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Local stress measurements in laterally oxidized $GaAs/Al_xGa_{1-x}As$ heterostructures by micro-Raman spectroscopy

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Lattice deformation induced in surface GaAs layers by the selective lateral oxidation of buried $Al_xGa_{1-x}As$ layers was determined from the energy shift of the GaAs phonon line in Raman spectra excited with a focused laser beam. The procedure included a correction for the laser beam induced heating effects. The surface GaAs layer was found under small tensile stress in the oxidized regions of our samples with respect to the unoxidized regions. The deformation is 8×10^{-4} and is the same, within experimental error, for heterostructures incorporating pure AlAs layers or $Al_{0.98}Ga_{0.02}As$. Strong differences in Raman efficiency, as well as in photoluminescence efficiency, were observed for different samples, which are discussed in terms of the GaAs/oxide interface nonradiative recombination efficiency. © *1997 American Institute of Physics.* [S0003-6951(97)04043-6]

The technique of selective wet oxidation of buried layers in $GaAs/Al_{1-x}Ga_xAs$ heterostructures has recently received much interest because of its potential to incorporate insulating oxide regions with good structural, electronic, and optical properties in various types of heterostructure devices.[1] In particular, vertical-cavity surface emitting lasers have been realized with such buried oxide layers acting as current limiting as well as optical confinement regions.[2] These devices have shown an important reduction of the threshold current in parallel with enhancement of the conversion efficiency, associated with the incorporation of the buried oxide layers.[3–5] This approach has also been proposed for the fabrication of GaAs-based metal-oxide-semiconductor field effect transistors,[6] large bandwidth Bragg mirrors,[7] optical elements (e.g., micro-lenses[8]), and optical waveguides.[9]

Among the various issues which are still not resolved with respect to this technique, the question of the strain induced in the layers is important, as on one hand it may affect the performance of devices[2] or their lifetime and on the other hand it may play a role in the oxidation process itself (modification of the oxidation rate[10]).

Comparing the Al atomic densities in AlAs and in the γ-Al_2O_3 phase which has been reported to result from oxidation,[11,12] an important thickness contraction of up to 20% is expected for the oxidized layers.[2] Indeed, from the direct measurements performed on transmission electron micrographs of such samples,[2,13] a thickness contraction of 10%–20% is deduced. Stress induced by the lateral oxidation manifests itself as a mechanical instability of the multilayers for oxidized thicknesses of more than 300 nm. Oxidized GaAs/AlAs multilayers were also shown[2] to be mechanically unstable under thermal processing. Better stability to thermal processing is found for layers containing a small concentration (e.g., 2%) of Ga.[2] This behavior was attributed[2] to the smaller linear contraction measured in oxidized $Al_{0.98}Ga_{0.02}As$ layers, compared to AlAs layers. Evidence of stress effects was also found in the oxidation kinetics.[10] However, to the best of our knowledge, no direct measurement of strain in oxidized GaAs/AlAs multilayers was reported yet.

In this work, we have directly measured the strain induced in a GaAs layer by the lateral oxidation of an underlying AlAs or $Al_{0.98}Ga_{0.02}As$ layer. This was done by recording the energy shifts of the longitudinal optical (LO) phonon lines in inelastic light scattering spectra. This approach has already been applied extensively to the investigation of the local stresses induced in Si integrated circuits by the various processing steps, e.g., by the field oxide formation in local oxidation structures (LOCOS) (for a review of this topic, see Ref. 14).

The samples investigated were grown by molecular beam epitaxy on semi-insulating (100) GaAs substrates. A GaAs buffer layer (300 nm) is first grown, followed by an AlAs layer (thickness: 100 nm) for sample No. 1, or an $Al_{0.98}Ga_{0.02}As$ layer (100 nm) for sample No. 2. The structure is terminated by a surface GaAs layer (100 nm). All these layers were nominally undoped. The layer thicknesses were determined from reflection high energy electron diffraction (RHEED) oscillations before growth. These values are accurate to within 5%.

