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Effect of erbium doping on the defect structure of GaSb crystals

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Abstract. GaSb single crystals with different Er concentrations have been studied by cathodoluminescence in the scanning electron microscope. Low Er doping has been found to reduce the concentration of native acceptors. In crystals with higher Er concentrations, Er–Sb precipitates form and doping becomes less efficient in suppressing the acceptors. In these samples intraionic Er luminescence is observed.

1. Introduction

The interest of rare earth doping of semiconductors has increased in the past years owing to its possible applications in optical devices requiring temperature stability, such as semiconductor lasers and optical amplifiers. Advantages of these systems are the presence of a sharp and temperature-independent rare-earth luminescence and the possibility of activation of the emitting rare-earth centres by minority carrier injection. The luminescence peaks are due to the intra-4f-shell transitions in the rare-earth ions and its high stability with temperature is related to screening of the crystal field by the outer levels. In the case of erbium, one transition between 4f levels corresponds to an energy of about 805 meV which is in the region of minimum transmission loss in silica-based optical fibres. The 800 meV intracentre transition of erbium ions has been reported for different Er-doped III–V compounds such as GaAs, InP, InGaP or GaN [1–5]. Er-doped GaSb layers have been investigated by Sun and Wu [6] and Sun *et al* [7] who reported a lowering of carrier concentration due to suppression of native acceptors in the presence of erbium. As-grown GaSb is always p type in nature owing to the native acceptors which are complexes formed by a gallium vacancy and a gallium antisite. The reduction of acceptor concentration is a subject of technological interest for the development of GaSb-based devices. Since the native acceptors are responsible for a luminescence emission at about 777 meV, which is usually called band A, luminescence techniques are useful tools to investigate the presence or evolution of acceptors in GaSb. In the case of Er-doped GaSb, luminescence enables the study of the influence of Er ions on acceptors as well as the emission properties of the dopant ions. In [6] and [7], photoluminescence spectra dominated by free and bound exciton lines were reported.

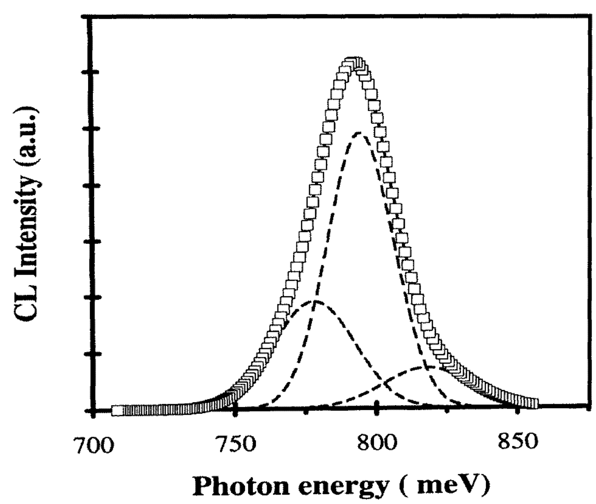

Figure 1. Typical CL spectrum of an undoped sample at 78 K. Points are experimental data and broken lines show best fit.

In the present work the effect of erbium doping on the luminescence and on the native acceptors of GaSb crystals has been investigated by cathodoluminescence (CL) in the scanning electron microscope (SEM). This technique which provides information on the nature and spatial distribution of recombination centres has been previously applied to characterize GaSb crystals [8–10].

2. Experimental method

The Er-doped GaSb crystals were grown by the vertical Bridgman technique with Er concentrations in the melt of 0.05%, 0.1% or 0.4% by volume. The crystals were 12 mm in diameter and about 40 mm in length. Wafers of

