Epitaxial mosaic-like \( \text{Mn}_5 \text{Ge}_3 \) thin films on Ge(001) substrates

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Epitaxial mosaic-like \( \text{Mn}_5 \text{Ge}_3 \) thin films were grown on Ge(001) substrates using reactive deposition epitaxy process from Mn deposition or Mn-Ge co-deposition at a substrate temperature of 250 °C using magnetron sputtering. The cross-sectional transmission electronic microscopy analyses reveal an abrupt interface at the atomic scale; two equivalent epitaxial relationships are found between the substrate and the \( \text{Mn}_5 \text{Ge}_3 \) crystallites: Ge(001)[110] and [110] || \( \text{Mn}_5 \text{Ge}_3 \) (111) || [110]. The \( \text{Mn}_5 \text{Ge}_3 \) axis forms an angle of 45° with the substrate plane. Rietveld analysis from a synchrotron 2-dimensional diffraction pattern revealed that remanent deformations of about 1% exist in the films and that the magnetic anisotropy is directly related to the easy axis of magnetization lies along the hard axis of \( \text{Mn}_5 \text{Ge}_3 \) forms an angle of 45° with the substrate plane. The hexagonal crystal structure of \( \text{Mn}_5 \text{Ge}_3 \) has lattice parameters \( a=b=5.184 \text{ Å} \) and \( c=5.053 \text{ Å} \), whereas the films elaborated by Mn deposition saturate at different values depending on the orientation of the applied magnetic field: \( M_H=545 \text{ kA} \text{m}^{-1} \) and \( M_{sat}=774 \text{ kA} \text{m}^{-1} \). This \( M_{sat} \) difference is attributed to shape anisotropy of crystallites and interface quality of the films.

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1. Introduction

Spintronics, unlike traditional electronics, offers faster data processing and lower power consumption [1], among other advantages as non-volatile data storage [2,3]. The spin field effect transistor (spin-FET) is one of the most promising spintronic devices, which allows data processing at the same time that information can be stored within the magnetic orientation of the emitter and collector in order to manipulate the input and output of the transistor [4]. One of the requirements for the fabrication of a spin-FET is the selection of a suitable material for the emitter, which will take the role of an efficient ferromagnetic (FM) spin injector [5]. Diluted magnetic semiconductors (DMS) allow spin injection with an efficiency of ~10% at around 40 K. However, the use of DMS is limited due to their low Curie temperature (\( T_C \)) [6]. Additionally, it is difficult to preserve the spin polarization in a FM/insulator/semiconductor multilayer heterostructure due to the multilayers that produce a loss of spin coherence. A heterostructure that provides a better approach for an efficient spin injection is a FM/semiconductor multilayer with a Schottky barrier at the interface [7]. For this purpose, \( \text{Mn}_5 \text{Ge}_3 \) has become an important material for its FM ordering with a \( T_C = 296 \text{ K} \) and polarization above 42% [8,9]. The hexagonal crystal structure of \( \text{Mn}_5 \text{Ge}_3 \) has lattice parameters \( a=b=7.184 \text{ Å} \) and \( c=5.053 \text{ Å} \) [10], which allows the growth on Ge(111) substrates within a lattice mismatch of 3.7%. \( \text{Mn}_5 \text{Ge}_3 \) can be easily synthesized as a thin film on Ge(111) using molecular beam epitaxy (MBE) technique. The epitaxial relationship is Ge(111)[110] || \( \text{Mn}_5 \text{Ge}_3 \) (001) [100] [11]. However, the development of these structures is not suitable for the microelectronic industry, since Ge(111) is not compatible with the Si(001) technology used in most of the microelectronic devices. \( \text{Mn}_5 \text{Ge}_3 \) heterostructures offer the possibility of integration of an efficient spin injector into the Si(001) technology [12,13]. Recent studies of \( \text{Mn}_5 \text{Ge}_3 \) thin films grown on Ge(111) substrates demonstrate that the easy axis of magnetization lies on the basal (001) plane for films with thickness <25 nm, while the hard axis of magnetization lies along the c axis (oppositely to the bulk properties where the c axis is the easy axis) [14]. It has been observed that the magnetic anisotropy is directly related to the thickness of the films, as the thickness increases the easy axis turns progressively out the basal plane, but it never becomes perpendicular to the film plane [15–17].

