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Influences of As flux on the lattice constants, magnetic and transport properties of (Ga, Mn)As epilayers

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Abstract

A series of (Ga, Mn)As epilayers have been prepared on semi-insulating GaAs (001) substrates at 230 °C by molecular-beam epitaxy under fixed temperatures of Ga and Mn cells and varied temperatures of the As cell. By systematically studying the lattice constants, magnetic and magneto-transport properties in a self-consistent manner, we find that the concentration of As antisites monotonically increases with increasing As flux, while the concentration of interstitial Mn defects decreases with it. Such a trend sensitively affects the properties of (Ga, Mn)As epilayers.

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1. Introduction

Up to now, the Curie temperature (T_C) of III–V diluted magnetic semiconductor (Ga, Mn)As has continuously been enhanced to 150–173 K [1–3]. However, for practical applications in spintronics, (Ga, Mn)As with room-temperature ferromagnetism is in strong demand. Therefore, the growth of (Ga, Mn)As epilayers with high T_C by changing the growth conditions, and understanding how both magnetic and transport properties of (Ga, Mn)As layers vary, is still the focus of effort for scientists and technologists.

It is a well-established fact that the high concentration of As antisites (As_{Ga}) is almost inherent to GaAs layers grown by low-temperature molecular-beam epitaxy (LT-MBE) [4–7]. Ferromagnetic (Ga, Mn)As growth demands an As-rich condition and a LT-MBE technique. As a result, a high concentration of As_{Ga} is inevitably present in (Ga, Mn)As epilayers, as in the case of GaAs layers grown by LT-MBE. In addition, any incorporated Mn atoms can also induce new defects such as substitutional Mn (Mn_{Ga}) and interstitial Mn (Mn_I) [8–14]. A comprehensive understanding of the behaviors of these defects and their interplay in the host crystal is closely linked to how to further improve both the magnetic and transport properties of (Ga, Mn)As layers. It is also well known that low-temperature (LT) annealing can lead to the removal of Mn atoms from interstitial positions due to their low activation energy [8–11,15,16]. By contrast, As_{Ga} is very inert below 400 °C since it has high activation energy [4]. This indicates that the As_{Ga} concentration will remain unchanged during LT annealing. It can only be adjusted by changing the growth conditions, such as the flux ratio of V/III and substrate temperature (T_S) [17]. The calculation by Mašek et al. [18] also suggests that the lattice constant of (Ga, Mn)As can be determined by the following formula:

$$a = a_0 + 0.02x_{Mn_{Ga}} + 1.05x_{Mn_I} + 0.69x_{As_{Ga}}. \quad (1)$$

(Here, a_0 is the lattice constant of GaAs, $x_{Mn_{Ga}}$, x_{Mn_I} and $x_{As_{Ga}}$ are the Mn_{Ga}, Mn_I, and As_{Ga} concentrations, respectively.) Nevertheless, it is often found in experiments that the lattice constant of (Ga, Mn)As doesn't monotonically change with an increasing V/III flux ratio, as observed by Sadowski et al. [10] and Schott et al. [19]. Many controversial results [14,20,21] reported to date still need to be clarified by further experimental studies.

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In the present work, we carry out an investigation of how the lattice constant of (Ga, Mn)As layers varies with the V/III flux ratio over a wide range of As flux. We try to trace out the manner of the incorporation or removal of Mn_I and As_{Ga} defects by studying the lattice constants, magnetic and magneto-transport properties in a self-consistent manner for a series of (Ga, Mn)As samples grown at several temperatures of the Mn cell (T_{Mn}) and As cell (T_{As}). It is found that the As_{Ga} concentration monotonically increases with increasing T_{As}, while the Mn_I concentration decreases with it. Such a trend dominates the overall characteristics of (Ga, Mn)As epilayers, from their lattice constant up to their magnetic and transport properties.

