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Scanning tunneling spectroscopy study of erbium doped GaSb crystals

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Er doped GaSb single crystals have been studied by scanning tunneling spectroscopy (STS) and cathodoluminescence (CL) in a combined scanning electron microscope–scanning tunnelling microscope system. The surface band gap in doped samples has been found to be about 0.5 eV while in undoped crystals the gap is close to the bulk value. Inhomogeneities in the local electronic properties of the doped crystals are studied by a correlation of the CL images and STS data.

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I. INTRODUCTION

Rare-earth doping of semiconductors has potential applications in optical devices requiring temperature stability, such as semiconductor lasers. In these systems a sharp and temperature-independent rare earth luminescence is present due to the intra- $4f$ -shell transitions in the ions. The rare-earth centers can be activated by minority carrier injection. In the case of erbium, one transition between the $4f$ levels corresponds to an energy of about 800 meV which is in the region of minimum transmission loss in silica-based optical fibers. The 800 meV transition has been reported for different Er-doped III–V compounds.^{1–5} In the case of GaSb the effect of erbium has been studied by photoluminescence^{6,7} and by cathodoluminescence (CL) in the scanning electron microscope (SEM).⁸ In Refs. 6 and 7 photoluminescence spectra dominated by exciton lines were reported. CL microscopy⁸ showed that at moderate erbium doping the native acceptor concentration decreases while at high Er concentration doping is not as efficient in suppressing acceptors due to the formation of Er–Sb precipitates. It appears that variations of Er concentration influences the local electronic structure of GaSb. This is for instance detected by the changes of the acceptor related A band at 777 meV observed in CL.

The SEM-based techniques used to investigate electronic recombination in semiconductors, as CL, have space resolution in the micron or submicron range. On the other hand STM and STS enable us to investigate the structural and electronic properties of semiconductors with higher resolution. In particular, spatially resolved spectroscopic methods as current imaging tunneling spectroscopy (CITS)⁹ provide images that reveal nanometer scale variations in the surface electronic structure of the samples.¹⁰ In some cases it appears of interest to study the electronic properties of the sample with SEM and STM techniques and to obtain correlative information with different orders of magnitude of magnifica-

tion and of space resolution. The use of a combined SEM–STM instruments is particularly suitable for such correlative studies. In the present work a SEM–STM system, consisting of a STM implemented in the chamber of a SEM prepared for CL detection, has been used to study local electronic properties of erbium-doped GaSb crystals.

II. EXPERIMENT

The pure and Er-doped GaSb crystals were grown by the vertical Bridgman technique with Er concentration of 0.4 wt% in the melt. The crystals were 12 mm in diameter and about 40 mm in length. Wafers were cut perpendicular to the growth axis. The wafers were chemomechanically polished to a mirror finish and observed in a Hitachi S-2500 SEM or a Leica 440 SEM at accelerating voltages of 20–30 kV in the secondary-electron and the CL modes. Details of the experimental setup for CL measurements have been described elsewhere.¹¹ For the STM measurements the combined SEM–STM based on a Leica 440 SEM operating under a vacuum of 10^{-6} Torr was used. The small size of the STM enabled it to be mounted on the SEM specimen holder. The main features of this system are similar to the ones previ-

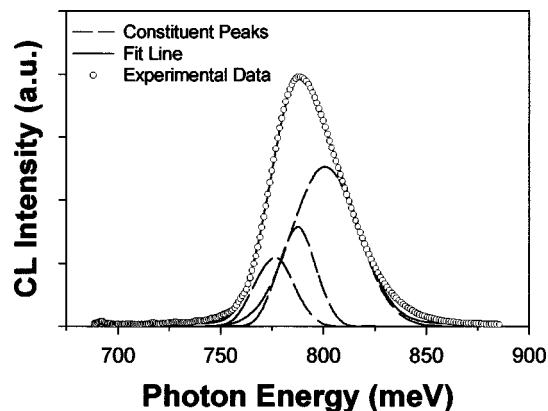


FIG. 1. Typical CL spectrum of GaSb:Er at 90 K. Dots correspond to experimental data, and dashed lines show the fit to 777, 794, and 800 meV bands, respectively.

