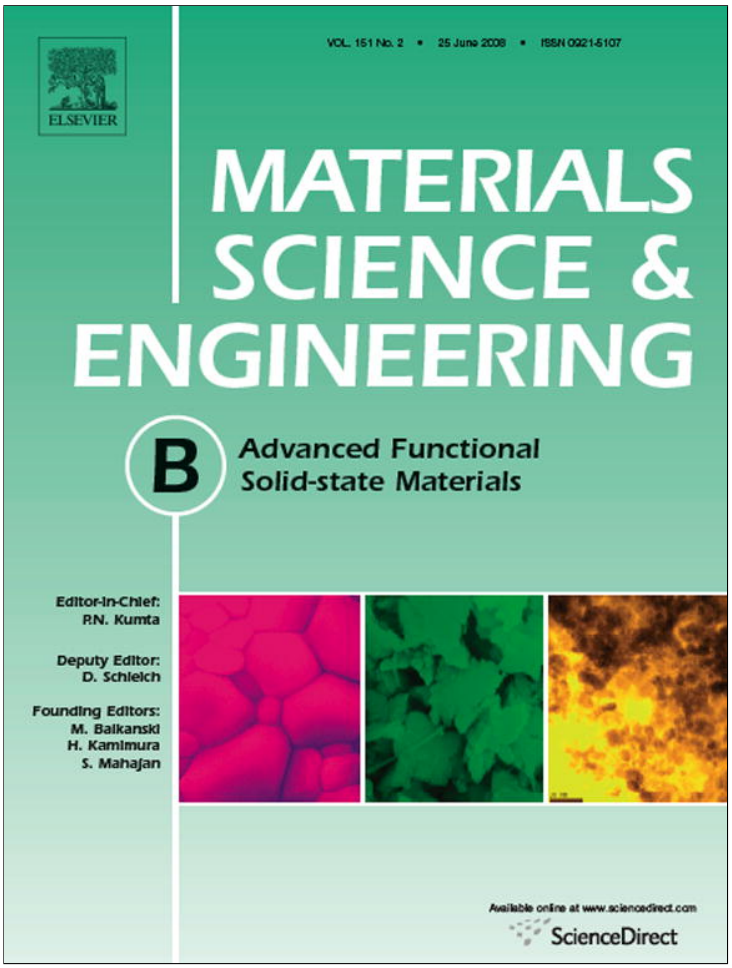

Materials Science and Engineering B 151 (2008) 152–156

Contents lists available at ScienceDirect

Materials Science and Engineering B

journal homepage: www.elsevier.com/locate/mseb

Increasing Mn substitution in magnetic semiconductors through controlled ambient annealing processes

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ARTICLE INFO

Article history:
Received 1 March 2008
Received in revised form 25 May 2008
Accepted 27 May 2008

Keywords:
Magnetic semiconductors
Crystal field theory
High Curie temperature
Magnetic GaAs
Magnetic InP
Magnetic InSb

ABSTRACT

We report on a controlled ambient annealing technique aimed at increasing the amount of Mn incorporation in III–V semiconductors. The aim is to reduce the number of hole carrier and magnetic element compensating entities, such as Mn interstitials and anti-site defects, to increase the magnetic Curie temperature. The idea is (a) to increase the number of Group III vacancies through annealing in Group V vapor rich conditions and (b) judicious use of crystal field theory to reduce/stabilize Mn interstitials. Our experimental results constitute the *highest reported* T_c (~130 K) in Mn doped InSb and Mn doped InP. The possibility of ferrimagnetism in Mn and Cr incorporated GaAs, was noted.

1. Introduction

The discovery of magnetism, through transition element doping, in traditional non-magnetic semiconductors such as GaAs [1], GaSb, Ge among others [2] has spurred a new field of inquiry into the possibility of harnessing the electron spin for various practical purposes and has brought forth the discipline [3] of spintronics. In addition to a renewed understanding of magnetic phenomena, spintronics provides an opportunity for the creation of new devices and technologies, examples of which include spin transistors which are the solid state analogs of the electro-optic modulator [4], spin-LEDs (Light Emitting Diodes) that can emit polarized light, spin valves, and the intriguing possibility of a quantum computer [2]. Another exciting development is the electrical manipulation of magnetic character [5,6], where an applied voltage can be used to tune the degree of magnetism in Mn doped III–V semiconductors.