Sample processing consisted in defining ridges (by reactive ion etching) 220 μm wide, and approximately 0.3 μm deep. The lateral oxidation of the Al-rich layer was then performed at 430 °C, with N_2 as the carrier gas passing through a H_2O bubbler maintained at 95 °C. Oxidation times were 80 min for sample No. 1 (AlAs layer), and 60 min for sample No. 2 ($Al_{0.98}Ga_{0.02}As$ layer). The corresponding oxidation lengths, determined by observation of the optical contrast, were 80 and 32 μm, respectively. In all the samples the central part of the ridge was unoxidized.

The micro-Raman measurements were made on the top (001) surface, in backscattering geometry. The scattering condition was $z(x,y)\bar{z}$. Under these conditions, only the LO phonon line can be detected. This line occurs near 291 cm^{-1} on unstrained GaAs. Assuming a bi-axial stress symmetry ($\sigma_{xx}=\sigma_{yy}=\sigma$ and $\sigma_{zz}=\sigma_{xy}=\sigma_{yz}=\sigma_{zx}=0$, z being the growth direction), the GaAs–LO phonon line shift is given by[15]

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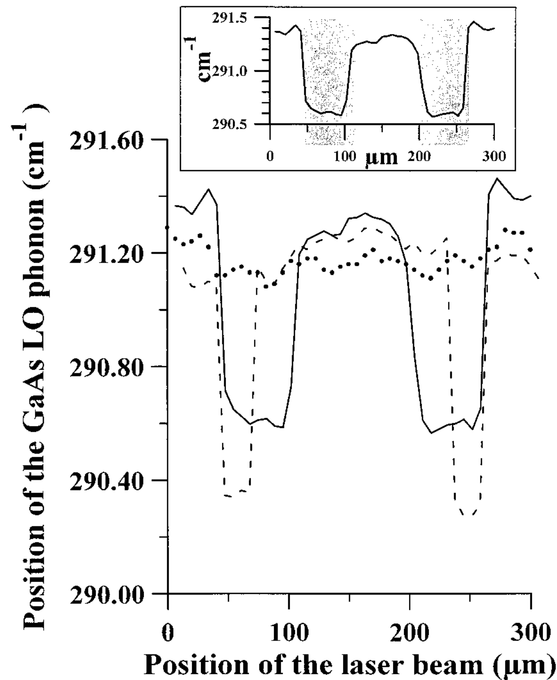

FIG. 1. Energy shift of the GaAs-LO phonon (raw data) recorded on line scans across sample No. 1 with $x_{Ga}=0$ (full line) and sample No. 2 with $x_{Ga}=2\%$ (dashed line). The evolution measured across sample No. 1 before oxidation is also shown (dots). In the inset, the GaAs-LO peak shift is superposed to an optical micrograph of the oxidized ridge on sample No. 1 (top view). Dark regions are the oxidized ones.

$$\Delta\omega_{LO}(\mathrm{cm}^{-1}) = -3.9\times\sigma(\mathrm{GPa}) = 0.49\times10^3\epsilon_{\parallel}.$$

The spectral resolution used for the measurements is $3.6\ \mathrm{cm}^{-1}$, and the spectral interval between pixels on the charge coupled device (CCD) detector $\approx 0.5\ \mathrm{cm}^{-1}$. A Gaussian/Lorenzian line shape is used for the fitting procedure. Reproducibility tests yield a root mean square value of $0.1\ \mathrm{cm}^{-1}$ for the shifts of the phonon line from its average position.

The absorption length of the 514 nm Ar laser line (≈ 100 nm) ensures that the signal originates only from the surface GaAs layer, and not from the GaAs buffer.

