

Tuning macro-twinning domain sizes and the *b*-variants content of the adaptive 14-modulated martensite in epitaxial Ni–Mn–Ga films by co-sputtering

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Abstract

In order to obtain modulated martensite in our epitaxial Ni–Mn–Ga films, we tuned the composition by using a co-sputtering process. Here we present how the composition affects the variant distribution of the 14-modulated martensite at room temperature. The nature of such modulated martensites is still strongly debated for magnetic shape memory alloys. It has been very recently demonstrated that the modulated martensites in Ni–Mn–Ga are adaptive phases. The results presented here corroborate this theory for the first time, for three different compositions. Moreover, we demonstrate with the help of the adaptive modulations theory that *b*-variants of the 14-modulated martensite form close to the free surface of the film to release the stress induced by branching of macro-twinning domains during the martensitic transformation on a rigid substrate. At room temperature, the content of such *b*-variants is found to strongly decrease when the macro-twinning domain sizes increase.

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1. Introduction

Shape memory alloys (SMAs) exhibit a displacive phase transition from the high temperature austenite phase to a martensite phase with a lower crystallographic symmetry. The martensitic transformation (MT) requires the accommodation of the martensite on a habit plane. This lattice invariant interface fixes the geometrical relationship between both crystallographic structures, the lattice mismatch being relaxed by twinning in the martensite [1–4]. Such martensite structures are thus composed of twinned

variants of different crystallographic orientations. The variants are separated by highly mobile twin boundaries, allowing rearrangement of the structure under rather low stress by preferential growing of the most favorable variant at the expense of the others. The maximal achievable strain of this super-plastic behavior depends on the tetragonality of the martensite unit cell and can reach up to 8% in Ni–Ti alloys [5].

Magnetic shape memory alloys (MSMAs) constitute a new class of SMA. In addition to the MT, these metallic alloys exhibit magnetic response. The magnetic properties of MSMA have been found to strongly depend on the alloy system. In Ni–Co–Mn–In alloys, the transformation from a cubic ferromagnetic austenite to a lower-symmetry non-magnetic or paramagnetic martensite gives rise to a large inverse magneto-caloric effect [6,7]. In Ni–Mn–Ga, the magneto-structural coupling between magnetic moments

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and martensite variants leads to a large panel of properties like magnetic induced martensite (MIM) [8] or magnetic induced rearrangement (MIR) of martensite variants [9]. This last effect has gained considerable attention as magnetic induced strains reaching 10% have been observed in Ni–Mn–Ga single crystals [10–16]. In fact, these strains are about two orders of magnitude larger than that commonly observed in magneto-strictive or piezo-electric materials [17].

Today large efforts are carried out to develop MIR-active Ni–Mn–Ga films because of their promising applications as new micro-actuators or micro-sensors for micro-electro-mechanical systems (MEMSs) [3,4,9,17,18]. Highest strains being only reported in bulk single crystals, epitaxial growth is considered to be the most promising process. Moreover, evidences of MIR in films have only been observed in epitaxially grown layers to date [4,9,17,18].

A key requirement to obtain MIR is that the magnetically induced stress exceeds the mechanical stress needed to rearrange the structure. Large magneto-crystalline anisotropy and low twinning stress of the martensite phase are thus prerequisites. These conditions can only be fulfilled in modulated structures like the 10-modulated (10M) or the 14-modulated (14M) martensites. The twinning stress of the non-modulated (NM) martensite, which is thermodynamically the more stable phase, is too high to allow MIR [19].

At room temperature (RT), the structure of the martensite has been found to strongly depend on the alloy composition, the proportion of NM martensite increasing with the average valence electron concentration per atom [19]. Tuning the composition of Ni–Mn–Ga films has been achieved by means of various techniques like changing the target composition [3], the deposition temperature [4], the sputtering reactor pressure [20] or applying a negative bias voltage on the substrate [21].

In this article we use the simultaneous deposition of a ternary Ni₅₆Mn₂₂Ga₂₂ alloy and a pure manganese target in order to tune the film composition. All parameters have been fixed to facilitate epitaxial growth on (0 0 1) MgO substrates and the composition has been varied by increasing the applied power on the manganese target. In all of our films, austenite, 14M martensite and NM martensite coexist at room temperature, due to too high average valence electron concentration per atom (e/a) factors. Here we demonstrate that the variant distribution of the 14M martensite depends on the composition. Orientations of each phase have been determined by pole figures measurements. The results corroborate for the first time the theory of adaptive modulations of Ni–Mn–Ga martensite, which was very recently developed by Kaufmann et al. [2] following Khachaturyan's concept [1]. Finally, the study focuses on the influence of composition on the surface morphology of twins. The theory of adaptive martensite and normalized integrated intensities of pole figures are also used to study the content evolution of 14M b -variants.

2. Experimental

Epitaxial Ni–Mn–Ga films have been deposited by magnetron sputtering. A low residual pressure in the range of 10^{-6} Pa was used to avoid any oxidation of the films. Epitaxial films have been grown in a confocal sputtering reactor equipped with six cathodes: three operated in direct current (DC) and three operated at radio frequency (RF). In the following, we use only two of these. The ternary Ni₅₆Mn₂₂Ga₂₂ alloy was inserted in a DC cathode whereas the pure Mn target was inserted in an RF cathode to enable tuning of the composition. In order to ensure chemical homogeneity and constant film thicknesses, the depositions were made with the substrate rotating at a speed of 5 rpm. The (0 0 1) MgO monocrystalline substrate temperature has been fixed at 773 K and the applied power (86 W) on the ternary target was optimized to obtain a deposition rate of $1 \mu\text{m h}^{-1}$. The sample batch has been deposited by increasing the power applied on the Mn target from 0 to 30 W for a fixed deposition time of 30 min. The deposition rate of the Mn target was $(2/3) 10^{-2} \mu\text{m h}^{-1} \text{W}^{-1}$, leading to film thicknesses ranging from 500 nm (no power on the Mn target) to 600 nm (30 W on the manganese target). More details on the deposition process can be found in Ref. [22].

The film compositions were determined by energy dispersive X-ray (EDX) spectroscopy using a JEOL 840A scanning electron microscope (SEM). The structural and textural characterizations were realized using X-ray diffraction (XRD). Measurements have been carried in a four-circle instrument (Seifert MZ IV) with copper K_α radiation. The diffractometer was equipped with an optic (Xenocs) enabling a low divergence (0.06°) of the X-ray beam and a rear monochromator in order to enhance the signal to noise ratio. Alignment of the samples was realized using the (0 0 2) reflection of the MgO substrate. It allows probing crystallographic orientations of all Ni–Mn–Ga phases in absolute coordinates, the MgO substrate being used as a reference system [2]. θ – 2θ scans have been measured for tilt angles ψ ranging from 0° to 10° , at two rotation angles Φ selected with respect to the epitaxial relationship between substrate and austenite, in order to determine optimized 2θ positions of the (4 0 0) (0 4 0) (0 0 4) martensites tilted lattice planes. After that, pole figures of (4 0 0) martensite reflections have been acquired in the range $\Phi = 0$ – 360° and $\psi = 0$ – 10° for both the NM martensite and the 14M martensite. The microstructures were analyzed using various microscopy techniques. Optical microscopy with polarized light (Zeiss microscope equipped with a Hamamatsu ORCA-ER digital camera) and back-scattered electron (BSE) detectors of a field-emission scanning electron microscope (FESEM Zeiss Ultra +: 8 kV, 15 mm, $100\times$) have been used to investigate sizes and morphologies of the macro-twinned domains at the surface of the films.

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