The fundamental materials issue is how to efficiently incorporate magnetic elements into a non-magnetic semiconductor host lattice so as to impart a net magnetic character to the material. Early efforts [7] focused on the substitution of transition metals, such as Mn, in II–VI semiconductors. Only Mn, among the transition elements, was seen to have energy levels residing within the semiconductor bands [8] and help modulate the transport properties while providing a magnetic moment. However, the Mn added II–VI semiconductors were ferromagnetic at very low Curie temperatures (T_c), in most cases, <5 K, due a predominance of anti-ferromagnetic interactions [8]. The use of Mn was then adapted to III–V semiconductors and the field received a boost in the year 1989, when ferromagnetism in $In_{1-x}Mn_xAs$ [9] with a T_c of 22 K was observed. The objective of magnetic semiconductor material research since then has been to achieve ferromagnetism at/near room temperature, reproducibly. This has led to much work in the synthesis of new III–V based magnetic semiconductors [10,11]. Much of the synthesis work has used non-equilibrium techniques such as low temperature Molecular Beam Epitaxy (MBE) [2], operating far from equilibrium to overcome the thermodynamic solubility limits of Mn incorporation (p, hole carrier concentration of $\sim 1.4 \times 10^{20}$ cm^{-3}) [10]. Other variants such as digital magnetic structures [12–14], δ-doping [15], co-doping [16] ion-implantation of magnetic ions and subsequent annealing [17,18], have also been attempted. Interestingly, it has been recently demonstrated [19] that annealing of Mn doped GaAs films, prepared by MBE, in air results in a net increase in the T_c by up to 50–70 K, to 160 K. Additionally, solid state reaction at high temperatures (>1300 K) has been used to prepare magnetic semiconductors such as Mn and Co doped ZnO [20] and (Ga, Mn)As [21]. Our preliminary experiments indicate that novel high T_c ferromagnetic semiconductors can be prepared and new phenomena, relating to the nature of magnetic interactions *vis-à-vis* semiconducting properties, can be observed

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doi:10.1016/j.mseb.2008.05.020

even in materials synthesized through alternate, less capital intensive processing techniques.

2. Principles of current approach

The focus is on devising ways to increase the T_c of magnetic semiconductors through optimal transition metal (e.g., Mn, Cr, V, etc.) addition. This necessitates that the underlying magnetic interactions be thoroughly studied. First, the current understanding [8,11,22–26] is outlined. It is to be noted that much of the theoretical development has focused on Mn doped GaAs: (Ga, Mn) As and this material will be the prototype of the discussion below. The same principles, however, are applicable to any III–V semiconductor [8,27] (e.g., InP, InSb, etc.).

According to widely accepted models, the ferromagnetism in magnetic semiconductors arises through Zener type [8,28] interactions where the holes introduced through Mn doping mediate the Mn moment alignment. This mechanism is closely related to the RKKY (Ruderman-Kittel-Kasuya-Yoshida) interaction [29,30] where the mutual anti-ferromagnetic alignment between the carriers (holes) and the Mn ion, can cause two separated Mn ions to be ferromagnetically oriented. This approach predicates that ferromagnetism (and T_c) in III–V semiconductors, is related to the amount of Mn that can be incorporated, and to the number of holes introduced. Various measurement techniques [11,21] including magnetic circular dichroism (MCD) and Zeeman spectroscopy, have indicates an acceptor like nature for Mn substituting for the Group III ion, as Mn^{2+}, with a ground state electronic configuration of [Ar] ($3d^5$ + hole) in the case of arsenides and antimonides and [Ar] ($3d^4$) for the phosphides. The holes introduced are responsible for the p-type semiconducting nature and for mediating the ferromagnetism between the Mn ($3d^5$) moments. While ferromagnetic characteristics has been observed in both the insulating [31,32] and conducting [11] regimes in III–V dilute magnetic semiconductors (DMS) the above explanation is focused on the conducting/metallic region where the highest Curie temperatures (>110 K) have been observed.

