack formation in further detail, we performed etching experiments at different temperatures. Figure 6 shows MFM measurements of three samples etched in the (almost) pure α phase (a), the α-β-stripe phase (b), and the pure β phase (c). Cracks (see arrows) are observed whenever an α-phase material is present in the MnAs film. In the pure β phase, no cracks are generated (c). This behavior is independent of the film thickness.[4] The top graph shows measured data for the α-phase content obtained from x-ray diffraction.[19] The stars denote the phase compositions at which the etching experiments (a–c) were performed. If the strain along the c axis alone would be the only cause of the crack generation, the thicker MnAs films that have accumulated more stress should crack independent of the etching temperature. An analysis of the extension of the cracks into the unetched areas revealed that with decreasing α content, i.e., increasing etching temperature, the cracks get shorter, however, the separation between the cracks remains the same.[4]

Thus, we conclude that it is the existence of the α phase that is essential for the generation of cracks. The presence of the α phase is associated with two phenomena. First, the formation of the α-β-stripe structure leads to a drastically altered strain state of the film.[26] It has to be noted that even below 10 °C, especially when approaching this temperature from higher temperatures, there is a minute amount of $\beta-$MnAs left in the film due to the first-order nature of the phase transition. This leads to a similar strain state even at lower temperatures. Second, the present etching process has a high affinity to α-phase MnAs. The stress in $\alpha-$MnAs may be large enough so that any initial nonuniformity of the etching may lead to the observed combination of interfacial etching, delamination, and crack formation.

V. ETCH-INDUCED STRESS RELEASE AND ITS INFLUENCE ON THE MAGNETIC PROPERTIES

As the first-order phase transition in bulk MnAs can be strongly influenced by external pressure,[9,31] one can expect that the magnetic properties are depending on the strain state of the MnAs-on-GaAs system. It has been demonstrated that the transition temperature decreases when the volume is reduced due to hydrostatic pressure, i.e., compressive strain.[31] Therefore, MFM is not only useful in determining the phase selectivity of the etching process through the magnetic identification of the remaining material, it is also an indirect probe for the strain state of the (partially) relaxed MnAs film in the course of the etching process. In a previous study, we have investigated the domain structure of a film exhibiting periodic cracks as a function of temperature.[32] It was found that close to the phase-transition temperature of the unetched and thus uncracked film, the remaining ferromagnetic material gets pinned in the vicinity of the cracks. We found a shift of the phase-transition temperature to higher values of up to 5 °C in these areas, showing that the film expands in the vicinity of the cracks.

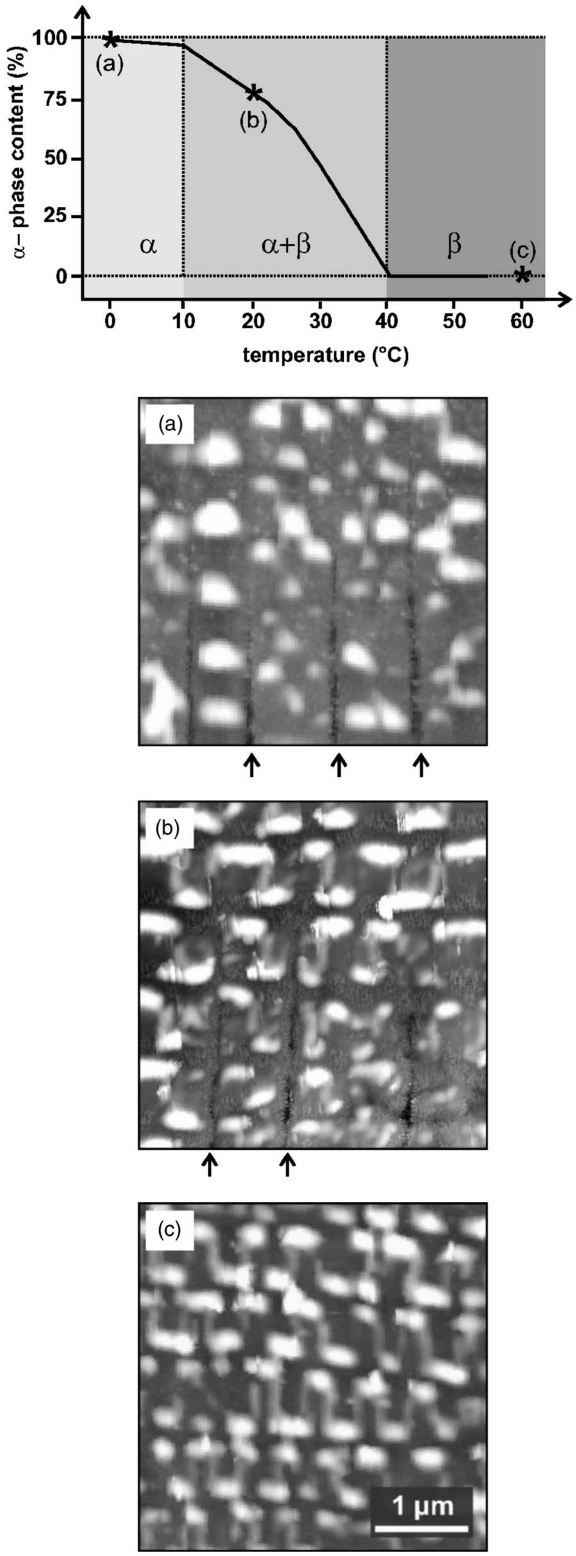

FIG. 6. Top graph: variation of the α-phase content over temperature measured by x-ray diffraction (see Ref. 19). Below 10 °C (a), the film is in its ferromagnetic α phase, whereas above ≈40 °C (c) it is in its paramagnetic β phase. In the phase coexistence regime between 10 and 40 °C (b), the α-phase content decreases steadily with temperature. Etching experiments were performed in all three structural regimes with the respective etching temperatures (and phase contents) indicated in the graph. The MFM scans below were recorded at room temperature after the etching process in the etch-protected areas of the film some micrometers above the etch window. Whenever α-MnAs was present during the etching, cracks running along the a-axis direction can be found [cf. (a) and (b)]. Etching in the complete β phase does not result in the formation of cracks.

VI. CONCLUSION

In conclusion, we have shown that MnAs films on GaAs(001) can be etched phase selectively. In the α-β-stripe phase, which exists over a wide temperature range, the stripe period and phase ratio is a function of film thickness and temperature, respectively. This interdependence of film parameters makes it difficult to directly obtain the etch rates of the two phases, as the film thickness decreases during the etching. By employing MFM and AFM, we identified ferromagnetic $\alpha-$MnAs as the faster etched phase. Apart from the periodic stripe phase structure that can be transferred into the film by etching, a periodic formation of cracks was observed in the perpendicular direction. The formation of cracks releases the remaining strain in the film and it is directly coupled to the presence of $\alpha-$MnAs during the etching process. Both immanent periodic structures together are the basis for the self-organized patterning of two-dimensional arrays of magnetic islands. Finally, the formation of cracks is accompanied by a strain release. This has a strong influence on the magnetic properties and reflects itself, e.g., in an elevated phase-transition temperature compared to the uncracked film.

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