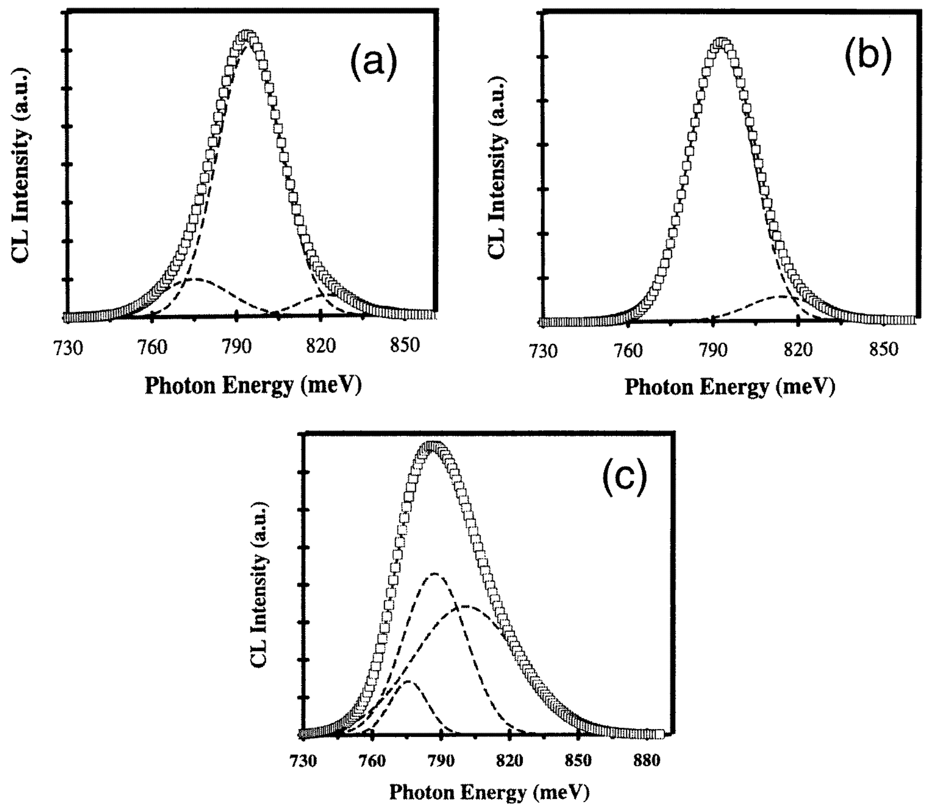

Figure 2. CL spectrum of (a) sample 25, (b) sample 26 and (c) sample 27 at 78 K. Broken lines show Gaussian bands that best fit the experimental data.

equivalent positions of each sample were cut perpendicular to the growth axis. The samples were labelled 25, 26 and 27 corresponding to the 0.05%, 0.1% and 0.4% concentrations respectively. Wafers were chemomechanically polished to a mirror finish and observed in a Hitachi S-2500 SEM at 78 K and at accelerating voltages of 20–30 keV in the secondary-electron and the CL modes. The details of the experimental setup for CL measurements have been described elsewhere [11]. In order to study the composition of some of the features observed in SEM images, x-ray microanalysis for Er, Ga and Sb was performed in a Jeol JXA-8900 M superprobe.

3. Results and discussion

Figure 1 shows the CL spectrum of an undoped GaSb sample. The dominant emissions correspond to the near-band-edge emission at 796 meV and the above-mentioned band A of native acceptors at 777 meV. Typical CL images of undoped samples reveal the presence of subgrain boundaries as dark lines in a brighter, almost featureless background [12]. Er doping has been found to increase the total CL intensity and to cause spectral changes. Figure 2 shows CL spectra of the three samples used in this work. The spectra, which are well reproducible all over the samples, have been deconvoluted and the best fits obtained. Sample 25, corresponding to the low Er concentration, presents three CL bands peaked at about 777 meV, 795 meV and 815 meV. Band A appears with a