2. Sample preparation and experiments

The (Ga, Mn)As epilayers were grown on epiready semi-insulating GaAs (001) substrates by the LT-MBE technique in a V80MARKI system equipped with solid sources of elemental Ga, As, and Mn. Reflection high-energy electron-diffraction (RHEED) patterns were used to *in situ* monitor surface reconstruction throughout all the growth processes, and the T_S was measured by a W-Re thermal coupler. In all the growth runs, a 100-nm-thick undoped GaAs buffer layer was first grown at $T_S = 560$ °C on a deoxidized substrate to smooth the surface, and then T_S was reduced to 230 °C. Eventually, the growth of (Ga, Mn)As was commenced by opening the shutters of the Ga, As, and Mn cells at the same time. For the sake of comparison, three runs of samples were grown. A 2-inch intact epiready GaAs substrate was equally cleaved into five parts, and the five parts were simultaneously fixed to five molybdenum-blocks by molten indium to ensure a reproducible growth temperature for each small sample. In each growth run, all the five samples were grown under the same temperature of Ga cell (T_{Ga}), T_{Mn}, and T_S, while their T_{As} were different. The first growth run was used to obtain (Ga, Mn)As samples with a lower Mn composition, when the As flux varied over a wide range. In other words, T_{Mn} was fixed at 780 °C to maintain an Mn concentration of 3.5%, and T_{As} was adjusted to be 285, 290, 295, 300 and 310 °C successively. Then, the V/III flux ratio was monotonically changed from 8 to 36. Therefore, T_{As} was taken to be an appropriate variable to control the V/III flux ratio. For the second growth run, T_{Mn} still remained at 780 °C, but T_{As} was taken to be 288, 290, 292, 295 and 297 °C, respectively. This elaborate adjusting of T_{As} was designed to check the reproducibility between different growth runs. The third run was for growing (Ga, Mn)As samples with a higher Mn composition ($T_{Mn} = 800$ °C, equivalent to 6% Mn), while T_{As} was adjusted at five different temperatures 285, 285, 290, 300 and 310 °C for five parts from the same wafer, respectively. The two samples grown at the same $T_{As} = 285$ °C were used for checking possible growth fluctuations.

After growth, the samples were cleaved into several small parts. Some of them were used for measuring the lattice constant and magnetization before and after annealing. The others were used for transport measurements. For annealing, a set of samples in the same run were annealed on a hot plate at 280 °C (with a temperature precision of ±0.5 °C) simultaneously. Double-crystal x-ray diffraction (DC-XRD) measurements under symmetric (004) Bragg reflections were carried out at room temperature with Cu$K\alpha1$ radiation of a wavelength of 0.15405 nm (RigakuSLX-1AL). DC-XRD curves were employed to trace out the variation of the lattice constant of (Ga, Mn)As epilayers with both T_{As} and annealing conditions. The temperature dependence of remnant magnetization was measured by a superconducting quantum interference device (SQUID) in order to check the magnetic precipitates and T_C. A fixed procedure was employed as follows. A magnetic field of 1 T was applied along the [110] crystal direction to magnetize (Ga, Mn)As samples at 5 K, and then it was reduced to 0 T. Afterwards, the remnant magnetization was gradually recorded from 5 K to 200 or 380 K. The samples for magneto-transport measurements were all of Hall bar geometry with ohmic contacts made by indium bonding. The Hall resistance and sheet resistance measurements were performed by a standard ac technique over a magnetic field range from −0.4 to 0.4 T at different temperatures ranging from 25 to 300 K.

3. Results and discussion

In LT-MBE grown (Ga, Mn)As epilayers, only Mn_{Ga} forms a shallow acceptor and supplies free holes. By contrast, both Mn_I and As_{Ga} are double donors which compensate for the Mn_{Ga} shallow acceptor, and reduce the concentration of free holes in (Ga, Mn)As. As a result, the carrier concentration and the resistivity of (Ga, Mn)As samples may directly be affected by such compensation. On the other hand, now that the ferromagnetic coupling between Mn-ion local moments in (Ga, Mn)As is mediated by the free holes, the magnetic properties of (Ga, Mn)As are influenced by the As_{Ga} and Mn_I defects as well. In addition, both the lattice constant and T_C are all the functions of the Mn composition in (Ga, Mn)As, as known from Vegard's law and Zener's model [22], respectively. Accordingly, some correlations between the lattice constant, transport and magnetic properties could exist as they can all be controlled by incorporating or removing As_{Ga} and Mn_I defects into or from (Ga, Mn)As under different growth and annealing conditions.