While the path to increasing T_c is simple in principle, i.e., increase the amount of Mn incorporated; thermodynamics and practical materials science impose limitations. For example, in the case of GaAs, added Mn has been shown both to partly substitute for Ga (Mn_{Ga}) and partly reside in the lattice as interstitials [33] (Mn_i). The latter along with the intrinsic point defects, e.g., As_{Ga} anti-site defects, are donor like ($3d^54s^2$) and compensate [34,35] substitutional Mn_{Ga}. It has also been shown [36,37] that Mn_i interacts anti-ferromagnetically with Mn_{Ga} reducing the net magnetic moment. The limited solubility of Mn [16,36,38] in the GaAs lattice could also cause a large part of added Mn to precipitate out, either in an elemental form or as Manganese Arsenides. Additionally, Electro-Chemical-Voltage (ECV) profiling indicates electrical activity of only about 30% of the Mn atoms (ECV profiling was used instead of Hall Effect measurements to circumvent the problems associated with the intrinsic magnetization, such as the Anomalous Hall Effect).

A major effort has then focused on bypassing the thermodynamic solubility limit by working away from thermodynamic equilibrium using: (i) low temperature MBE [1], (ii) the growth of "digital structures" with alternating Mn and GaAs layers [12], and (iii) ion-implantation [17], none of which have succeeded in reliably increasing T_c. What did succeed in raising T_c to 160 K, from an "intrinsic limit" of 110 K [36], was the simple process of annealing and subsequent homogenization [19], with the maximum annealing temperature being restricted to the temperature of MBE growth (~525 K). The raise was attributed to the destruction of Mn_i and overall homogenization [39]. However, these processes also result in the removal of Mn_{Ga}.

In view of the above, we propose a technique, which has shown promise in our preliminary experiments, that can reduce the numbers of hole compensating entities, such as As_{Ga} and Mn_i and consequently increase the effective concentration of Mn_{Ga}. The resulting increase in hole carrier concentration would be responsible for raising the T_c. This is done through judicious incorporation of Mn along with Cr into the lattice and materials processing in controlled ambients. The rationale is that (1) the substitution of Mn for the Group III ion (e.g., Mn_{Ga}, Mn_{In}) is enhanced by Group III vacancies (v_{III}), which can be induced to form through annealing in Group V (e.g., P, Sb, As) vapor rich conditions.

However, one also has to account for the presence of deep level defects which are often present in III–V semiconductors. For example, in GaAs, in addition to Mn_i, donor-like As anti-site defects (As_{Ga}) can also electrically compensate [40] the Mn_{Ga}. In the case of annealing under an anion rich ambient, the following equilibrium reaction has to be considered: $v_{Ga} \Leftrightarrow As_{Ga} + V_{As}$. This reaction can be driven to the left, i.e., an increase of v_{Ga} in As rich conditions. However, while the Mn incorporation is enhanced by the vacancies, one should also reckon with the As_{Ga} defects, which form deep level states ~0.75 eV into the GaAs band gap and cannot be easily annealed out as they are stable up to 800–900 °C [40]. The addition of acceptor like Cr, which also form mid-gap states ~0.79 eV, can partially compensate the As_{Ga} and consequently increase Mn_{Ga}.

The above procedure can be placed on a quantitative footing by a calculation of the electronic band structure and the Fermi energy (E_F). The relative ratio of interstitial (Mn_i) to substitutional (Mn_{III}) is related to how close the E_F is to the valence band maxima [36,38].

Additionally, Cr can be used to pin the E_F and stabilize the Mn_i/Mn_{III} fraction, through the introduction of deep level acceptor states [40].

