

Identification of the interstitial Mn site in ferromagnetic (Ga,Mn)As

T. A. L. Lima,¹ U. Wahl,² V. Augustyns,¹ D. J. Silva,³ A. Costa,² K. Houben,⁴ K. W. Edmonds,⁵ B. L. Gallagher,⁵ R. P. Campion,⁵ M. J. Van Bael,⁴ M. R. da Silva,⁶ J. G. Correia,² J. P. Araújo,³ K. Temst,¹ A. Vantomme,¹ and L. M. C. Pereira^{1, a)}

¹KU Leuven, Instituut voor Kern- en Stralingsfysica, 3001 Leuven, Belgium

²Centro de Ciências e Tecnologias Nucleares, Instituto Superior Técnico, Universidade de Lisboa, 2686-953 Sacavém, Portugal

³IFIMUP and IN-Institute of Nanoscience and Nanotechnology, Universidade do Porto, 4169-007 Porto, Portugal

⁴Laboratory of Solid-State Physics and Magnetism, KU Leuven, 3001 Leuven, Belgium

⁵School of Physics and Astronomy, University of Nottingham, Nottingham NG7 2RD, United Kingdom

⁶Centro de Física Nuclear, Universidade de Lisboa, Lisboa 1649-003, Portugal

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We determined the lattice location of Mn in ferromagnetic (Ga,Mn)As using the electron emission channeling technique. We show that interstitial Mn occupies the tetrahedral site with As nearest neighbors (T_{As}) both before and after thermal annealing at 200 °C, whereas the occupancy of the tetrahedral site with Ga nearest neighbors (T_{Ga}) is negligible. T_{As} is therefore the energetically favorable site for interstitial Mn in isolated form as well as when forming complexes with substitutional Mn. These results shed new light on the long standing controversy regarding T_{As} versus T_{Ga} occupancy of interstitial Mn in (Ga,Mn)As.

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(Ga,Mn)As has become the model system, in which to explore the physics of carrier-mediated ferromagnetism in semiconductors and the associated spintronic phenomena.^{1,2} In particular, as the most widely studied dilute magnetic semiconductor (DMS), (Ga,Mn)As is the perfect example of how the magnetic behavior of DMS materials is strongly influenced by local structure. In typical high Curie temperature (T_C) (Ga,Mn)As thin films (several % Mn regime), the majority of the Mn atoms substitute for Ga (Mn_s), while a minority fraction (several % of all Mn) occupies interstitial sites (Mn_i).^{3,4} Mn_s provides both the localized magnetic moment and the itinerant hole that mediates the magnetic coupling, whereas Mn_i has a twofold compensating effect: (i) magnetically, as Mn_i - Mn_s pairs couple antiferromagnetically and (ii) electrically, since double donor Mn_i compensates Mn_s acceptors.³ For a given Mn_s concentration, Mn_i therefore determines the hole concentration, the Fermi level and the effective Mn_s concentration (of non-compensated Mn_s moments), all of which define the magneto-electronic behavior of (Ga,Mn)As. The existence of such a crucial role of Mn_i is clearly reflected in the effect of the Mn_i concentration on the two relevant figures of merit: T_C and magnetization.³⁻⁵

Despite this central role in the understanding of (Ga,Mn)As, and, consequently, of Mn-doped III-V DMS materials, interstitial Mn is far from being a well understood defect. The presence of Mn_i in ferromagnetic (Ga,Mn)As was first reported based on ion channeling measurements.⁶ Although consistent with Mn_i occupying tetrahedral (T) interstitial sites, the measurements did not allow to discriminate between the two nonequivalent T sites: coordinated by four Ga atoms (T_{Ga}) or by four As atoms (T_{As}). Transmission electron microscopy measurements using the (002) diffracted beam indicated that Mn_i predominantly occupies the T_{As} site.⁷ X-ray absorption fine structure (XAFS) techniques were later applied, suggesting