3.1. Lattice constant of (Ga, Mn)As

A clear (1 × 2) reconstruction pattern was kept during the growth of the (Ga, Mn)As layer for all the samples, and no sign of a second phase was observed by RHEED patterns. The DC-XRD measurements also revealed the high crystalline quality of all the (Ga, Mn)As samples. The lattice constant was calculated from the position of the (004) diffraction peak of the (Ga, Mn)As epilayer. The angular precision of the measured peak position was within ±0.001°, corresponding to an error in the lattice constant smaller than $\pm 1 \times 10^{-4}$ Å. The fluctuation in the lattice constant was smaller than 4×10^{-4} Å for (Ga, Mn)As samples repeatedly grown under the same conditions and in the same run.

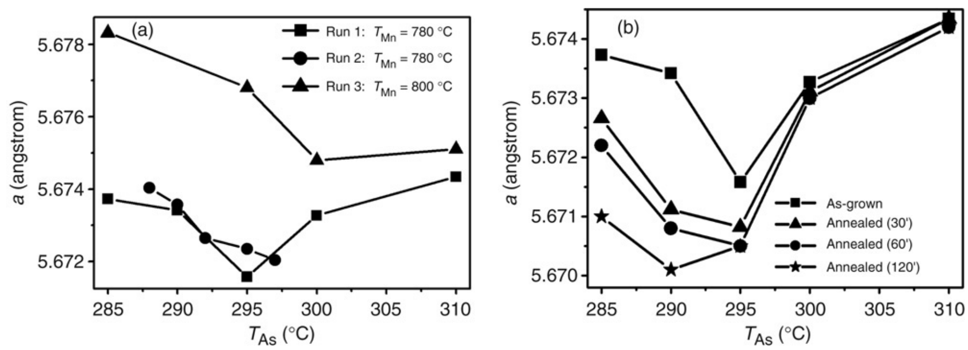

Fig. 1. (a) The dependence of the lattice constant of (Ga, Mn)As layers on the As-cell-temperature at room temperature for the three growth runs: run 1 with $T_{Mn} = 780$ °C, run 2 with $T_{Mn} = 780$ °C and run 3 with $T_{Mn} = 800$ °C. (b) For the samples grown in the first run ($T_{Mn} = 780$ °C), the As-cell-temperature dependence of the lattice constant of (Ga, Mn)As annealed at 280 °C for 0 (as-grown), 30, 60 and 120 min.

Fig. 1(a) shows the dependence of the lattice constant of (Ga, Mn)As epilayers on T_{As} for all the three growth runs. One can see that, for the first growth run with ($T_{Mn} = 780$ °C), the lattice constant of (Ga, Mn)As first decreases and then increases with increasing T_{As}. Since the samples, prepared in the same run, were grown at the same conditions except for T_{As} being adjusted differently, the variation in the lattice constant of (Ga, Mn)As should be mainly caused by different T_{As}. According to Eq. (1), the lattice constant of (Ga, Mn)As should have a linear dependence on the concentration of Mn_{Ga}, Mn_I and As_{Ga} respectively. When an over-rich As (poor Ga) condition is prevalent at growth, both Mn and As atoms are more easily able to occupy the empty sites of Ga. As a result, the lattice constant of (Ga, Mn)As should appear to increase with increasing T_{As}. However, one has to consider that there exists an opposite trend during the growth. When there is slightly rich As in (Ga, Mn)As, it also becomes favorable for Mn_I to form. In this case, Mn_I is going to expand the lattice even more effectively than As_{Ga}, and the lattice constant of (Ga, Mn)As tends to increase again with decreasing T_{As}. Such combined effects from both the increased number of Mn_I at low T_{As} and the increased number of As_{Ga} at high T_{As} may account for the observed non-monotonic behavior in Fig. 1(a). In order to check the reproducibility of such T_{As} dependence, two other runs of (Ga, Mn)As samples were also grown. The lattice constant of (Ga, Mn)As consistently shows the aforementioned evolution with T_{As}, as seen in Fig. 1(a). Even at the higher Mn concentrations, the lattice constant is found to decrease first with increasing T_{As} due to the reduced concentration of interstitial defects of Mn_I, and then to increase slightly as T_{As} goes over 300 °C, when more As_{Ga} defects are incorporated to make the (Ga, Mn)As lattice expand again. The minimum in the lattice constant of (Ga, Mn)As epilayers shifts to the higher T_{As} side for (Ga, Mn)As with a higher Mn concentration. In fact, our results are consistent with recent theoretical [13] and experimental work [10].