Crystal field chemistry could also serve as a guide to reduce Mn_i and increase T_c. A typical III–V semiconductor crystal structure (Fig. 1) consists of a face-centered cubic arrangement of Group III cations, with one half the tetrahedral interstitial sites filled by Group V anions. If one were to introduce ions which have a larger affinity [41] (i.e., higher crystal field stabilization energy: CFSE) for occupying the tetrahedral sites compared to Mn^{2+} (CFSE: 0.00 eV),

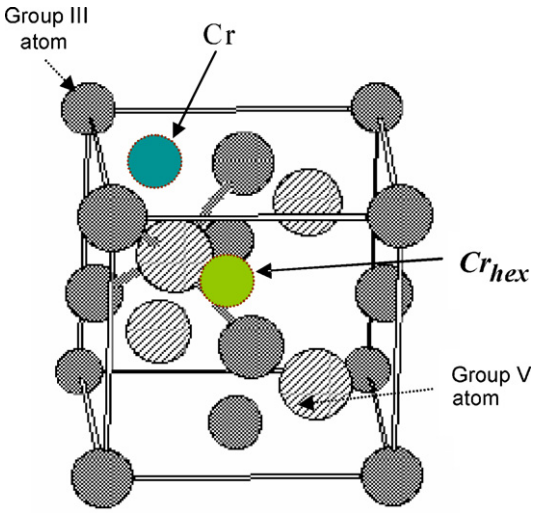

Fig. 1. The crystal structure (zinc-blende: $F\bar{4}3m$) of a III–V semiconductor. The Group III ions (e.g., Ga, In) form a face centered cubic lattice with the Group V ions (e.g., As, P, Sb) occupying one-half of the tetrahedral interstices. Based on crystal chemistry considerations, the co-addition of Cr along with Mn is advocated to reduce Mn_i, inhibit Mn_i diffusion and increase the T_c.

viz. Cr^{3+}(0.69 eV), Co^{2+} (0.64 eV), Ni^{2+} (0.37 eV), Fe^{2+} (0.34 eV), the formation of Mn_i can be prevented. From ligand field considerations [42,43], Cr is also seen to favor high co-ordination hexagonal interstitial sites when placed in a III–V semiconductor lattice while Mn shows no such preference. *The co-doping of Cr along with Mn could reduce the formation of* Mn_i.

It has been shown that a smaller value of the spin-orbit coupling [8,23], which in turn is proportional to the fourth power of the nuclear charge, can potentially increase T_c. The introduction of a smaller (ionic radii of Cr^{3+}: 0.75 Å, Mn^{2+}: 0.81 Å) and lighter ion and the reduction in the lattice constant would be beneficial. Our preliminary work has supported the above hypotheses and shows promise in increasing carrier concentrations and the T_c. The addition of Cr and other transition metals would not be trivial due to solubility limits, and further work on non-equilibrium processing is necessary to enhance the diffusion of the added elements within the semiconductor.

3. Experiment

We use a relatively simple synthesis method for proof of principle, focusing on the co-annealing of InP, GaAs, and InSb along with Mn and Cr. Semi-insulating GaAs ($\rho \sim 10^8\ \Omega$ cm), InP ($\rho \sim 10^7\ \Omega$ cm), and InSb ($\rho \sim 10^5\ \Omega$ cm) (1 0 0) oriented wafers, 600 μm thick, from University wafers Inc., were used for the experiments. The non-magnetic semiconductors were ultrasonically cleaned in organic and inorganic reagents and placed along with Mn and Cr (99.99% purity) in evacuated ($\sim 10^{-6}$ Torr) quartz ampoules in an electric furnace. Additionally, the relative incorporation of Mn and Cr incorporation into the substitutional and interstitial positions was studied through using different weights of anion elements (e.g., As, P and Sb) to vary the background/ambient anion pressure in the range of 0.1–15 atm. The annealing was performed at various temperatures in the range of 600–1000 K and for times, 1–100 h. From the published values of the diffusion constant (D) for Mn into GaAs [22], we estimated D of approximately 10^{-14} cm^2 s^{-1}. Consequently, for our annealing conditions, Mn would penetrate 400–500 nm which was the thickness used for calculating the volume of our samples. It has been shown earlier [18,19], that Mn concentrations of up to 10^{20} cm^{-3}, comparable to those currently obtained by MBE, can be achieved using diffusion and annealing processes. Careful control of the ambient vapor pressures [44] can increase the amount of Mn incorporated even further and hence increase the T_c.

