Compositional disordering of strained InGaAs/GaAs quantum wells by Au implantation: Channeling effects

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Compositional disordering of strained InGaAs quantum wells by the implantation of Au ions has been examined as a function of the incident implantation angle. Together, photoluminescence and secondary ion-mass spectrometry demonstrate that mixing at depths significantly greater than the mean-implantation range is due to the creation of point defects by ions which have channeled into the crystal. Compositional disordering effects due to the rapid thermal diffusion of Au ions was negligible.

The use of focused ion beams (FIB) in a variety of areas has increased significantly in the last few years. In particular, patterned implantation through quantum wells (QW) and subsequent annealing to produce zero and onedimensional structures by compositional disordering of the QW has been demonstrated. However, recent reports have shown that this effect occurs at depths significantly beyond the expected range of the ion. Petroff and co-workers have used cathodoluminescence (CL) from GaAs QW of different thicknesses located at depths as far as 500 nm below the surface, to monitor the implantation effects of 50–150 keV Ga+ ions (predicted mean range, R=60 nm; range straggling, ΔR=28 nm). This study demonstrated, by measuring the energy shift and the overall intensity of the luminescence, that changes occurred 500 nm below the surface. A number of possibilities were discussed with ion channeling being chosen as the likely mechanism. Indeed, one supplier of FIB equipment has considered channeling as a potential problem, and offers the option of doing the implantations at an angle to the surface normal.

Somewhat surprisingly, there has been resistance to the idea that ion channeling is the mechanism responsible for deep disordering. To clarify this issue, we have grown a GaAs test structure which contains four 3.0 nm thick, strained In0.53Ga0.47As QW of differing compositions (x = 10, 15, 20, 25% nominal) located at varying depths from the surface (50, 100, 250, 450 nm, respectively). The sample was capped with 15 nm of Al0.3Ga0.7As to reduce surface recombination. A second structure with the same nominal compositions and thicknesses but with the order of the QW reversed, was grown to confirm that none of the effects observed were due to the particular ordering. The samples were grown by solid-source molecular-beam epitaxy using Ga, In, Al, and As. GaAs and AlGaAs were grown at 650 °C at a growth rate of 0.7 μm h⁻¹, while the InGaAs was grown after the substrate temperature was ramped rapidly to 565 °C.

Two different implantation facilities were used in this study. First, a JIBL 104 ultrahigh vacuum FIB was used to implant uniform areas (250×250 μm) with 100 keV Au+ ions obtained from a Au-Be-Si source. A current of 14 pA with a beam diameter of ~70 nm was used to implant doses ranging from 3×10¹² to 6×10¹³ ions/cm². The geometry of the target stage and ion column is such that the implantation direction is normal to the epilayer surface (nominally a 0° alignment angle). Second, on pieces from the same wafer, 250 keV Au+ ions from a 1.7 MV Tandetron were implanted using a five-axis target stage, which permits precise orientation of the substrate with respect to the trajectory of the Au ions. The orientation of the epilayer was determined by measuring the Rutherford backscattering yield of 1 MeV He ions from a different area of the same sample as a function of tilt and the azimuthal angle. Implantations were done with the Au beam parallel to the surface normal (nominal 0°, similar to the FIB implant), at 4° and 12° to the surface normal, and into the aligned (100) axis. Finally, an implantation was also done through a 20 nm Si₃N₄ cap at the nominal angle of 0°. The samples were rapid thermally annealed (RTA) at 850 °C for 60 s in a N₂ atmosphere, using a Heatpulse 410 RTA to initiate the compositional disordering of the QW and remove nonradiative recombination sites. The sample surfaces were protected during annealing by placing a second, larger piece of GaAs on top of them.