In order to verify the fact that the Mn_I concentration decreases when increasing the As_{Ga} concentration, a post-growth annealing test at low-temperature was employed. Fig. 1(b) shows the T_{As} dependence of the lattice constant of (Ga, Mn)As samples, grown in the first run and annealed for different times. Apparently, more Mn_I defects are removed with a prolonged annealing time, as indicated by the curves being continuously shifted to the side of smaller lattice constants. At higher T_{As} (where As_{Ga} concentrations are higher), the decrease in the lattice constant of (Ga, Mn)As becomes less. The minimum point of the lattice constant of (Ga, Mn)As gradually shifts to the lower T_{As} with prolonged annealing time. These can be attributed to the reduction in Mn_I with increasing As_{Ga}.

3.2. *Magnetic properties of (Ga, Mn)As*

Fig. 2 gives the temperature dependence of the remnant magnetization for as-grown (a) and annealed (b) (Ga, Mn)As samples ($T_{Mn} = 780$ °C), and the insets are the enlarged portions around the T_C. Because the T_C of our (Ga, Mn)As samples are all below 200 K, the remnant magnetization is measured over a temperature range from 5 to 200 K. However, the measurement is also extended up to 380 K whenever a second phase is present. For (Ga, Mn)As epilayers grown at $T_{As} = 300, 295, 290$ and 285 °C, the T_C are 25, 35, 45 and 35 K, respectively. For the sample grown at $T_{As} = 310$ °C, there exists a transition showing up around 50 K, and a nonzero remnant magnetization persists until 380 K, as seen from the magnified remnant magnetization (×5), although no sign of a second phase was observed by *in situ* the RHEED pattern or DC-XRD measurement. The latter possibly indicates that while the T_C of (Ga, Mn)As is actually about 50 K, an unknown second complex is present and responsible for the residual magnetization observed. As mentioned above, while measuring the temperature dependence of remnant magnetization, all the samples experienced the same magnetization history. Therefore, we may get useful information about evolvement of defect concentrations from analyzing the T_{As} dependences of their remnant magnetizations. As shown in Fig. 2, when T_{As} is reduced from 310 to 290 °C, the remnant magnetization monotonically increases. However, there exists a crossover between $T_{As} = 285$ and 290 °C. The remnant magnetization for the sample grown at $T_{As} = 285$ °C becomes smaller again. After these samples are annealed at 280 °C for 30 min, both the remnant magnetization and T_C increase. The T_C values after annealing become 28, 40, 55 and 50 K for the (Ga, Mn)As samples grown at $T_{As} = 300, 295, 290$ and 285 °C,