X-ray diffractometry (Philips X-Pert, Rigaku) and θ–2θ scans, using Cu Kα radiation (λ: 1.542 Å), at room temperature was used to determine the lattice parameters and characterize the crystal quality, the presence of impurity phases, and strain in the Mn incorporated semiconductors. The shift in the lattice parameter, t a Vegard's law dependence of the lattice parameter on Mn concentration, was used to estimate the amount of Mn substitution. A Superconducting Quantum Interference Device (SQUID, Quantum Design, Inc.) magnetometer was used to measure the Magnetization (M)–Temperature (T) characteristics and estimate T_c. The samples were cooled from room temperature to 5 K, and a magnetic bias field of 0.1 T was then applied in the plane of the film. Hysteresis loops (M–H) indicated the presence of ferromagnetic/ferrimagnetic phases.

4. Results and discussion

We present preliminary results representative of a wide variety of magnetic behavior seen in magnetic element doped InSb, InP and GaAs. InSb, when annealed along with Mn, indicates a Weiss-like mean field behavior for the M vs. T characteristic (Fig. 2a), typical of a metallic magnetic semiconductor [24] system (the observed magnetization, in emu, has been normalized by the total volume of the sample; the thickness is estimated t the approximate Mn diffusion distance). An approximate T_c around 130 K is obtained through extrapolation. The magnetization, however, is seen to saturate at a constant value, of ~5 emu cm^{-3}. One possible explanation would be a background anti-ferromagnetic phase. However, preliminary magnetic analyses indicates a well defined hysteresis loop (Fig. 2b), with a coercivity (H_c) ~ 200 Oe. Structural analysis did not show evidence of extraneous crystalline

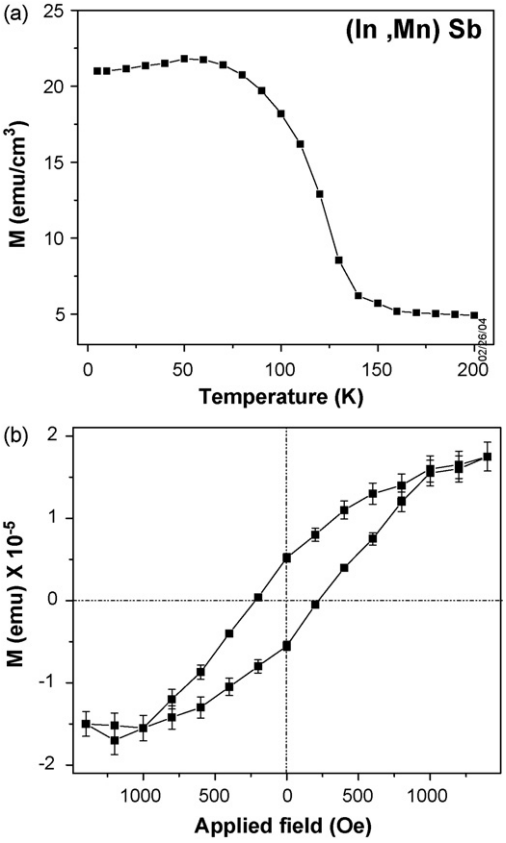

Fig. 2. (a) Magnetization plotted as a function of Temperature for InSb co-annealed with Mn for 97 h at 450 °C, $T_c \sim 130$ K. The background anion pressure is 800 Torr. (b) The magnetic hysteresis (M vs. H) loop, of annealed (In, Mn) Sb at 110 K.

