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Magnetic domain observations of freestanding single crystal patterned Ni_2MnGa films

Qi Pan[a)]
Department of Aerospace Engineering and Mechanics, University of Minnesota, Minneapolis, Minnesota 55455

J. W. Dong and C. J. Palmstrøm
Department of Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, Minnesota 55455

J. Cui and R. D. James
Department of Aerospace Engineering and Mechanics, University of Minnesota, Minneapolis, Minnesota 55455

Ni_2MnGa thin films have been grown pseudomorphically on a 6-monolayer thick $Sc_{0.3}Er_{0.7}As$ interlayer on GaAs(001) by molecular-beam epitaxy. They have a tetragonal structure ($a=b=5.65$ Å and $c=6.18$ Å) which is different from any of the known bulk phases. Magnetic measurements reveal Ni_2MnGa to have an in-plane easy axis and a Curie temperature around 350 K. The magnetic properties of these films are given and compared to the corresponding measurements in bulk material. In contrast to bulk material, single crystal films have been predicted to exhibit exact austenite–martensite interfaces (without fine twinning of the martensite). Films have been patterned along the predicted interfaces using the conventional photolithography and reactive ion etching. The patterns are then released from the substrate by backside photolithography and selective wet chemical etching, to yield freestanding films. The martensitic transformation of the freestanding films has been observed slightly above the room temperature. Magnetic domain observations (by MFM) on the martensitic films are presented. © *2002 American Institute of Physics.* [DOI: 10.1063/1.1446114]

I. INTRODUCTION

Ferromagnetic shape memory (FSM) alloys are a new class of materials that undergo a thermodynamically reversible martensitic phase transformation and are also ferromagnetic. In the martensitic state the variants of martensite are separated by mobile twin boundaries that have been demonstrated to move by moderate external fields and/or stress.[1–3] The macroscopic shape change is produced by rearranging the martensitic twinning structure, i.e., adjusting the volume fraction of the twins, yielding strains of up to 6.2%,[4] some 60 times those that are typical in giant magnetostrictive materials.

In bulk, the Heusler alloy Ni_2MnGa is a typical FSM alloy. For a stoichiometric compound of Ni_2MnGa, the Curie temperature is ~376 K and the martensitic phase transformation temperature varies with composition near room temperature. Above the phase transformation temperature, Ni_2MnGa is a cubic $L2_1$ crystal structure with weak magnetic anisotropy. Upon cooling, it transforms to a tetragonal structure with greatly enhanced magnetic anisotropy. The magnetocrystalline anisotropy of the single crystal martensite is about 2.5×10^6 ergs/cm^3 and the c axis is easy. (The anisotropy constant k_1 of austenite is approximately two orders of magnitude smaller than that of the martensite[5]). Theory indicates that thin films of FSM alloy can deliver large work output because they exhibit large deformation modes in which the work output scales as the thickness (rather than the thickness cubed, as in typical bending actuators). To access these modes, released films are essential, as the constraint of all but the thinnest substrates would be sufficient to prevent transformation. These features, together with the possibility of remote actuation, make Ni_2MnGa a promising candidate for magnetic field driven actuator material in microelectromechanical systems (MEMS). Theoretical predictions also indicate that such films support austenite/single variant martensite interfaces; in contrast, the martensite occurs as two finely twinned variants of martensite in bulk austenite/martensite interfaces.[6] Several conceptual designs based on released single crystal FSM thin films have been proposed.[7] The first single crystal growth of Ni_2MnGa thin film has been reported[8] and martensitic phase transformation was observed in a partially released film.[9] In this article, we report the growth, characterization, and patterning of 900 Å thick single crystal Ni_2MnGa films and magnetic force microscopy study on the patterned films to demonstrate the potential application of this device in MEMS actuators.

II. EXPERIMENTAL PROCEDURE

The 900 Å thick Ni_2MnGa films were grown using MBE on GaAs substrates using a 6 monolayer (ML) $Sc_{0.3}Er_{0.7}As$ interlayer in an ultrahigh vacuum environment. The detailed description of the growth has been reported elsewhere.[8] After removal from the vacuum chamber, the samples were characterized by x-ray diffraction, transmission electron micros-

a)Electronic mail: qipan@aem.umn.edu

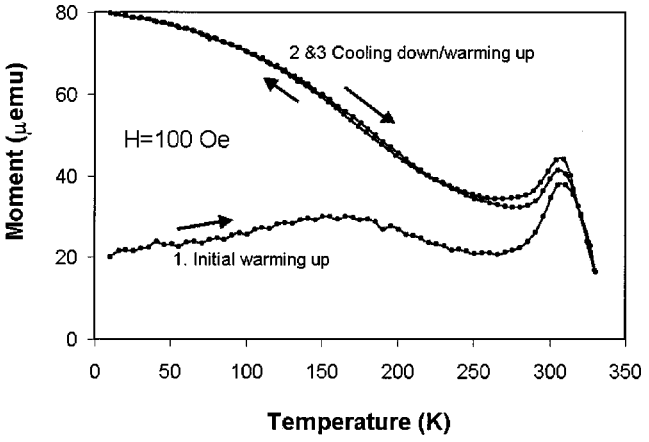

FIG. 1. Temperature dependence of the magnetization of the epitaxial Ni_2MnGa films after partial release from the substrate. The martensitic transformation occurs around 300 K.

