TEM of Nano-LEDs made by laser writing

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Abstract. Using a focused laser beam, nanoscale current channels and nanoscale light emitting diodes (LEDs) have been made by the controlled thermal diffusion of manganese interstitial ions (Mnᵢ) out of a semiconductor heterostructure containing a layer of p-type ferromagnetic (GaMn)As. Here we use EDX, EELS and dark field imaging to probe directly the diffusion of Mn interstitials in the vicinity of the nano-LED region.

1. Introduction
Recently, it has been demonstrated how the controlled thermal diffusion of manganese interstitial ions (Mnᵢ) out of a semiconductor heterostructure containing a layer of p-type ferromagnetic (GaMn)As can be used to create nanoscale current channels [1-2] and nanoscale light emitting diodes (LEDs) [3]. Our devices are p-i-n resonant tunnelling LEDs with a two-dimensional AlAs/GaAs/AlAs quantum well embedded in the intrinsic (i) layer. This is sandwiched between a bottom n-type GaAs contact layer and a top p-type layer of Ga₁₋ₓMnₓAs with x=5%. By annealing a small spot with a focused laser beam, we are able to create a preferential path for charged carriers when a forward bias is applied to the p-i-n diode, thus activating a nanoscale region of the device to emit light at a bias well below the threshold voltage for emission from the non-annealed regions. The technique also provides real-time in situ characterisation and control of the nanoscale current channels during their formation and allows us to produce an ordered array of LEDs or else a shaped, light-emitting region with submicron precision [3].

2. Experimental
For TEM analysis, we selected a region annealed with a focused laser beam of diameter <1000nm, wavelength λ=515nm and power P=40mW. These laser annealing conditions produced visible surface effects, allowing accurate location of the annealing region in the FIB-SEM, while producing enhanced photoluminescence and electroluminescence in close proximity to the annealed region. This allowed us to probe how the material composition changes with increasing distance from the laser spot, i.e. with decreasing annealing temperatures, which we estimate to be >500°C inside the laser spot and decreasing to Tₐ<100°C at distances d>5000nm.

In preparing the samples for TEM analysis, it is important to minimize amorphisation of the sample and further diffusion of the Mn interstitials. Lift-out samples were initially prepared for TEM analysis.
from a specific microscopic area containing a nano-LED region in an FEI Quanta 200 3D. These were attached at one side to an Omniprobe support and were then further FIB milled, finishing at 10kV, 16pA. The samples were plasma cleaned for ten minutes in an inductively couple plasma of Ar/25% O$_2$. The sample was then further milled in a Fischione 1010 argon ion miller, finishing at 500V, 5mA. Samples were further plasma cleaned for five minutes immediately prior to TEM analysis.

The Mn-distribution within and outside the active region of the LED was analysed using EDX, EELS and 002 dark field imaging in a JEOL 2100F equipped with an Oxford Instruments INCA energy TEM 250 EDX system and a Gatan Tridium image filter and Gatan Orius camera.

3. Results and discussion

TEM analysis reveals a significantly altered region comprising a complex structure of amorphous and polycrystalline material directly under the annealing point, of the order of ~400nm wide and ~200nm deep in the case of a 40mW laser anneal (Figure 1a). The transition from heavily restructured to apparently intact GaAs structure appears to be fairly abrupt, with the quantum well structure still clearly visible within ~50nm of the restructured material (Figure 1b).

![Figure 1](image1.png)

**Fig. 1.** a) Bright field TEM image showing the extent of the structurally altered region (arrowed) directly under the laser during annealing. b) HRTEM image showing that the device structure is largely preserved immediately adjacent to the region structurally altered by the laser annealing.

Elemental mapping using EDX reveals that there has been significant evaporation of As from the structure at the immediate point of the laser anneal. The Ga and Mn signals in this heavily restructured region correspond to a strong O signal, indicating a high level of oxidation. However, an Al-rich layer is still clearly visible, albeit shifted slightly towards the substrate, and appears to correspond to a region with a low level of oxidation (see Figure. 2).

In a region of ~50nm at the edge of the highly damaged region, there has been strong diffusion of Mn towards the surface, where EDX indicates that it has been oxidised. There is no evidence of
significant diffusion of other elements in this region, with the AlAs/GaAs/AlAs interfaces remaining clearly defined. However, within ~100nm of the edge of the significantly amorphised region, the overall Mn distribution is comparable to that of regions several hundred nm from the annealed region (see Figure 3). Below the AlAs barrier layers, the level of Mn appears to be generally below the detectable limits in this EDX system, both near to the anneal and at some distance.

**Fig. 2.** EDX maps of the interface between the damaged region and the intact, active device structure. DF-STEM image is 365 nm across.

**Fig. 3.** EDX maps acquired at ~500nm from the edge of the annealed region. Gradation can be seen in the Mn composition, increasing to the surface of the sample. DF-STEM image is 125nm across.

002 dark field imaging has the potential to probe the distribution of Mn interstitials specifically, as the structure factor is not significantly affected by substitutional Mn, but is strongly reduced by the energetically favoured type 2 interstitials (four As nearest neighbours)[4]. However, for analysis over
a larger area this technique requires a flat sample with little buckling, meaning that it can be sensitive to sample preparation. 002 dark field analysis of a ~250nm thick lift out sample shows a darker intensity region both above and below the quantum well structure, extending ~500nm from the edge of the amorphised region. Noting the relatively high temperature \( (T_A >500^\circ C) \) required to create the amorphised region of GaAs and the characteristic temperature for the Mn, diffusion \( (T_A <200^\circ C \ [1]) \), the extended dark regions in Figure 3 could be associated with the diffusion of interstitial-Mn from the top (GaMn)As layer into the underlying structure. Further corroborative evidence is required before this can be firmly concluded.

![Image](image_url)

**Fig. 3.** 002 Dark Field image, with the annealed region to the right. The darker region to the right of the image is a thickness artefact. Some surface damage to the sample may have occurred during ion beam milling.

### 4. Conclusions

The microstructure and elemental compositions of a nanoLED created by laser-induced thermal annealing have been appraised. At 40mW, the laser has produced a central largely amorphised region depleted of As, surrounded by a 50nm wide region where Mn has diffused towards the surface without otherwise affecting the structure of the material. 002 Dark Field shows darker intensity below the QW structure and over a few hundred nm from the amorphised region, which may indicate the presence of interstitial Mn, but requires further corroborative evidence.

### References

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