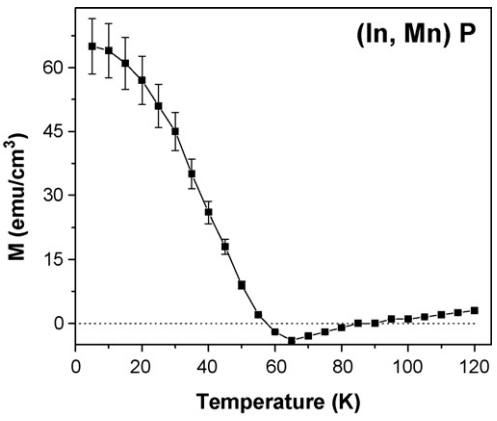

Fig. 3. Magnetization vs. Temperature for InP co-annealed with Mn for 72 h at 600 °C, $T_c \sim 55$ K.

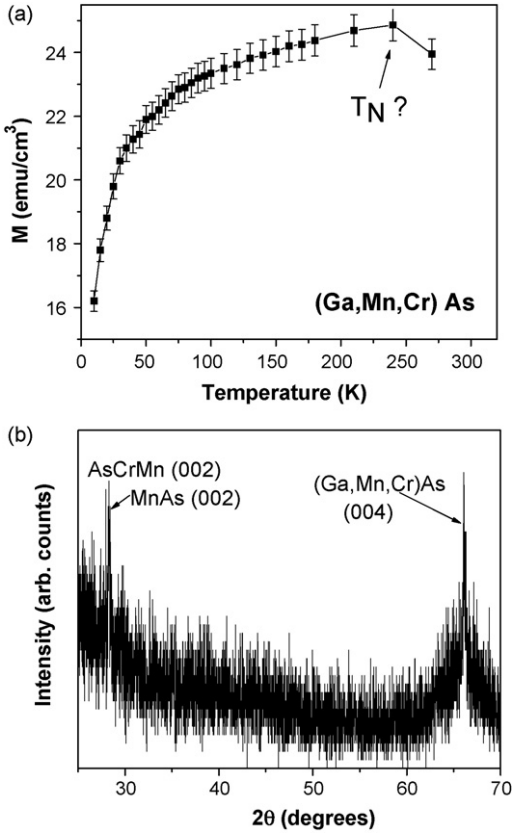

Fig. 4. (a) Possible occurrence of ferrimagnetism, with an indicated Néel temperature (T_N) by co-doping GaAs with both Mn and Cr, in an As overpressure of 15 atm. (b) X-ray diffraction spectrum of (Ga, Mn, Cr) As. In addition to the main peak at 2θ at ~66°, one observes the additional presence of extraneous orthorhombic phases, MnAs and CrMnAs [48], the effects of which have to be deconvoluted to verify the Mn and Cr incorporation into the III–V lattice.

phases, and verifies lattice substitution of the Mn for In. InP co-annealed with Mn (Fig. 3) is measured with a ferromagnetic Curie temperature of around 55 K. A background impurity phase was not identified, but cannot be ruled out in this case either.

In the case of GaAs, co-annealed in the presence of both Mn and Cr we observe a clear ferrimagnetic behavior (Fig. 4a), with a likely

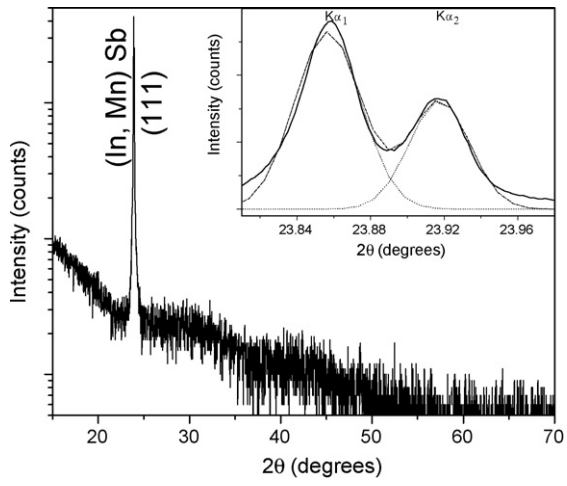

Fig. 5. X-ray diffractogram of InSb co-annealed with Mn. The inset shows a Gaussian fit (dotted) of the (In, Mn) Sb (1 1 1) peak (solid), used to estimate the extent of Mn substitution into the InSb lattice.