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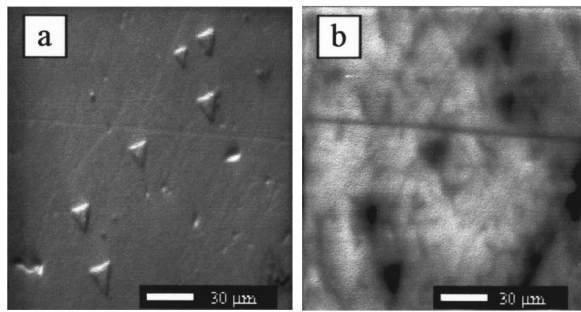


FIG. 2. (a) Secondary electrons image of the sample doped with erbium showing triangular precipitates. (b) CL image of the same area. Precipitates appear with dark contrast.

ously described in Ref. 12. Electrochemically etched or mechanically sharpened Pt–Ir and Au wires were used as STM probe tips. For CITS measurements the constant current topograph was obtained in a 128×128 pixel grid, the feedback loop interrupted for 2 ms, and the voltage digitally ramped from the tunnel voltage to a set of 44 predetermined values while the current was sampled. This provides 44 tunneling current files at different voltages in addition to the current constant topograph. To analyze the data, the normalized differential conductance, $(dI/dV)/(I/V)$, spectra were used. This quantity provides a rather direct measure of the surface density of states.¹³ For the STM measurements the samples were cleaned with hydrochloric acid and acetone to remove oxides, dried in a flow of dry nitrogen, and immediately mounted in the microscope chamber.

III. RESULTS AND DISCUSSION

The CL spectra of the undoped samples show the band to band transition at 796 meV and the transition at 777 meV due to native acceptors.¹⁴ Typical CL images of the undoped samples reveal the presence of subgrain boundaries in a brighter, almost featureless background.¹⁵ CL spectra of the doped samples show the bands present in the undoped samples and a band at 800 meV, which corresponds to Er intraionic transitions (Fig. 1). Secondary electron images of these samples show triangle shaped precipitates usually with the same geometrical orientation [Fig. 2(a)] which in the CL images appear as dark regions in an inhomogeneous back-

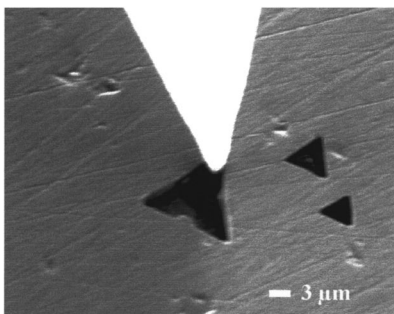


FIG. 3. SE image of an electrochemical tip on a precipitate of erbium.

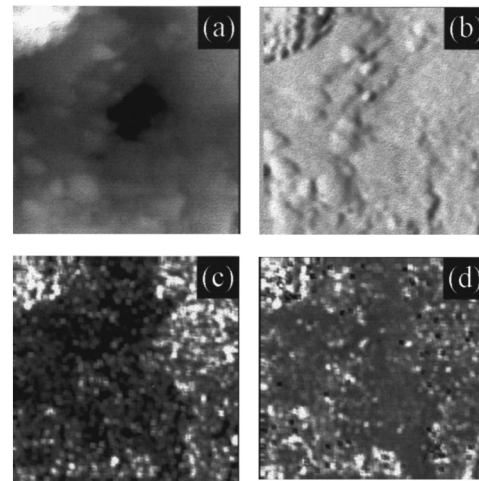


FIG. 4. $500 \times 500 \text{ nm}^2$ STM image of a GaSb:Er sample. (a) Topography image acquired with a sample voltage of +0.9 V and 0.7 nA tunneling current. (b) Corresponding image in derivate. (c)–(d) Corresponding CITS images obtained at +1.35 V (c) and -1.35 V (d) sample voltages.

ground [Fig. 2(b)]. The precipitates have been previously found by x-ray microanalysis measurements to be composed of Er and Sb.⁸