T_{Ga} occupancy (e.g., Refs. 8 and 9). However, XAFS is not well suited to distinguish neighboring elements with similar atomic numbers, as is the case for Ga and As, especially in such cases of multi-site occupancy (substitutional and interstitial), where the site to be identified is in fact the minority one (interstitial). In pioneering work, using X-ray scattering techniques to probe dopant lattice location, comparable T_{Ga} and T_{As} occupancies have been reported.¹⁰⁻¹² Coexisting T_{Ga} and T_{As} occupancies would suggest that the Mn_i lattice site depends on the presence of neighboring Mn_s atoms, i.e., that isolated Mn_i and Mn_i in Mn_i - Mn_s pairs occupy different T sites. This is however in contradiction with density functional theory (DFT) calculations which predicted that for typical ferromagnetic (Ga,Mn)As samples (where the Mn_s concentration is significantly larger than that of Mn_i), T_{As} is the most stable site for Mn_i regardless of being isolated, in Mn_i - Mn_s pairs, or even in Mn_s - Mn_i - Mn_s triplets.¹³ Solving this inconsistency would allow for a better understanding of the mechanisms of electric and magnetic compensation by Mn_i which, as introduced above, plays a central role in the behavior of (Ga,Mn)As.

Using the emission channeling (EC) technique, we show here that interstitial Mn occupies only T_{As} sites in ferromagnetic, high Curie temperature (Ga,Mn)As thin films. The EC technique makes use of the charged particles emitted by a decaying radioactive isotope. The screened Coulomb potential of atomic rows and planes determines the anisotropic scattering of the particles emitted isotropically during decay. Along low-index crystal directions of single crystals, this anisotropic scattering results in well-defined channeling or blocking effects. Because these effects strongly depend on a) the initial position of the emitted particles, they lead to emission patterns which are characteristic of the lattice sites occupied by the emitting probe atoms. We have previously applied

^a lino.pereira@fys.kuleuven.be

the EC technique to determine the lattice location of Mn in GaAs in the ultra-low doping regime ($<0.05\%$ Mn).^{14,15} With emission channeling, the unambiguous distinction between T_{Ga} versus T_{As} sites in the GaAs (zincblende) lattice is a direct structural consequence of the mirror-asymmetry of selected crystal axis (typically $\langle 211 \rangle$ or $\langle 110 \rangle$).¹⁵

We report on two separate experiments on the lattice location of radioactive ^{56}Mn (half-life $t_{1/2}=2.56$ h) implanted into (Ga,Mn)As thin films grown by molecular beam epitaxy (MBE).¹⁶ Experiment A consisted of implanting a $\text{Ga}_{0.94}\text{Mn}_{0.06}\text{As}$ thin film (25 nm thick, grown directly onto the GaAs substrate) with ^{56}Mn to a fluence of $2 \times 10^{12} \text{ cm}^{-2}$, at an energy of 40 keV, and a beam angle of 60° with respect to the surface normal. The high implantation angle was chosen in order to maximize the fraction of ^{56}Mn probes implanted in the (Ga,Mn)As thin film. The near-Gaussian depth profile, simulated using SRIM-2008 (Ref. 17), is characterized by a peak concentration of $6.2 \times 10^{17} \text{ cm}^{-3}$ (≈ 0.003 at. %, i.e., negligible increase in Mn concentration), a projected range (average depth) of 17 nm

and a straggling of 11 nm, with an estimated $<27\%$ of the ^{56}Mn probes implanted into the GaAs substrate. Experiment B consisted of implanting a $\text{Ga}_{0.95}\text{Mn}_{0.05}\text{As}$ thin film (200 nm thick, grown onto a 200 nm AlAs buffer layer, on a GaAs substrate) with ^{56}Mn to a fluence of $7 \times 10^{12} \text{ cm}^{-2}$, with an energy of 30 keV and an angle of 17° (resulting in a peak concentration of $2.4 \times 10^{18} \text{ cm}^{-3}$, a projected range of 21 nm and a straggling of 11 nm, i.e., with all the ^{56}Mn within the film). In order to monitor the degree of disorder upon implantation of the radioactive probes and subsequent annealing, we also determined the lattice location of As using radioactive ^{73}As ($t_{1/2}=80$ d) in experiments C and D. These experiments consisted of implanting a thin film as in experiment B with ^{73}As to a fluence of $5 \times 10^{13} \text{ cm}^{-2}$, with an energy of 50 keV and an angle of 10° (resulting in a peak concentration of $1.5 \times 10^{19} \text{ cm}^{-3}$, a projected range of 26 nm and a straggling of 13 nm, i.e., with all the ^{73}As within the film). Both ^{56}Mn and ^{73}As were implanted at room temperature, at the on-line isotope separator facility ISOLDE at CERN.