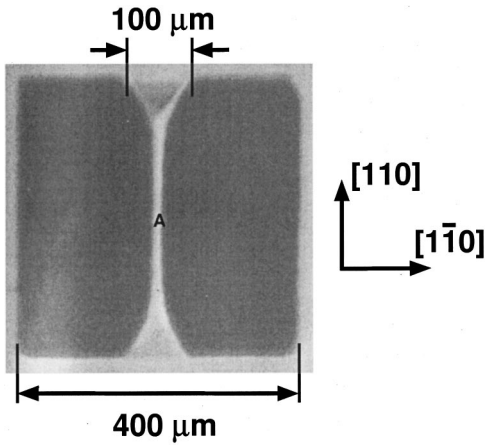

FIG. 2. Released 400 μm×100 μm×900 Å Ni_2MnGa film at room temperature showing that it has clearly popped up. The full ''dog bone'' in the center is released.

copy (TEM), and Rutherford backscattering spectrometry (RBS). Magnetic measurements were performed using a vibrating sample magnetometer (VSM) and a superconducting quantum interference device magnetometer (SQUID). The patterning and releasing of the epitaxial films from the GaAs substrate were carried out by a multistep microfabrication technique combining photolithography, reactive ion etching, and wet etching. The step-by-step sample preparation process used to pattern the film is as follows: first, a thin layer of photoresist is spin coated and exposed under UV light with a top layer of photomask. After the photoresist is developed, the sample is reactive ion etched to transfer the patterns to Ni_2MnGa by Ar/Cl_2 plasma. The photoresist is then stripped away, leaving the patterns on the front side of the substrate. The backside releasing of the patterns is made by backside IR alignment, photolithography, and selective chemical etching.

The released patterns are observed under a polarizing optical microscope with a temperature controlled specimen chamber. It was indicated by magnetic measurements (Fig. 1) that the austenite/martensite transformation does occur on the released films, with a transformation temperature slightly above room temperature. The domain structure and topography are observed with magnetic force microscopy (MFM) at room temperature.

MFM observations provide very high spatial resolution approaching 20–50 nm.[10] The MFM used for the domain imaging was Digital Instruments (DI)′ multimode NanoScope III scanning probe microscope with an inhouse-built electromagnet to apply external in-plane fields up to 500 Oe at the specimen position. Topographic and magnetic images were obtained with the instrument operated in tapping and lift mode using the standard MFM CoCr tips delivered by Digital Instruments. This tip coating has a high coercivity that minimizes tip remagnetization by stray fields from the sample and from external applied field. All the MFM images were collected with the tip magnetized along the *z* direction (perpendicular to the sample surface). The thickness of the CoCr coatings is around 50 nm and the typical lift height is 30–50 nm.

III. RESULTS AND DISCUSSION

The magnetic properties of the Ni_2MnGa MBE grown films have been reported elsewhere:[11] the saturation magnetization of the parent film is around 250 emu/cm^3 at room temperature and around 450 emu/cm^3 at 10 K, respectively. No strong in-plane anisotropy was observed when measured along the three principal in-plane directions, i.e., [110], [1$\bar{1}$0], and [100]. This is consistent with the results on bulk single crystals of Ni_2MnGa,[1] which shows that, in the high temperature stable cubic phase, Ni_2MnGa is a soft magnetic material with very weak anisotropy. By comparing the out-of-plane and in-plane hysteresis loops, the films were determined to have in-plane easy axes. Magnetization versus temperature shows no sign of the martensitic phase transformation in the unreleased sample. This is explained by a strong effect of constraint of the thick substrate.

Figure 1 shows the temperature dependence of the magnetization of the epitaxial Ni_2MnGa films after partial release from the substrate. The samples are initially zero field cooled down to 10 K. Then the magnetization is recorded by the SQUID magnetometer during warming–cooling–warming sequences, under a static in-plane magnetic field of 100 Oe. In the figure, the Curie temperature T_c is about 350 K, which is lower than the temperature reported for the bulk stoichiometric cubic $L2_1$ phase (about 376 K). This could be attributed to the difference on crystal structures. Since the properties, especially the magnetic properties, of the Heusler alloy Ni_2MnGa are very sensitive to the chemical ordering (see Ref. 12), the unique structure of the epitaxial films may demonstrate different magnetic properties. There is a significant change of the magnetic moment at around 300 K. In the freestanding part of the film, if it undergoes an austenite–martensite phase transformation, the induced magnetic moment of the low temperature magnetically hard phase is smaller than that of the high temperature magnetically soft phase. This is consistent with the magnetic properties of bulk austenite and martensite phases.[5]