The obvious advantage of implanting Au+ ions into GaAs (instead of Ga+ ions) is that the depth distribution of ions for the different alignments can be obtained directly by secondary ion mass spectrometry (SIMS). In this case, a Cameca IMS-4f ion microprobe was used with 10 keV Cs+ ions (14.5 keV net-impact energy) for sputtering, while 197Au negative ions were detected. We have used photoluminescence (PL), using a He-Ne laser source with the sample held at 4 K, to monitor the changes in composition and width of the QW. The resolution of the
CHANNELING IONS SHIFTED TO GREATER DEPTHS BECAUSE OF THE NEGATIVE IONS DUE TO MATRIX EFFECTS IN EITHER THE INGaAs WITH THE MEAN RANGE AND THE DEPTH OF PENETRATION OF THE Au IONS (EXPECTED $R_p \sim 27$ nm, $\Delta R_p \sim 10$ nm) AS IMPLANTED USING THE FIB AT A NOMINAL ANGLE OF $0^\circ$. IMMEDIATELY OBVIOUS IS THE LONG TAIL OF THE DISTRIBUTION WHICH EXTENDS THROUGH THE THIRD QW (AT A DEPTH OF 250 nm) WITH A SMALL FRACTION REACHING THE FOURTH QW (DEPTH 450 nm). THIS IS A DISTANCE TWENTY TIMES THE PREDICTED $R_p$ OF 27 nm. ALSO NOTEWORTHY IS THAT THE SHAPE AND NUMBER OF IONS IN THE TAIL IS ESSENTIALLY INDEPENDENT OF THE DOSE FOR THE TWO DOSES SHOWN [FIG. 1(A) IS $5.5 \times 10^{13}$ IONS/cm$^2$; FIG. 1(B) IS $1.1 \times 10^{13}$ IONS/cm$^2$]. THIS EFFECT CAN BE UNDERSTOOD IN TERMS OF CHANNELING. EVEN FOR A 'PERFECTLY' ALIGNED BEAM, THERE IS A SUBSTANTIAL AMOUNT OF DECHANNELING OF THE Au IONS IN THE NEAR-SURFACE REGION. AS THE IMPLANTATION PROCEEDS, THE DAMAGE CREATED BY THE DECHANNELLED IONS INCREASES THE DECHANNELING RATE UNTIL NO FURTHER IONS ARE CHANNELLED DEEP INTO THE BULK. THIS SELF-LIMITING PROCESS LEADS TO A SATURATION OF THE DAMAGE CREATED AT GREATER DEPTHS, AND THEREFORE, TO A SATURATION IN THE SHIFT OF LUMINESCENCE, AS OBSERVED PREVIOUSLY.

As can be seen in Fig. 2(a), the general shape of the distribution of the 250 keV Au ions (expected $R_p \sim 51$ nm; $\Delta R_p \sim 18$ nm) implanted with the crystal aligned along the normal (100) channel 2(a), is similar to that of Fig. 1, with the mean range and the depth of penetration of the channelled ions shifted to greater depths because of the higher energy (250 vs 100 keV). Note, there is no evidence in these SIMS profiles of an increased yield of the Au negative ions due to matrix effects in either the InGaAs layers or the Si-doped substrate (0.9 $\mu$m). The intensities of the luminescence from the QW were greatly reduced, but not shifted in energy.

Upon annealing, the situation changes drastically, as can be seen in Fig. 2(b). First, Au has diffused throughout the entire epilayer into the substrate. The sudden increase in Au concentration observed at the epilayer/substrate interface (0.9 $\mu$m) is due to a change in the diffusion mechanism. This feature allows us to estimate that over one-third of the original implant has diffused into the substrate after only a 60 s anneal. Second, Au has accumulated in all four InGaAs QW. We were not able to obtain any significant luminescence intensity from the first two QW (for any implant discussed here) because of either complete disordering, and/or incomplete removal of nonradiative sites. Energy shifts in the PL were now observed from the third and fourth QW after annealing, indicative of changes in the composition and width (Table I). As observed previously, the energy shifts saturated after the 60 s annealing. The intensity was comparable to the pre-implanted levels. The question remaining at this point is, was the compositional disordering caused (enhanced) by the point defects created by the implantation of the Au ions, or was it simply due to the rapid thermal diffusion of the Au through the QW?