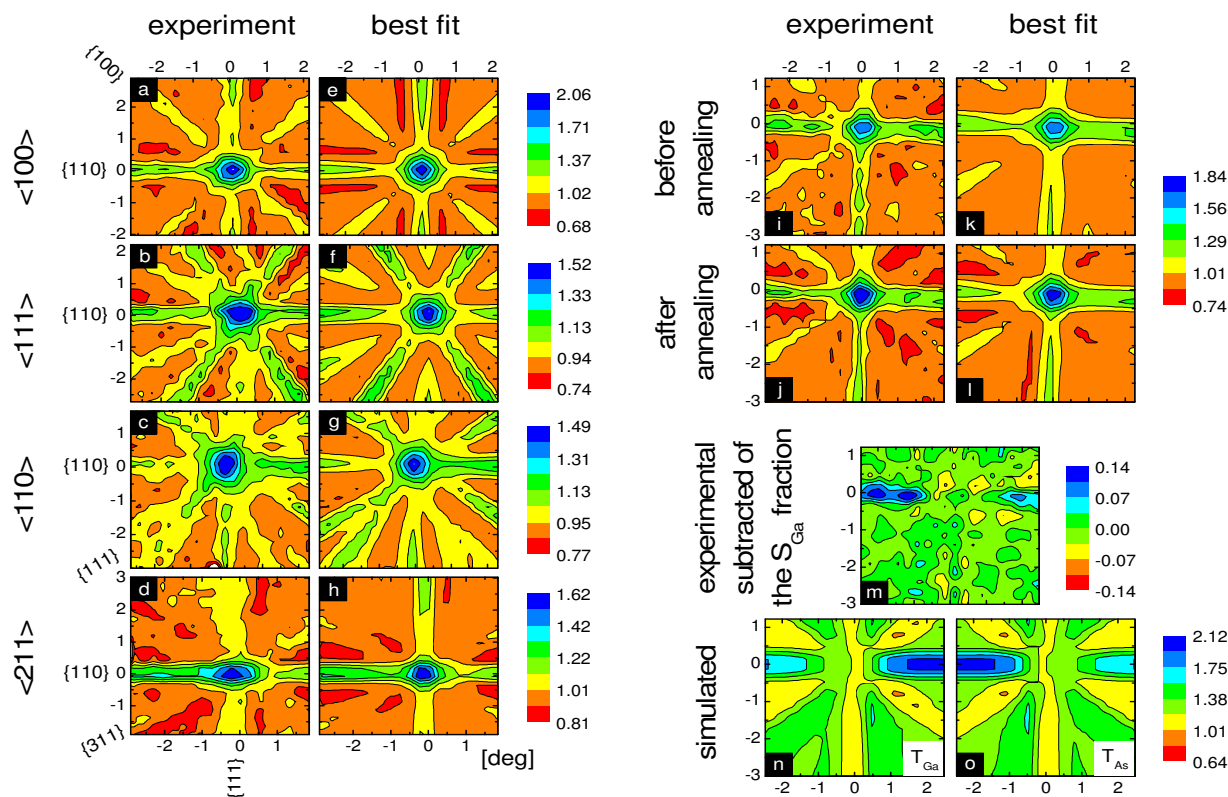


FIG. 1. (a)–(d) Experimental β^- emission patterns of experiment B after thermal annealing (10 min, in vacuum), along the four measured directions, and best fits of theoretical patterns (e)–(h). The best fit is obtained for 71% of the ^{56}Mn atoms on S_{Ga} (Mn_s) and 25% on T_{As} (Mn_i) sites. (i) and (j) Experimental patterns and best fit (k) and (l) for experiment A (only the $\langle 211 \rangle$ direction), before and after annealing (3 h, in air). The best fits are obtained for 64/77% of the ^{56}Mn atoms on S_{Ga} (Mn_s) and 25/15% on T_{As} (Mn_i) sites before/after annealing. (m) Experimental pattern [of part (j)] after subtraction of the fitted S_{Ga} component. (n) and (o) Simulated patterns for ^{56}Mn on T_{Ga} and T_{As} sites, respectively.