The growth of \( \text{Mn}_5 \text{Ge}_3 \) on Ge(001) substrates using the solid phase epitaxy (SPE) method [18], which consists on Mn deposition...
at room temperature (RT) followed by thermal annealing, produces randomly distributed Mn5Ge3(001) nanoislands with average diameters of 80 nm and heights of 20 nm [19,20]. Thus another growth process needs to be considered to obtain thin films. Dang et al. [21] elaborated Mn5Ge3 thin films by MBE using reaction deposition epitaxy (RDE) method, consisting on the deposition of Mn or co-deposition of Mn and Ge at a substrate temperature, Tc, higher than RT. The thin films were grown by Mn and Ge co-deposition at 150 °C on GaSb(001) and GaAs(001) substrates. The obtained Tc were 350 and 320 K. The epitaxial relationships were identified as GaSb(001) || Mn5Ge3(111) and GaSb(001) || Mn5Ge3(111), respectively. In this work we have found a similar epitaxial relationship between Mn5Ge3 and Ge(001) but instead of the (111) plane is the Mn5Ge3(111) plane that is parallel to the Ge(001) surface within an angle of 6°.

Furthermore, doping Mn5Ge3 with carbon allows an increase of Tc up to ~450 K, due to a modification of the electronic structure [22–24]. Carbon doping increases the thermal stability of the compound and suppresses the segregation of Mn towards further deposited layers, in particular Ge [25,26]. For these reasons C-doped Mn5Ge3 is a promising candidate to be used as a spin injector on IV-group semiconductors.

In this paper, we report the growth of Mn5Ge3 thin films on Ge(001) substrates employing the RDE method. The thin film crystal growth obeys to a Stranski-Krastanov growth mode. The analyses performed by transmission electron microscopy show epitaxially grown crystallites with a sharp interface with the substrate and two epitaxial relationships. In-plane (IP) and out-of-plane (OP) magnetic M-H measurements revealed an IP easy axis of magnetization.+

2. Experimental methods

2.1. Experimental setup

Epitaxial layers were grown in a sputtering system equipped with a reflection high-energy electron diffraction system (RHEED) from STAIB operating at 30 kV to control the morphology and the crystalline structure of the sample surface. A quartz balance is used to measure the deposition rate of Mn and Ge at different powers, which was previously calibrated by depositing thin films with nominal thicknesses and then measured by scanning electron microscopy. The growth chamber has a base pressure better than 5 × 10⁻⁸ Torr and the atomic flux sources are two 2-inch high-purity 99.99% Mn and Ge targets eroded at 45 and 30 W with growth rates of 1.4 and 1.2 nm/min, respectively. Thin films were elaborated by direct deposition of Mn or by co-deposition with Ge. The operating Ar pressure is 3 mTorr, and sputtering was achieved using radio-frequency power sources. Supplemental structural characterization of the investigated sample was performed by grazing-incidence two-dimensional x-ray diffraction (GI-2D-XRD) at beamline 11–3 of the Stanford Synchrotron Radiation Light-source (SSRL). Grazing incidence experiments take advantage of the relatively large surface area bathed by the x-rays. Applied wavelength was λ = 0.97354 Å. Measurements were accomplished with a mar 345 detector. The experimental setup was calibrated with a LaB6 standard. The sample–detector distance was of 125 mm; pixel and spot linear dimensions were of 0.1 mm and incidence angle was 1°. One-dimensional (1D) pattern was obtained by integration of the Debye rings, processed by the software FIT2D [27]. Atomic force microscopy (AFM) images were obtained with a Veeco SPM MultiMode equipment in tapping mode. The transmission electron microscopy (TEM) sample preparation in cross-section was carried out using a focused ion beam (FIB) with a JEOL JEM-9320FIB equipment. The high-resolution (HR-TEM) images were acquired in scanning-TEM (STEM) mode with a JEOL JEM 2200Fs + Cs equipment operating at 200 kV. Finally, M-H and M-T curves were obtained with a superconducting quantum interference device (SQUID) from Quantum Design.