Table 1
The amount of Mn incorporated into InSb was calculated from the peak shift and peak fit of the InSb (1 1 1) peak (Fig. 5)

λ_{Cu}	Peak position (2θ, degrees)	Observed lattice strain	x ($In_{1-x}Mn_x$)Sb
$K_{\alpha 1}$	23.86919	0.004133	0.0997
$K_{\alpha 2}$	23.92849	0.004095	0.0988
$K_{\alpha 1}$	48.85061	0.003932	0.0948
$K_{\alpha 1}$	23.80380	0.001437	0.0346
$K_{\alpha 2}$	23.86206	0.001363	0.0329
$K_{\alpha 1}$	23.85676	0.003621	0.0873
$K_{\alpha 2}$	23.91750	0.003644	0.0879

All the samples were annealed at 450 °C for 72 h). λ refers to the specific Cu X-ray radiation used for determining the lattice parameter.

Néel temperature (T_N) at ~270 K. This gives rise to the possibility of magneto-optic recording [45] through Curie point writing, [46] in these materials. In this case, however, extraneous phases, such as MnAs and (Mn, Cr) As *were* observed through XRD (Fig. 4b). It was noticed that the lattice parameter was expanded to 0.566 nm (a_{GaAs}: 0.56533 nm) when the GaAs was annealed with Mn alone, but the concurrent addition of Cr results in a net lattice contraction to 0.564 nm. More structural and magnetic studies and correlations are needed to understand the effects of Mn and Cr. We are currently working on using magneto-optic techniques to distinguish magnetic effects intrinsic to a magnetic semiconductor from those due to the formation of extraneous phases, to shed light on such issues.

On the other hand, we do not observe any crystalline extrinsic phases for InP and InSb annealed with Mn (Fig. 5). However, in some cases the precipitation of metallic In at the surface was seen—a common issue with InP and InSb processing which could be mitigated by use of a capping layer (e.g., SiO_2) or increasing the P/Sb background vapor pressure. We estimate the concentration of Mn by analyzing the peak shift due to lattice strain (the strain due to quenched-in vacancies was estimated to be $<10^{-5}$). The analysis for (In, Mn) Sb is tabulated in Table 1, from where it is seen that Mn incorporation varies approximately through $6 \pm 3\%$. The controlled ambient annealing technique seems to be successful in aiding the incorporation of Mn into InSb.

While further work, i.e., electrical transport measurements and structural analysis are necessary confirm intrinsic ferromagnetism, our results constitute some of the *highest reported* T_c in Mn doped InSb [47] and Mn doped InP based magnetic semiconductor [18], and are clearly indicative of the promise of the current approach.

5. Conclusions

The proposed controlled ambient annealing technique will complement existing approaches, such as MBE, for the efficient incorporation of Mn and other magnetic elements into III–V semiconductors, and provide an alternative for the rapid prototyping of magnetic semiconductor materials. The annealing treatments are hypothesized to increase the amount of Mn incorporation and reduce the effect of Mn interstitials, both of which are important for increasing the ferromagnetic Curie temperature (T_c). By annealing non-magnetic InSb, InP and GaAs, in the presence of Mn and Cr, under excess anion partial pressure, we have successfully synthesized new magnetic semiconductors-with a T_c of up to 130 K being observed in Mn doped InSb. The possible occurrence of ferromagnetism in Mn-containing InP and ferrimagnetism in Mn and Cr incorporated GaAs, were noted. Further study, using magneto-optic characterization and transport measurements, is expected to yield additional insight into the suggested means for increasing the incorporation of Mn, Mn segregation and the occurrence of metastable phases, all of which are major problems.

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