Emission channeling measurements were carried out in the as-implanted state and after annealing at 200 °C: in air for 3 h in experiments A and C; in vacuum ($<10^{-5}$ mbar) for 10 min in experiments B and D. Angular-dependent emission patterns were recorded along various crystallographic axis using a position- and energy-sensitive detection system similar to that described in Ref. 18. Experiments A and B (with relatively short-lived ^{56}Mn) were performed on-line using the EC-SLI setup,¹⁹ experiments C and D (the longer-lived ^{73}As) were performed off-line.¹⁸ In experiments B, C, and D, four axes were measured ($\langle 100 \rangle$, $\langle 111 \rangle$, $\langle 110 \rangle$, and $\langle 211 \rangle$), typical of emission channeling experiments on host semiconductors with cubic structure (cf. e.g., Refs. 14 and 15). In experiment A, the measurements were limited to the $\langle 211 \rangle$ axis, which as will be shown below is sufficient to unambiguously determine the site occupancy (at least when combined with experiment B, where all four axes were measured). The implantation parameters, film thickness, and annealing time of experiment A were carefully chosen to allow us to determine the lattice site(s) occupied by Mn_i in a stage of annealing for which the Mn_i fraction would have decreased to half of the as-grown/as-implanted value (based on magnetometry measurements). The theoretical emission patterns for probes occupying possible lattice sites are calculated using the many-beam formalism for electron channeling in single crystals.²⁰ The lattice location was quantitatively determined by fitting the experimental patterns with theoretical ones using the two-dimensional fit procedure outlined in Ref. 21. More details onto the experimental approach and data analysis, including a description of the lattice sites and how they can be unambiguously distinguished with emission channeling, can be found in Ref. 15. Other examples of emission channeling studies using short-lived isotopes of transition metals can be found in the following Refs. 22–24 for Mn, Refs. 23 and 25 for Co, and Refs. 19 and 26 for Ni.

As a representative example, Figs. 1(a)–1(h) show the experimental β^- emission patterns of experiment B after thermal annealing (10 min, in vacuum), along the four measured directions ((a)–(d)), as well as the best fits to the theoretical patterns ((e)–(h)). Similarly, Figs. 1(i)–1(l) compares experimental patterns ((i) and (j)) and best fit ((k) and (l)) for experiment A (only the $\langle 211 \rangle$ direction), both before and after annealing (3 h, in air). Fig. 1(m) qualitatively illustrate how the mirror asymmetry of the $\{110\}$ -planar channeling measured in the vicinity of the $\langle 211 \rangle$ direction (horizontal plane in the figure) allows us to distinguish between T_{As} and T_{Ga} sites: after subtracting from the experimental data the fitted component occupying substitutional Ga (S_{Ga}) sites, the asymmetry of the residual $\{110\}$ -planar channeling corresponds to that of T_{As} , i.e., stronger channeling on the left versus right in Fig. 1(m), which is the opposite of T_{Ga} . Quantitative analysis is provided by numerically fitting the data to calculated patterns, as mentioned above, from which the fractions of ^{56}Mn atoms in the different lattice sites are obtained – compiled in Fig. 2 (top) for experiments A and B. Figure 2 (bottom) illustrates the technique’s sensitivity specifically for the case of T_{As} versus T_{Ga} occupancy, by showing for