2.2. Surface preparation

Prior to the thin film growth on Ge(001) substrates, an ex-situ substrate cleaning was carried out. N-type Ge(001) sections of 1 × 1 cm² were first wet chemical cleaned by sonication for 20 min using trichloroethylene, acetone and ethanol and deionized water after each solvent bath. A sample substrate was then introduced into the growth chamber through a load lock chamber. The RHEED pattern at this stage shown in Fig. 1(a) consists on a 1 × 1 ordered strikes corresponding to a non-reconstructed surface, as the GeO2 native oxide is still present on the sample surface. The sample is then subjected to an in-situ cleaning. The first step is a thermal degassing at 400 °C for 4 h and second, a surface etching with an Ar-biased plasma at the sample holder with a power of 40 W for 20 min at RT to remove remaining C-based molecules and the native GeO2 layer. The result is an amorphous surface, which

Fig. 1. RHEED patterns of the substrate cleaning process and the thin film growth. (a) 1 × 1 ordered strikes corresponding to a non-reconstructed surface. (b) Amorphous surface after plasma etching. (c) 2 × 1 reconstructed surface. (d) Initial steps of the Mn5Ge3 growth.
RHEED patterns consists only of a continuous background as depicted in Fig. 1(b). Finally, to induce crystalline order at the surface of the sample, a rapid thermal annealing at 700 °C for a few minutes is performed until a 2 × 1 reconstructed surface is observed by RHEED as shown in Fig. 1(c). The sample is then cooled slowly to 250 °C to perform crystal growth. Mn5Ge3 thin films were grown following two different approaches: 1) Direct Mn deposition on Ge(001) substrate and 2) Mn and Ge co-deposition at stoichiometric calibrated fluxes to match the Mn5Ge3 composition. The used method for the film growth in both cases is RDE with $T_i = 250$ °C. This temperature is selected as it is higher than the initiation temperature, $T_i$, at which the reaction between Mn and Ge takes place to form Mn5Ge3 [28], but lower enough to avoid the formation of the Mn5Ge3 phase [29]. In the case of Mn and Ge co-deposition, only local diffusion is required so Mn and Ge atoms find the most convenient position minimizing the surface free energy and the interface free energy of the layer upon the experimental conditions, i.e., $T_i = 250$ °C.

3. Results and discussion

The RHEED pattern at the initial steps of the thin film growth (thickness of 5 nm) is shown in Fig. 1(d), the ordered signal is lost rapidly as the thickness of the layer increases due to the beam dispersion and to the high RMS roughness of the surface. The spots on the RHEED diffraction pattern indicate electron diffraction on transmission mode due to the high roughness of the surface. Nevertheless, one can note certain symmetry of the obtained spots along the principal specular spot indicating a good crystalline quality of the film. The evolution of the morphology of the surface (evaluated by atomic force microscopy) for different thicknesses is shown in Fig. 2(a)-(d) for Mn deposition and in Fig. 2(e)-(h) for Mn and Ge co-deposition.

The evaluation of the RMS roughness and the $z$-scale is an indication that 3-dimensional growth takes place for both approaches. However, by co-deposition it seems that coalescence and/or surface diffusion plays a major role as the grain size increases with the film thickness and hence its RMS roughness. For example, for a thickness of 1 nm, the RMS roughness is 1.15 nm. Meanwhile, the roughness for a film obtained by Mn deposition with a thickness of 1 nm is 0.26 nm, which is equal to the RMS roughness of a bare substrate, indicating that a wetting layer is perfectly formed over the substrate followed by a 3-dimensional growth. In both cases, the wetting layer covers the Ge surface completely, but Mn deposition leads to a more ordered growth regarding the morphology of the layer (i.e., more close to a layer-by-layer growth). In both cases a Stranski-Krastanov growth mode takes place.