However, local heating by the focused laser beam yields another contribution to phonon peak shifts (mainly due to thermal expansion). The corresponding ω_{LO} variation is $\approx -1.75\times10^{-2}\ \mathrm{cm}^{-1}\times\mathrm{K}^{-1}$.[16] To take into account this heating effect, we have introduced a correction procedure described further. This correction procedure also yields information on the nonradiative recombination properties of the interfaces as we will show.

Figure 1 shows the GaAs–LO phonon peak shift as a function of the laser beam position across sample No. 1 (full line) and sample No. 2 (dashed line). The peak variation across an unoxidized ridge (sample No. 1, dotted line) is also shown. These measurements have been performed with the laser beam at 10 mW outside the spectrometer which converts to $\approx 70\ \mathrm{kW/cm}^2$ at the sample surface. The data on Fig. 1 do not include any correction for the heating effects. A downward shift of the LO peak ($0.7-1\ \mathrm{cm}^{-1}$) is seen on the oxidized region on both samples. The shape and spectral width of the Raman lines, on the other hand, do not change at all, indicating homogeneous stress (except in a small region—less than 5 μm—around the oxidation front).

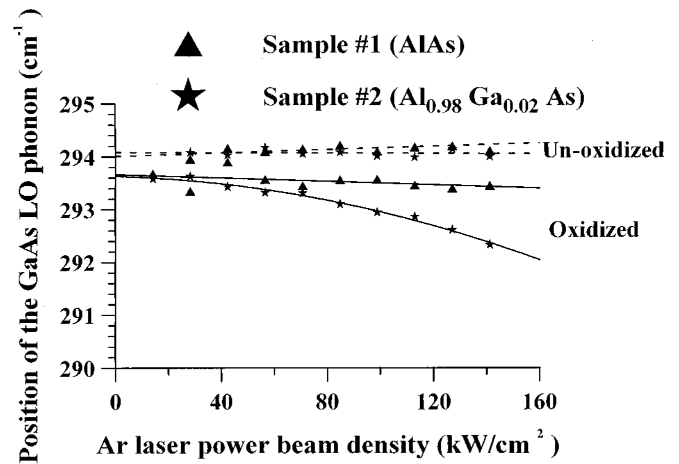

FIG. 2. Energy shift of the GaAs-LO phonon with the beam power density. Fits to the data (with 2nd order polynomials) are also shown: full lines for the oxidized region, dashed lines for the central unoxidized region.

To separate the heating effect, we have analyzed the pump power dependence of the shift on two different locations on each sample (oxidized and unoxidized regions). For these measurements, we have adopted the procedure suggested in Ref. 14 which consists of allowing a plasma line from the Ar laser to be detected simultaneously with the phonon line on the Raman spectra. This plasma line is used for the absolute calibration of the spectrometer for each individual measurement.

Figure 2 reports the peak positions as a function of the laser power density. For the measurements in the central area (unoxidized), the peak shift is very small for both samples. On the other hand, in the oxidized region, a stronger variation (up to $2\ \mathrm{cm}^{-1}$) is observed for sample No. 2, whereas for sample No. 1 the variation is still small. This fact is related to a strong difference in local heating of the two samples. However, when extrapolating the two sets of curves to zero power density, the same value is obtained for the differential peak shift (oxidized region—bare region) for both samples: $0.4\pm0.05\ \mathrm{cm}^{-1}$, corresponding to $8\pm1\times10^{-4}$ tensile deformation induced in the GaAs layer by oxidation. This means that, contrary to what is generally believed, oxidation of $Al_{0.98}Ga_{0.02}As$ layers induces the same strain as oxidation of pure AlAs layers. We confirmed that our samples do show a better resistance to delamination during rapid thermal processing with the 98% composition compared with the pure AlAs case, as reported previously.[2]