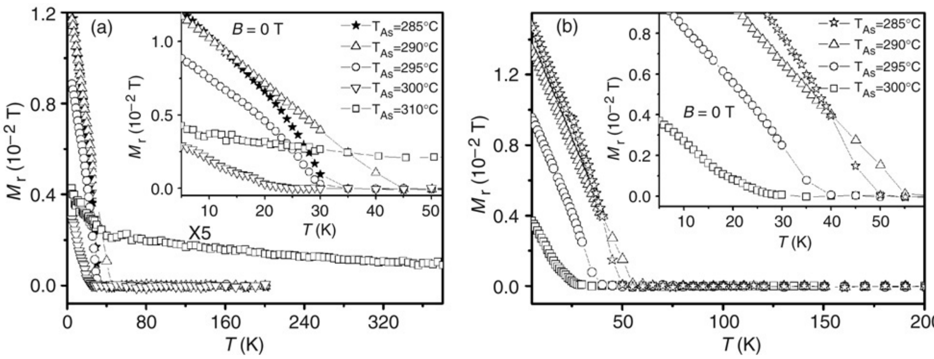

Fig. 2. For the samples grown in the first run (T_{Mn} = 780 °C), the temperature dependence of the remnant magnetization of (Ga, Mn)As grown at T_{As} = 285, 290, 295, 300 and 310 °C before (a) and after (b) a 30 min of annealing at 280 °C.

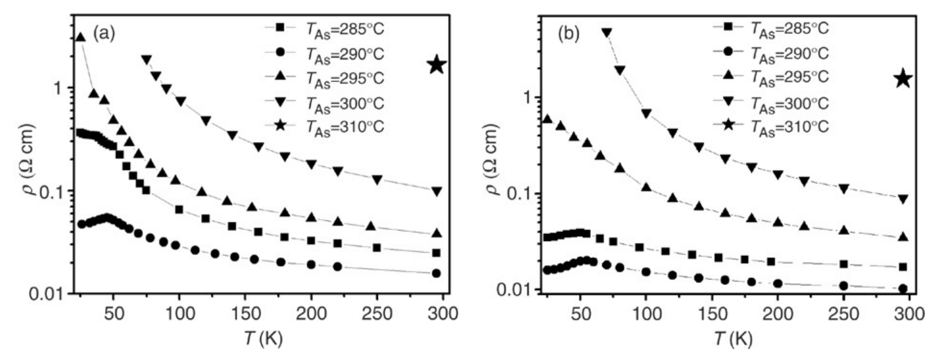

Fig. 3. For the samples grown in the first run (T_{Mn} = 780 °C), under a zero magnetic field, the temperature dependence of the resistivity of (Ga, Mn)As grown at T_{As} = 285, 290, 295, 300 and 310 °C before (a) and after (b) 30 min of annealing at 280 °C.

respectively. The crossover for the remnant magnetization obviously occurs between T_{As} = 285 and T_{As} = 290 °C. These results are plausibly in accordance with the increased content of Mn_I with decreasing As_{Ga}. Following the calculation of Mahadevan and Zunger [13], it is known that the formation energy of Mn_{Ga} is lower than Mn_I under rich-As conditions. The increase of Mn_{Ga} is in favor of the formation of the Mn_{Ga}–Mn_I–Mn_{Ga} complex, which is also ferromagnetic. This fact may possibly account for the magnetic properties observed at very high temperatures for the sample grown at T_{As} = 310 °C. When the As flux is very high, the prevalent occupation of the Mn atoms on Ga sites may lead to the formation of Mn-Ga clusters, whose T_C is higher than 400 K [23]. Whatever the second precipitate is, an over-rich As condition is unfavorable for growing ferromagnetic (Ga, Mn)As.