To examine the crystalline nature of the film, HR-TEM experiments were performed on numerous samples with a thickness of 50 nm elaborated by both approaches Mn-deposition and co-deposition of Mn and Ge. Due to the similarity of the samples grown by Mn deposition and co-deposition, in Fig. 3(a) is shown a HR-TEM micrograph of a representative region of the Mn5Ge3 thin film grown by co-deposition of Mn and Ge. The image shows an epitaxial growth of Mn5Ge3 on Ge(001) substrate exhibiting an abrupt interface, as required for efficient spin injection. The interatomic distance of 6.74 Å is close to 6.22 Å which is the projection of the lattice parameter $a = 7.184$ Å along the [110] direction of Mn5Ge3 (6.22 = 7.184cos30°). The higher value indicates a tensile deformation of about 8.3% near the interface. Distortions up to 10.2% and without the formation of misfit dislocations have been observed on Mn5Ge3 nanoislands epitaxially grown on Ge(001) [19]. The interplanar distance of 4.80 Å matches the interplanar distance along the c axis, which is 5.053 Å, with a compressive deformation of ~5%. Both distances allow the identification of the $c_{\text{hex}}$-axis [001] direction of Mn5Ge3 to form an angle of 45° with the substrate plane. A similar angle has been reported on nanoclusters of Mn5Ge3 embedded in a highly diluted Ge:Mn matrix [30].

Fig. 3(b) shows a HR-TEM image, corresponding to another region of the same sample (i.e., a different region than that shown in
The atomic rows that are perpendicular to the interface correspond to either (100) or (010) atomic planes; their periodicity, measured within the resolution of the line profile scan of the HR-TEM micrograph, is 5.95 Å against 6.22 Å for the bulk lattice constants. Here is important to note that the atomic periodicity of Ge atoms along the [TT0] direction is 4 Å, which implies that 3 Ge cells match with 2 Mn₅Ge₃ cells. Thus the appropriate interatomic distance should be 6 Å instead of 5.95 Å measured upon the resolution of the line profile scan. Along with this direction, the lattice mismatch is 5.22% calculated from the expression \( \Delta a/a = (a_{\text{film}} - a_{\text{sub}})/a_{\text{sub}} \) where \( a \) stands for the lattice parameter and the subscript indicates if it corresponds to the film or the substrate.

In this case, the parallel directions between the film and the substrate are Ge[1T0] \( \parallel \) Mn₅Ge₃[TT0] compared to Ge[110] \( \parallel \) Mn₅Ge₃[TT0] of the previous HR-TEM micrograph. The conclusion to this point is that the film is composed of epitaxial crystallites forming a mosaic-like microstructure. Each crystallite is observed as a single grain in the AFM micrograph in Fig. 2(h), each crystallite has its epitaxial relationship where the difference between the orientation of two crystallites is a simple rotation of 90° in agreement with the four-fold symmetry of the substrate.

To find the plane of Mn₅Ge₃ that is parallel to the substrate surface, we simulate the crystalline structure of Mn₅Ge₃ based on the HR-TEM observations. Fig. 4(a) and (b) show the projection of the crystalline structure in a side and top view, respectively. The side view of the unit cell shows that four atomic planes are stacked along the \( c \) axis. The planes \( z = 0 \) and \( z = 1/2 \) contain only Mn atoms (denoted as Mn-I) while the planes \( z = 1/4 \) and \( z = 3/4 \) contain Mn-II and Ge atoms in equal amounts forming a Mn-Ge layer [31]. To match the lattice parameter of the substrate.

However, the most common epitaxial relationship between embedded clusters [32] or endotaxially [33] grown Mn₅Ge₃ nanoislands [19] is Ge(001)[110] \( \parallel \) Mn₅Ge₃[001][110].

Theoretical calculations of the interface energy of different Mn₅Ge₃-Ge systems show that the interface energy of the heterostructure of Ge(111)/Mn₅Ge₃(001) is 35 meV/Å², whereas the interface energy of Ge(001)/Mn₅Ge₃(001) is 87 meV/Å² [34], which is less stable than the films grown on Ge(111). Therefore, in our samples, the \( c \) axis rotates 45° to minimize the interface energy and to match the lattice parameter of the substrate.