Figure 2 illustrates the released 400 μm×100 μm Ni_2MnGa bridge with 900 Å thickness at room temperature

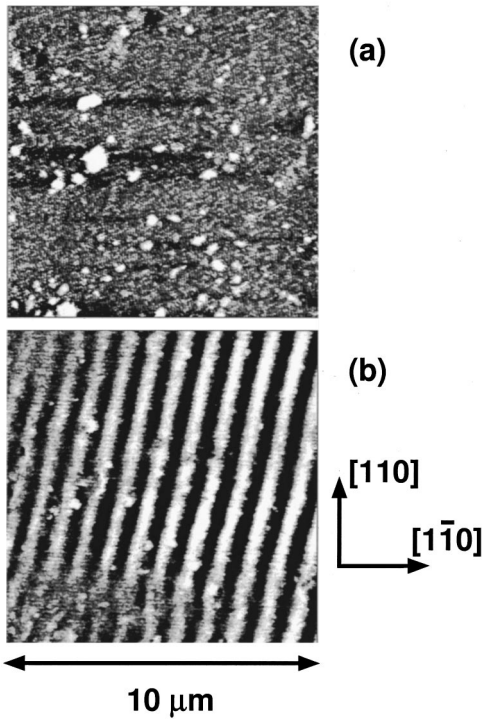

FIG. 3. (a) Topographic and (b) MFM images obtained from the center of the released bridge. Image taken from region A in Fig. 2.

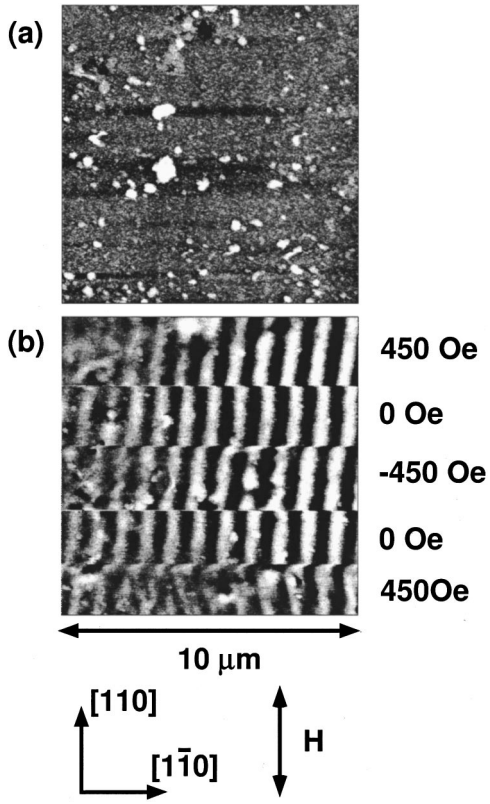

FIG. 4. (a) Topographic and (b) MFM images of the film obtained from the center of the released bridge (region A in Fig. 2) under applied in-plane magnetic field. The field was changed four times during the scanning procedure.

and the film is clearly popped up after the removal of the constrained stress from substrate. Since the phase transformation occurred at the temperature slightly above the room temperature, it is expected that the released bridge is in the low temperature hard magnetic (martensitic) phase.

The MFM results on the released MBE-grown Ni_2MnGa film are quite different from the MFM results obtained on the bulk Ni_2MnGa alloy.[13] In Fig. 3 there are the topographic (a) and MFM (b) images obtained from the center of this released bridge without any magnetic field. The orientation of Figs. 2 and 3 are the same ([110] is up). We have surveyed by MFM the full released bridge and only the center of the bridge has this alternate black and white magnetic signal and the contrast gradually decreases as the magnetic tip is moved away from the center. This might be due to the uneven stress on the bridge, i.e., the further away from the constrained edge, the more clear image the released film. Also the out-of-plane contrast is consistent with the micromagnetic prediction that the strong uniaxial anisotropy of the martensite is sufficiently strong to overcome the demagnetization energy associated with the specimen being a thin film.[6]

Figure 4 gives the topographic (a) and MFM (b) images when the film is under in-plane magnetic field parallel to the long direction of the bridge. Again, it is oriented as in Fig. 2 ([110] up) and the scan is taken from the center of the bridge. There is no significant difference while we cycle from -500 to 500 Oe due to the low temperature magnetically hard phase. It is still not very clear if the image shift is due to the twin boundary movement or just because of the drift of the MFM instrument under applied magnetic field. The bridge shown in Fig. 2 is similar to the ''tents'' found from a magnetoelastic membrane theory.[7] This connection will be explored in future work.

ACKNOWLEDGMENTS

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