3.3. Magneto-transport properties of (Ga, Mn)As

In order to further understand the above results, the transport properties were also studied. Fig. 3(a) and (b) present the temperature dependence of the resistivity of the various (Ga, Mn)As samples grown at fixed T_{Mn} = 780 °C. For the sample grown at T_{As} = 290 °C, the (Ga, Mn)As is on the metallic side of the insulator–metal transition with a critical temperature of T_C (below which resistivity decreases with lower temperatures. That is the clear indication for the metallic phase). The others show insulating behavior, as evidenced by a rapidly monotonic increase in resistivity below T_C. It is well documented that (Ga, Mn)As with an intermediate Mn composition, as is the case for the (Ga, Mn)As used here, is usually metallic. Therefore, the appearances of the insulator phases in the samples grown at $T_{As} \neq 290$ °C in Fig. 3(a) and (b) may imply some influence of the compensation effect from the Mn_I and As_{Ga} on the transport properties. After annealing, the resistivity of all the (Ga, Mn)As samples generally decrease. It seems that the removal of the Mn_I defects does improve their electrical properties. It is interesting to see that, for the as-grown insulating (Ga, Mn)As sample grown at T_{As} = 285 °C, there appears a broad hump in the temperature dependence of resistivity around T_C after annealing. This suggests a revival of the metal phase for this sample, that is, the original insulating (Ga, Mn)As sample with incorporated Mn_I becomes very metallic after annealing. On the other hand, the transport property of insulating (Ga, Mn)As sample with incorporated As_{Ga} is improved only slightly by annealing.

Fig. 4(a) and (b) plot the temperature dependences of the Hall coefficient of (Ga, Mn)As before and after 30 min of annealing. In order to extract the variation of the hole concentrations from the Hall measurement, one needs to recall that the Hall resistance in magnetic semiconductors, R_{Hall}, can be expressed as

$$R_{Hall} = \frac{R_o}{d} B + \frac{R_S}{d} M \qquad (2)$$

where R_o is the ordinary Hall coefficient ($R_o = \frac{1}{ep}$), M is the perpendicular magnetization of the sample, and R_S is the anomalous Hall coefficient, which is proportional to

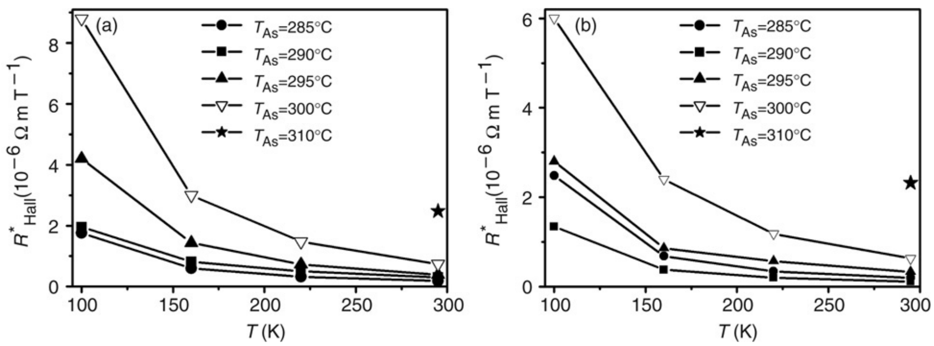

Fig. 4. For the samples grown in the first run (T_{Mn} = 780 °C), the temperature dependence of the nominal Hall coefficient of (Ga, Mn)As grown at T_{As} = 285, 290, 295, 300 and 310 °C before (a) and after (b) 30 min of annealing at 280 °C.

ρ^{γ} with a temperature independent exponent γ. Parameter γ is a constant between 1 and 2, depending on whether the scattering mechanism is dominated by the skew-scattering or side jump [24]. In Eq. (2), M is given by the Curie–Weiss law, $M = \frac{\chi_c}{T-\theta} B/\mu_0$, $\chi_c = \mu_0 p g^2 J(J+1)\mu_B^2/(3\kappa_B)$, where θ is the Curie–Weiss temperature, μ_0 is the magnetic permeability of the vacuum, μ_B is the Bohr magneton, κ_B is the Boltzmann constant, and $g = 2$, $J = 5/2$. Obviously, it is found that the anomalous Hall contribution is proportional to a term of $\frac{1}{p^{\gamma-1}\mu^{\gamma}}$, and μ is the hole mobility of the sample. From the above analysis, both the ordinary and anomalous terms in Eq. (2) increase monotonically with reductions in the hole concentration. Since our main purpose in the present work is to see how the hole concentration changes with different T_{As}, one can still define a nominal Hall coefficient R_o^* and a nominal hole concentration p^* by linearly fitting the measured R_{Hall} with Eq. (2). From Fig. 4(a) and (b), the temperature dependence of the nominal Hall coefficient R_o^* is similar to that of the resistivity ρ. The nominal Hall coefficient R_o^* is also reduced after annealing. Usually, when there are more compensation defects in the host crystal, the hole concentration gets smaller. The same is true of the mobility, because ionized impurity scattering dominates in the entire temperature range [25]. Therefore, it is expected that both the ordinary and anomalous Hall terms increase with the increasing concentration of compensation defects. However, it is of interest to note that the nominal Hall coefficient R_o^* decreases by more than a factor of 10 with increasing the temperature from 100 K to room temperature. Such a large decrement traces the dominating contribution from the temperature dependence of the anomalous Hall effect.