Fig. 5(a) shows the result of the GI-2D-XRD experiment with a sample elaborated by co-deposition. The diffraction pattern consists of discontinuous rings attributed to hexagonal crystallites forming an axially-symmetric texture. Axial texture corresponds to a columnar morphology in agreement with the mosaic-like microstructure described before. Sputtering processes also induce columnar growth, so conditions for axial texture are highly favorable. Fig. 5(b) shows the 1D-XRD pattern obtained from the two-dimensional one, with its Rietveld refinement via Le Bail profile matching with R-factors: \( R_p = 5.92 \), \( Rwp = 7.47 \) and \( \chi^2 = 8.54 \). Peaks’ broadening in Fig. 5(b) is apparent. The principal sources of peak broadening are small crystal size, nonuniform microstrains and instrumental broadening. The AFM images allowed the measurement of crystallite size, which is approximately 37 nm. Instrumental broadening is known from the setup calibration. The representation of the cell dimensions physical microstrains via lattice parameters uncertainties are \( a = 7.26(8) \) Å and \( c = 5.08(6) \) Å (bulk lattice parameters are \( a = b = 7.184 \) Å and \( c = 5.053 \) Å), angles between \( a \), \( b \) and \( c \) axes remain unchanged. XRD result for crystallite size was 29 nm, close enough to the electron microscopy value.

With crystallite size and instrumental broadenings taken into account, Rietveld analysis allowed estimating sample's nonuniform microstrains as roughly 1%. This value is an average over a volume that includes Mn₅Ge₃ and the Mn₅Ge₃–Ge interface. The deformations at the interface, as measured from the HR-TEM micrographs, are naturally larger.

The magnetic properties of the 50-nm-thick films elaborated by

Fig. 4. Simulated diagram of the Mn₅Ge₃ compound. (a) Side view (b) and top view. (c) Diagram depicting the epitaxial relationship Ge(001)[110] \( \parallel \) Mn₅Ge₃[1T1][TT0]. The Mn₅Ge₃[1T1] plane matches the Ge(001) plane within an anticlockwise rotation of 6°.
the two different approaches have been examined by SQUID magnetometry. In Fig. 6(a) and (b) are shown the IP and OP M-H loops at 10 K of the samples grown by Mn deposition and Mn-Ge co-deposition, respectively. The fact that IP and OP anisotropies (hysteresis on the M-H loops) are present simultaneously in both films is attributed to the 45° angle between the c axis and the normal of the film. However, the IP and OP anisotropies are not equal, nor in the Mn-Ge co-deposition sample neither on the Mn-deposition sample. Remanent magnetization ($M_r$) is higher on the IP M-H measurements, saturation magnetization ($M_s$) is also reached faster (at lower magnetic fields) in the IP M-H loops than in the case of the OP M-H curves, indicating that the film plane behaves as an easy magnetization plane, and the normal of the film as the hard axis of magnetization. For the normalized loops (not shown) $M_s = 0.5M_r$ for the Mn-deposition sample while for the sample elaborated from Mn and Ge co-deposition $M_s = 0.39M_r$. This difference is attributed to the interface extension (few atomic layers) and crystalline quality of the film. There is a clear difference on the growth mechanism of both films: while the Mn and Ge co-deposition sample has a Mn$_5$Ge$_3$ growth front obeying a Stranski-Krastanov growth mode, the film grown by Mn deposition is created additionally by Ge and Mn atomic diffusion. Therefore, the interface quality and the magnetic behavior of the film at the interface should be different, as it is evidenced by the differences in the shape of the M-H loops. Another significant difference between both samples are the different values of $M_s$. In the co-deposited layer, saturation reaches the same values in both IP and OP directions, $M_s = 636$ kAm$^{-1}$ while different values of $M_s$ are obtained on the layer elaborated by Mn deposition: $M_s^I = 545$ kAm$^{-1}$ and $M_s^O = 774$ kAm$^{-1}$. This kind of discrepancy between the $M_s^I$ and $M_s^O$ have related sometimes in the literature to compressive deformations [35,36]. However, in our case the deformation found is about 1%, which does not seem enough to explain such a behavior. It is possible that this latest sample possesses a better interface crystalline quality and that the magnetic domains may be preferentially extended along the film plane and by the effect of shape anisotropy of single crystallites the value of $M_s^O$ is larger than $M_s^I$ as a larger demagnetizing field may take place on the OP magnetization measurement. It has been proposed that the magnetic domains in Ge(111)/Mn$_5$Ge$_3$ thin films, where the c axis is perpendicular to the film plane, consist of stripes for critical thicknesses under 25 nm. Thicker films exhibit additionally OP oriented domains located between IP magnetic domains, which favor the OP anisotropy [14]. In our samples the c axis is turned towards the plane of the film by 45° and it cannot be neglected the existence of both orientation of the magnetic domains (parallel and perpendicular to the film plane). But, nevertheless, the magnetic measurements seem to indicate that IP anisotropy is favored (more notably on the samples elaborated by Mn deposition). This issue reinforces the proposed feature that IP plane domains are elongated in the film plane and the conjunction of the effect of shape anisotropy produces a higher value of $M_s^I$. Furthermore, in the film elaborated from the Mn deposition, the thin film growth is closer to a layer-by-layer growth at the initial growth stages. This is pinpointed by the lower RMS roughness values for all samples obtained by Mn deposition than those of the Mn-Ge co-deposited samples, indicating a more ordered crystal growth in agreement with the supposition of a higher interface crystalline quality.