The corresponding integrated intensities of the LO phonon lines are given in Fig. 3. For sample No. 1, similar intensities are obtained in the oxidized and unoxidized regions. The intensities measured for sample No. 2 are much higher than for sample No. 1. In addition an increase by a factor 2 is seen from the unoxidized to the oxidized region. These effects cannot be attributed to uncertainties in the layer thicknesses nor to changes in optical reflectivity of the layers (because of the amount of absorption in the surface GaAs layer). They are not due to Raman resonance effects either, since the wavelength chosen is far from any critical point in the electronic structure of GaAs.[17] The room tem-

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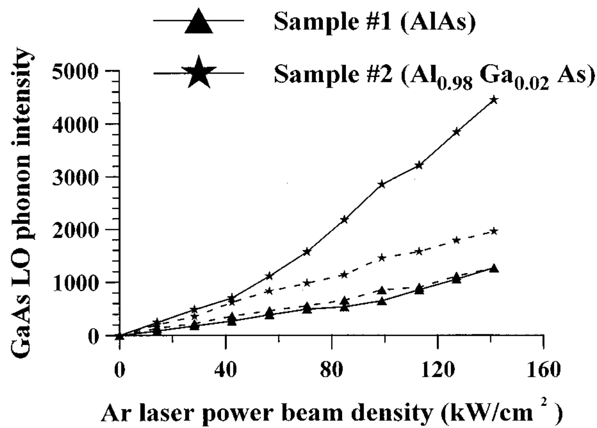

FIG. 3. GaAs-LO phonon integrated intensity as a function of the beam power density (full lines) oxidized region, (dashed lines) central unoxidized region.

perature photoluminescence (PL) spectra were simultaneously measured on the same spectrometer. We observe that the PL intensity is inverse-correlated to the LO phonon peak intensity. The unoxidized region of sample No. 1 shows an enhanced signal relative to that of sample No. 2 by $\approx 20\%$. For the oxidized regions, the PL signal is higher by a factor of 2 on sample No. 1. However, the PL intensity on one given sample drops when going from the unoxidized to the oxidized region. In these measurements, there is no contribution of the GaAs substrate and buffer to the PL signal due to the presence of the highly recombining substrate/buffer interface, so that the observed PL signal originates only from the top 100 nm GaAs layer.

These results can be directly interpreted in terms of nonradiative recombination at the interface between the GaAs surface layer and the underlying oxide (thereafter called ''top'' interface). It is clear that enhanced nonradiative recombination at this interface causes reduction of the PL intensity.

As for the Raman lines, the effect of nonradiative recombination at one interface is to increase the depleted layer thickness, and thus to increase the LO-phonon intensity.[18] We explain the PL intensity reduction and Raman line intensity increase in oxidized regions compared to unoxidized regions by enhanced nonradiative recombination at the top oxidized interfaces. The large increase in LO phonon intensity and large decrease in PL intensity observed for sample No. 2, compared to sample No. 1, can be attributed to the enhanced nonradiative recombination velocity at the top oxidized interface. This nonradiative recombination also explains the stronger heating effect observed in the oxidized regions of sample No. 2 (Fig. 2). The reason for this degradation of the top interface on sample No. 2 is not clear and might not be a general feature of pure AlAs vs $Al_{0.98}Ga_{0.02}As$ interfaces. Indeed, observation of this sample under the optical microscope after growth showed a slight anisotropic roughness, probably originating from nonoptimal growth conditions of the $Al_{0.98}Ga_{0.02}As$ layer. Lateral oxidation appears to reinforce the nonradiative recombination efficiency at this slightly rough interface.

In summary, we have measured the strain induced in a GaAs layer by the lateral oxidation of an underlying AlAs or $Al_{0.98}Ga_{0.02}As$ layer. We found the same magnitude ($8 \pm 1 \times 10^{-4}$ as compared to the GaAs layer in unoxidized regions) for both types of sample within experimental error. This demonstrates that, with respect to lateral oxidation, the difference observed between the two compositions is not related to strain effects, but rather to the chemical nature of the GaAs/oxide interfaces.

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