The SEM–STM combined system enables us to perform STM spectroscopic measurements in regions with different electronic recombination properties as revealed by SEM–CL images. Such correlation has been previously carried out in II–VI compound semiconductors.¹⁶ In the present case the nonemitting regions correspond mainly to the precipitates revealed in the SE images so that the STM tip could be selectively located on a precipitate or a precipitate free zone while observing the sample in the SE mode (Fig. 3). STM current constant and CITS images were recorded in both undoped and doped samples. CITS images of all samples show contrast due to local conductance variations with high spatial resolution. This contrast appears for voltage values below and above the bulk band gap and is higher in the doped samples. Figure 4 shows CITS images of a doped sample. Further information on the sample inhomogeneity is obtained from the normalized differential conductance curves. The

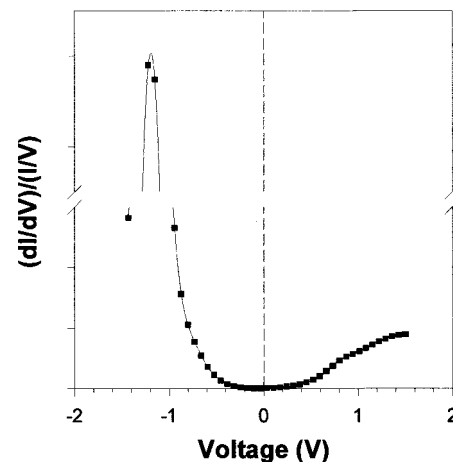


FIG. 5. Normalized differential conductance spectrum from an undoped sample of GaSb.

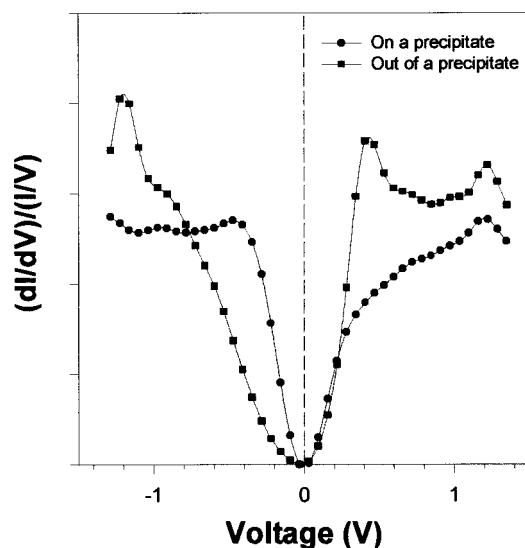


FIG. 6. Typical normalized differential conductance spectra from a Er doped sample: (a) from a free precipitates area and (b) from a precipitate.

curves were plotted from the data of the CITS images corresponding to the 44 selected voltage values by averaging the data on small regions. Figure 5 shows a curve of one of our undoped samples, in which a surface band gap of about 800 meV, close to the bulk value of 796 meV measured in the CL spectra, is observed. The *p*-type behavior of as-grown undoped GaSb is revealed by the asymmetry of the curve relative to the sign of the sample voltage.

Figure 6 shows normalized differential conductance curves of the Er-doped samples. Curve (a) recorded on a precipitate free area shows an energy gap of about 0.5 eV, which is lower than the value measured in the undoped sample. The influence of erbium impurities on the surface band gap is possibly due to local changes of stoichiometry or defect structure. On the contrary, the presence of erbium does not influence the bulk band gap value as revealed by the corresponding emission band in the CL spectra. Curve (b), showing an almost metallic behavior, was recorded on one of the triangular precipitates. Slight variations of the shape of the curve and of the measured gap relative to curve (b) are

obtained in different areas of a given precipitate. This indicates that the precipitates have nanoscaled, probably compositional, inhomogeneities.

IV. CONCLUSIONS

In summary, the effect of high Er concentration on the electronic properties of GaSb has been studied with a SEM–STM instrument. The presence of erbium causes the appearance of a CL band related to intraionic transitions but does not change the bulk band gap as measured from the CL spectra. STM–CITS reveals local differences in the surface band gap value with high spatial resolution. In particular an erbium induced reduction of the surface band gap, to a value of 0.5 eV, in precipitate free areas and the nearly metallic behavior on Er–Sb precipitates have been observed.

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