Fig. 5 plots the T_{As} dependence of the nominal hole concentration p^* and resistivity ρ of (Ga, Mn)As before and after 30 min of annealing at 280 °C. The nominal hole concentrationp^* of (Ga, Mn)As samples grown at T_{As} = 290 °C is the highest, and their resistivity ρ is the lowest. Both below and above that T_{As}, the nominal hole concentration p^* decreases, and the resistivity ρ increases. This value of T_{As} seems to be an optimum condition for growing (Ga, Mn)As (about 4% Mn). Under As-richer conditions, the As_{Ga} concentration increases, and the Mn_I concentration decreases. Since the increment of As_{Ga} is usually larger than the decrement of Mn_I, the total compensation defects increase. As a result, the electrical properties become worse.

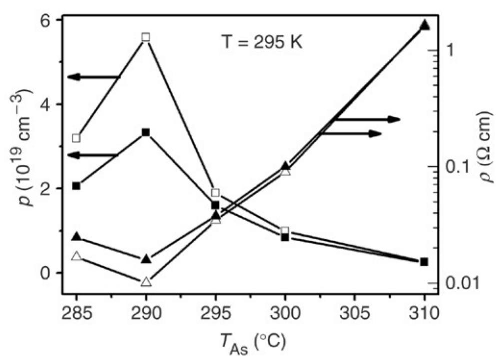

Fig. 5. At room temperature, the T_{As} dependence of the resistivity (triangles) and nominal hole concentrations (squares) of (Ga, Mn)As grown in the first run (T_{Mn} = 780 °C) before (closed) and after (open) 30 min of annealing at 280 °C.

As mentioned above, the compensation defects, As_{Ga} and Mn_I, have strong influences on the lattice constant, magnetic and magneto-transport properties of (Ga, Mn)As layers. The remnant magnetization, T_C, resistivity and nominal hole concentration all show that the growth of (Ga, Mn)As may be optimized at T_{As} = 290 °C, while the minimal lattice constant appears clearly at T_{As} = 295 °C. Generally speaking, both magnetic and transport properties are improved as the Mn composition increases. Nevertheless, due to defect-induced lattice expansion, the lattice constant in real (Ga, Mn)As with various compensation-type defects is primarily determined by the relative changes in the concentrations of the different compensation-type defects in a manner as shown in Eq. (1). On the other hand, both Mn_I and As_{Ga} are double donors, their compensation to the free holes will give rise to a similar deterioration of the magnetic and transport properties of (Ga, Mn)As layer. However, it is worth noting that Bouzerar et al. [14] calculated the influence on the ferromagnetism of (Ga, Mn)As by incorporating the compensation defects and found that Mn_I is the main source of compensation. From his calculation, it was also inferred that the lattice constant, magnetic and magneto-transport properties were easily changed by annealing. Therefore, our present work can provide a more comprehensive understanding about the influences of the various compensation defects noted on the lattice constant, magnetic and transport properties.

4. Summary

In summary, by systematically studying the crystallography, magnetic and transport properties of a series of (Ga, Mn)As samples grown at different temperatures of the As cell, we find that the As_{Ga} concentration monotonically increases with increasing T_{As}, while the Mn_I concentration decreases with it. Such a trend sensitively affects the properties of (Ga, Mn)As epilayers.

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