Comparing the depth distributions, after annealing, of the 4° off-normal implant and the aligned (100) axial implant [Figs. 3(a) and 3(b), respectively] it is clear that the

| TABLE I. Energy shift (meV) of QW luminescence after implantation-induced disordering. |
|------------------------------------------|------------------|------------------|
| Alignment                               | QW3 (250 nm)     | QW4 (450 nm)     |
| (as grown)                              | 1467.5 meV       | 1488.6 meV       |
| (100) axis                              | 10.8 meV         | 4.3 meV          |
| nominal 0°                              | 7.2 meV          | 3.2 meV          |
| 4° off-normal                           | 5.7 meV          | 1.1 meV          |
| 12° off-normal                          | 1.3 meV          | 0.9 meV          |
| nominal 0°, 20 nm Si$_3$N$_4$           | 1.1 meV          | 0 meV            |

FIG. 1. The depth profile of the 100 keV Au ions implanted using a FIB at nominal $0^\circ$ into the InGaAs/GaAs structure. The distribution was measured by SIMS using a 10 keV Cs$^+$ beam for sputtering while $^{197}$Au negative ions were detected. Two different doses are shown [curve (a) is $5.5 \times 10^{13}$ ions/cm$^2$; curve (b) is $1.1 \times 10^{13}$ ions/cm$^2$] to illustrate saturation of the channeling tail. The In data [curve (c)] are presented in arbitrary units, and serves to confirm the depth scale.

FIG. 2. The depth profile of 250 keV Au ions ($5 \times 10^{13}$ ions/cm$^2$) implanted into the test structure along the (100) channel before [curve (a)] and after [curve (b)] an RTA anneal at 850°C for 60 s. The epilayer/substrate interface is at 0.9 $\mu$m. Measurement conditions are similar to Fig. 1; again the In data [curve (c)] is in arbitrary units.
FIG. 3. The depth profile of 250 keV Au ions implanted into the test structure at 4° off normal [curve (a)] and along the (100) axis [curve (b)] after an RTA anneal at 850°C for 60 s. Measurement conditions are similar to Fig. 1; again the In data [curve (c)] is in arbitrary units.

extent of rapid-thermal diffusion is similar in both cases. This is evidenced by the comparable Au distributions at depths > 0.9 µm. Table I shows, however, that the energy shifts observed in the third and fourth QW are a strong function of the incident-implant angle, demonstrating that this rapid-thermal diffusion cannot be responsible for the observed disordering effects. As discussed previously, a change of the alignment angle between the Au-implantation beam and the crystal orientation drastically affects the depth distribution of the implanted ions. In Figs. 3(a) and 3(b), we observe significant differences in the amount of Au which has accumulated in the QW. For the 4° implant, the accumulation of Au and the shift in the PL energy in the fourth QW (Table I) is much smaller than that in the (100) implant. This result is consistent with the significantly reduced number of Au ions which reached the fourth QW during the original implantation at 4° (i.e., reduction of channeling). This resulted in fewer point defects being created in the vicinity of this QW and, subsequently, in less disordering upon annealing. The third QW of the 4° implant also shows the same trend (a reduced energy shift and Au accumulation) when compared to that of the (100) implant, but the differences are not as great. This again demonstrates that the amount of disorder depends on the amount of damage created by the original implant. As expected, these same observations were magnified for the 12° off-normal implant. Similar effects were observed for the sample with the order of QW reversed.

The channeling process should also be suppressed by implantation through an amorphous layer. For the implant through the 20 nm Si3N4 cap, the effects observed by either SIMS or PL are larger than a simple 30 nm shift of the 0° data. Significantly fewer ions channeled (due to the multiple scattering which occurs in the cap layer), with no accumulation of Au in the fourth QW (i.e., no disordering), and little in the third QW. Again, these results are consistent with the PL, which shows no change in the fourth QW, and very little in the third QW. As expected, the first and second QW were completely disordered.

We have clearly demonstrated that channeling effects must be taken into account in FIB implantation. Compositional disordering of the QW occurs only if implanted Au ions reach the layer during the implantation to create a reservoir of point defects. The disordering is not due to thermally diffusing Au.

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