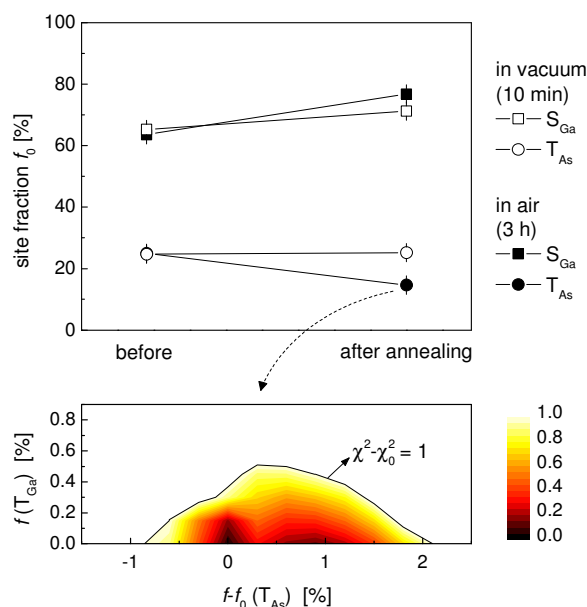


FIG. 2. Top: Fractions of ^{56}Mn atoms on S_{Ga} and T_{As} sites in experiments A and B. Bottom: Error associated with the fit $\chi^2 - \chi_0^2$, with χ_0^2 being the value for the best fit, for experiment A after annealing, as the fractions f on T_{As} and T_{Ga} sites are varied in the vicinity of the best fit values f_0 (15% for T_{As} and 0% for T_{Ga}), leaving all other fit parameters free. The black line contour corresponds to $\chi^2 - \chi_0^2 = 1$.

experiment A (after annealing) the error associated with the fit (χ^2) as the fractions on T_{As} and T_{Ga} sites are varied in the vicinity of the best fit values (15% for T_{As} and 0% for T_{Ga} , with χ_0^2), leaving all other fit parameters free. The black line contour corresponds to $\chi^2 - \chi_0^2 = 1$, giving standard deviations of 2% for the T_{As} fraction and of 0.5% for T_{Ga} . Note that the percentages are with respect to all the ^{56}Mn , i.e., even if there is indeed T_{Ga} occupancy within the techniques sensitivity, it is for all purposes negligible. Vanishing T_{Ga} occupancy (with comparable standard deviation) is found both before and after annealing, both in experiments A and B.

Before discussing in detail the T_{As} versus T_{Ga} occupancy, we make three relevant observations. First, while the interstitial Mn fraction is unaffected by the 10 min vacuum annealing (experiment B), annealing for 3 h in air decreases it by almost a factor of 2. This is consistent with our complementary magnetometry measurements (cf. supplementary material²⁷), and with the Mn_i annealing kinetics previously reported based on transport and magnetometry measurements.^{3,28} Second, in both experiments A and B, we observe an increase in substitutional fraction upon annealing, which we attribute to the recovery of disordered regions (created upon implantation), rather than interstitial Mn being converted to substitutional. This is confirmed by experiments C and D, where we see a comparable increase of the substitutional As fractions (cf. supplementary material²⁷). Third, it is important to note that the determined site-fractions correspond to the implanted radioactive ^{56}Mn probes, which are not necessarily (and most likely are not) the same as

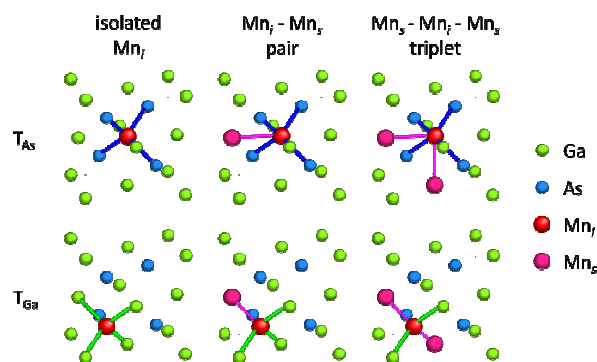


FIG. 3. Representation of the relevant Mn_i sites and coordination: in T_{As} (top) and T_{Ga} (bottom) sites, for isolated Mn_i , Mn_i-Mn_s pairs, and $Mn_s-Mn_i-Mn_s$ triplets. Our data show that regardless of the coordination, Mn_i always occupies T_{As} sites (top).

those of the stable ^{55}Mn atoms incorporated during MBE growth. The goal of these experiments was not to determine the absolute fraction of interstitial Mn in (Ga,Mn)As, but to evaluate the stability of the possibly occupied interstitial sites, which we discuss next.