Fig. 7 shows the magnetization dependence on the temperature $M$-$T$ curves for both samples measured along the plane of the film.
Mn$_5$Ge$_3$ forms an angle of 45° by Dang et al. [20] on the growth of Mn$_5$Ge$_3$ thin films on Ge showed a higher roughness than the samples grown by Mn deposition. The obtained epitaxial relationship is an island-like growth; this process corresponds to a Stranski-Krastanov growth mode. Thin films on GaSb(001) ($T_C=320$ K) and GaAs(001) ($T_C=350$ K) substrates with compressive lattice mismatches of $-9.5\%$ and $-1.3\%$, respectively. The authors propose that the lattice mismatch, and hence the induced deformation, is at the origin of the enhancement of $T_C$. In our experiments the final remanent deformation of circa 1% calculated from the Rietveld analysis is not large enough to produce such a significant increase on the $T_C$ as in the case of Mn$_5$Ge$_3$ nanoislands where an induced tensile deformation of 10% leads to an increase of $T_C$ up to 350 K [19].

4. Conclusions

We have obtained epitaxial mosaic-like Mn$_5$Ge$_3$ thin films on Ge(001) substrates using the RDE method through Mn deposition and co-deposition of Mn and Ge at a $T_S=250\, ^\circ C$ by magnetron sputtering technique. Thin films grown by co-deposition of Mn and Ge showed a higher roughness than the samples grown by Mn deposition while a roughness equal to that of the substrate is obtained on ultrathin films, concluding that a wetting layer has been formed at the initial growth steps. The growth is then followed by an island-like growth; this process corresponds to a Stranksi-Krastanov growth mode. The obtained epitaxial relationship is Ge[001]//Mn$_5$Ge$_3$(1T1) // TT0, where the c$_{\text{Ge}}$ axis [001] of Mn$_5$Ge$_3$ forms an angle of 45° with the substrate plane and the plane (1T1) of Mn$_5$Ge$_3$ forms an angle of 6° with the Ge[001] plane. Rietveld analysis from the synchrotron x-ray diffraction patterns revealed that micro tensions remain in the film with a deformation of 1%. The SQUID $M$-$H$ measurements show that the films obtained by co-deposition exhibit an in-plane and out-of-plane magnetization of 636 kA$m^{-1}$, whereas the thin films obtained by deposition of Mn show a $M_s = 545$ kA$m^{-1}$ and $M_{r} = 774$ kA$m^{-1}$. The reason of the enhancement of the in-plane $M_s$ is attributed to shape anisotropy of single crystallites of Mn$_5$Ge$_3$. Finally, the Curie temperature has been enhanced to 305 K due to the effect of residual microtensions.

Acknowledgments

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References


Fig. 7. M-T curves of the samples grown by Mn deposition and Mn-Ge co-deposition.