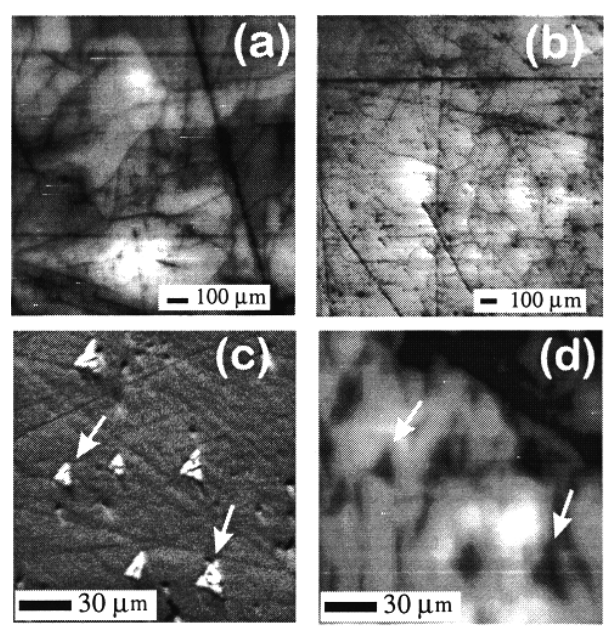

Figure 3. Representatives CL images of (a) sample 25 and (b) sample 26, (c) secondary electron image of an area of sample 27 with precipitates and (d) CL image of the area shown in (c). Arrows mark some of the precipitates appearing in micrographs (c) and (d).

lower intensity as in undoped samples, showing the erbium-related reduction of native acceptors in agreement with the results reported in [6] and [7]. Weak above-band-gap energy emission, as the 815 meV band observed here, has been previously suggested [9] to be related to tail states and shallow acceptors. Further reduction of acceptors with increasing Er concentration is clearly observed in the spectrum of sample 26 (figure 2(b)) in which band A is absent. However, when the Er concentration further increases band A reappears, as the spectrum of sample 27 (figure 2(c)) shows. In this case the CL spectrum is broader and is decomposed into emissions at 777 meV, 795 meV and 800 meV which correspond to band A, band-to-band transitions and Er intraionic transitions respectively. The fact that in sample 27 the acceptor band is present can be explained by Er precipitation processes that reduce the effective number of ions able to interact with native acceptors. This result has been confirmed by Hall measurements of carrier concentration. An increase in hole concentration from 1.2×10^{18} cm^{-3} in sample 26 to 2.5×10^{19} cm^{-3} in sample 27 has been found. On the other hand this result indicates that a relatively high threshold of Er concentration in GaSb is necessary for a clear detection of the Er ion related CL.

CL images of the three samples show differences in the spatial distribution of luminescence and provide information on the precipitation processes as a function of Er concentration. Figure 3(a) shows a representative CL image of sample 25. As in undoped samples, the main CL contrast is due to the existence of subgrain boundaries. In samples 26 and 27 (figures 3(c) and 3(d)) precipitates are observed in topographic SEM and in CL images. Secondary electron images show triangle-shaped precipitates usually with the same geometric orientation (figure 3(c)). CL images of sample 26 (figure 3(b)) reveal the precipitates as dark triangular regions and show a subgrain structure with a smaller size than in sample 25. The sample with the highest Er concentration (27) shows more inhomogeneous CL images with the presence of precipitates and marked differences in intensity level of the background. The subboundaries in this sample show a not very well defined CL contrast. These differences are attributed to an inhomogeneous distribution of erbium.

In order to investigate the nature of the Er-related precipitates x-ray microanalyses were performed at the precipitates and in the background area. In the precipitate-free regions, erbium was not detected (concentration below the detection limit) and the composition was found to be nearly stoichiometric with a slightly increased Sb composition. On the contrary, the triangular precipitates were found to be composed of Er and Sb. A possible compound could be Er_5Sb_3 which has a hexagonal structure and its formation has been concluded to have taken place in [6] from x-ray photoelectron data from GaSb:Er layers.

4. Conclusions

In conclusion, GaSb crystals with different Er concentrations have been grown by the vertical Bridgman technique. CL in the SEM shows that Er causes an increase of the total luminescence emission as well as spectral changes which depend on the erbium concentration. At moderate erbium doping the native acceptor concentration, as detected by the luminescence band A, decreases. At high Er concentrations doping does not appear to be as efficient in suppressing native acceptors, which is explained by the formation of Er–Sb precipitates. Emission due to intraionic transitions of Er is observed in the CL spectra of highly doped crystals. In this case, Er-doped GaSb appears to have interesting applications for light-emission devices in which the rare-earth element would play an active role as the emission centre.

Acknowledgments

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