The main finding here is that interstitial Mn occupies only T_{As} sites, with negligible (if any) T_{Ga} occupancy. In terms of the local structure of Mn_i (in T_{As} sites), there are essentially three possible situations to consider (Fig. 3): isolated Mn_i (coordinated by As and Ga in the first and second neighbor shell), Mn_i-Mn_s pairs (with a Mn_s substituting one of the Ga atoms in the second neighbor shell), and $Mn_s-Mn_i-Mn_s$ triplets (with two Mn_s substituting Ga atoms in the second neighbor shell). The effect of larger complexes (e.g., $3Mn_s-Mn_i$) can be neglected since the fraction of substitutional Mn in such configurations is significantly smaller than those considered (11% for $3Mn_s$ and even smaller for larger complexes, assuming random Mn distribution).²⁹ The thermal stability, i.e., the activation energy for diffusion E_a , increases from isolated Mn to Mn_i-Mn_s pairs to $Mn_s-Mn_i-Mn_s$ triplets:¹³ $E_a = E_m$ for isolated Mn, with E_m being the effective migration barrier in a $T_{As}-T_{Ga}-T_{As}$ path; $E_a = E_m + E_b$ for Mn_i-Mn_s pairs and $Mn_s-Mn_i-Mn_s$ triplets, with E_b being the binding energy of the complexes, which is larger for $Mn_s-Mn_i-Mn_s$ triplets due to the Coulomb interaction between ionized Mn_i donors and Mn_s acceptors (one/two Mn_s for pairs/triplets). Since the ^{56}Mn probes are randomly distributed upon implantation, and long range diffusion of Mn_s and Mn_i is suppressed at room temperature, we can expect the largest fraction of isolated Mn_i in the as-implanted state. At 200 °C, Mn_i is mobile and, therefore, isolated Mn_i and Mn_i in Mn_i-Mn_s pairs progressively populate the $Mn_s-Mn_i-Mn_s$ configuration (with the dissociation of Mn_i from $Mn_s-Mn_i-Mn_s$ triplets being the limiting reaction for long-range diffusion of Mn_i). Since we observe vanishing T_{Ga} occupancy both before and after annealing, we can conclude that the T_{As} site is the most stable site regardless of Mn_i being isolated, in Mn_i-Mn_s pairs or in $Mn_s-Mn_i-Mn_s$ triplets. In other words, regardless of the Mn_s coordination, the migration path of Mn_i between neighboring T_{As} and T_{Ga} sites is asymmetric, with T_{As} being the lower energy side. This picture is in qualitative agreement with previous ab initio calculations for the

different coordination configurations.¹³ An experimental assessment of the different energy barriers at play requires a detailed investigation of the lattice location as a function of annealing temperature. We are currently carrying out such studies.

T_{As} occupancy is consistent with the expectation of having the Mn cations (substitutional as well as interstitial) coordinated by As anions rather than Ga cations. For Mn_i-Mn_s complexes, however, one could expect the Coulomb attraction between oppositely charged Mn_s acceptors and Mn_i donors to counteract the repulsion between positively charged Mn_i and Ga cations, making the T_{Ga} site energetically more favorable. More specifically, since the distance between neighboring S_{Ga} and T_{Ga} sites (2.45 Å) is smaller than that between neighboring SGa and T_{As} sites (2.83 Å), the decrease in Coulomb energy by decreasing the Mn_s-Mn_i distance (from T_{As} to T_{Ga}) could eventually counteract the increase in energy of changing the Mn_i coordination from As to Ga cations. However, our results show that even if this occurs, the gain in energy associated with having the Mn_i cations coordinated by As anions is still the dominant factor, irrespective of the Coulomb interaction in Mn_i-Mn_s complexes.

In summary, we have shown that the interstitial Mn occupies only T_{As} sites both before and after thermal annealing at 200 °C, which implies that T_{As} is the energetically favorable site regardless of the interstitial Mn atom being isolated or forming complexes with substitutional Mn. These results solve the long standing controversy regarding T_{As} versus T_{Ga} occupancy, thus contributing to the understanding of electric and magnetic self-compensation in (Ga,Mn)As.

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