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Reduction of etched AlGaAs sidewall roughness by oxygen-enhanced wet thermal oxidation

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The authors demonstrate that the oxidation smoothing of sidewall roughness of dry-etched $Al_{0.3}Ga_{0.7}As$ ridge structures is enabled through a modified wet thermal oxidation process which involves the addition of dilute amounts of O_2 to the water vapor ambient. High magnification cross-section and top-view scanning electron microscope imagings both before and after oxide removal clearly show a substantial reduction of photolithography- and dry-etching-induced sidewall roughness (from $\sigma \sim 100$ nm down to $\sigma \sim 1-2$ nm), occurring only with the participation of added O_2. The smoothing process provides means to realize high-index-contrast GaAs-based optical waveguides with both low bend and scattering losses. © *2007 American Institute of Physics*. [DOI: 10.1063/1.2766859]

The scattering of light from rough sidewall surfaces in semiconductor optical rib waveguides is a primary source of wave propagation loss.[1–3] Photolithography and anisotropic dry-etching fabrication processes[4,5] transfer photo mask line-edge roughness and may introduce additional roughness to the etched sidewalls used to define the lateral effective index contrast, Δn_{eff}, required to confine an optical mode. Theoretical and experimental studies of scattering loss α (Refs. 1, 2, and 6–10) show that α typically scales with $(\Delta n_{\mathrm{eff}})^2$ or $(\Delta n_{\mathrm{eff}})^3$, σ^2, and $1/d^4$, where σ and d represent the root-mean-squared surface roughness and waveguide horizontal dimension, respectively. To achieve high-density photonic integration, a high index contrast (HIC) is required to achieve small radius of curvature bent waveguides with low bend loss, which in turn necessitates a reduction in d to maintain single mode operation. Given the clear fundamental tradeoff between reduced bend size and increased scattering loss,[3] minimizing the sidewall roughness σ becomes perhaps the most critical challenge to achieving a viable photonic integration technology.

There has been a tremendous level of research recently in silicon-based HIC (or "high delta," for high Δn) waveguides for dense integration of passive guided wave devices at the 1.55 μm optical communications wavelength where Si is transparent.[7,11] The use of an oxidation process to smooth Si sidewall roughness has been shown highly effective for HIC Si-on-insulator waveguides.[8,9] This oxidation smoothing process can be interpreted by the Gibbs-Thompson relation, indicating a different chemical potential at the peaks and troughs of a rough surface which consequently leads to a differential oxidation rate which eventually smoothes out the roughness.[12] It is significantly more challenging in III-V compound semiconductors to achieve HIC waveguiding structures with both optically smooth and electrically passivated surfaces as required for optoelectronically active devices (e.g., lasers, modulators, and photodetectors). In this letter, we demonstrate unambiguously that the use of oxygen-enhanced wet thermal oxidation results in a substantial (up to 100 times) reduction of etched sidewall roughness in low Al content AlGaAs as is typically employed in the core of AlGaAs/GaAs heterostructure waveguides, whereas a conventional wet thermal oxidation process does not. This III-V compound semiconductor oxidation smoothing mechanism is a key feature enabling the high efficiency of our recently reported HIC ($\Delta n_{\mathrm{eff}} \sim 1.7$) ridge waveguide injection laser devices[13] and their ability to support resonators with compact waveguide bends as small as $r = 10$ μm radius of curvature.[14]

The heteroepitaxial structure used here for etching and oxidation smoothing studies has a 1.5 μm thick unintentionally doped $Al_{0.3}Ga_{0.7}As$ layer grown on a GaAs buffer and substrate by metal-organic chemical-vapor deposition. Ridge structure fabrication begins with contact lithography to pattern 5 μm wide stripes in photoresist, followed by reactive ion etching (RIE) in a $BCl_3/Cl_2/Ar$ gas mixture. After removing the photoresist and freshening the etched surface in 1:4 $HCl:H_2O$, samples undergo wet thermal oxidation in a conventional wet thermal oxidation gas ambient (obtained by bubbling 0.67 l/min ultrahigh purity N_2 carrier gas through a 95 °C H_2O bubbler) or an oxygen-enhanced water vapor obtained by mixing in 7000 ppm, i.e., 0.7%, O_2 relative to the N_2 carrier gas. Etch staining is conducted (1:1:10 $HCl:H_2O_2:H_2O$ for 5 s) to enhance the image contrast in subsequent scanning electron microscope (SEM) imaging. Figure 1 shows the cross-section view (x-y plane) of (a) conventional versus (b) O_2-enhanced wet thermal oxidation of an etched $Al_{0.3}Ga_{0.7}As$ ridge structure (both for 20 min at 450 °C). Approximately 60 and 200 nm of native oxide is grown in the conventional and O_2-enhanced processes, respectively. The thicker oxide in Fig. 1(b) results from the oxidation rate enhancement due to O_2 participation.[15] The SEM images clearly show that for O_2-enhanced wet oxidation, the initial rough sidewall features of $\sigma \sim 100$ nm dimension are smoothed away at the inward progressing oxidation front, resulting in an apparent final sidewall roughness (x-y plane) as low as $\sigma \sim 1-2$ nm [see high magnification inset to Fig. 1(b)]. In contrast, with no added O_2 (i.e., conventional wet oxidation) [Fig. 1(a)], the rough sidewall features do not disappear for the same oxidation time and an even rougher interface results. Similar results have been obtained on $Al_{0.5}Ga_{0.5}As$ samples, and we have shown that even for comparably thick oxides no smoothing occurs under

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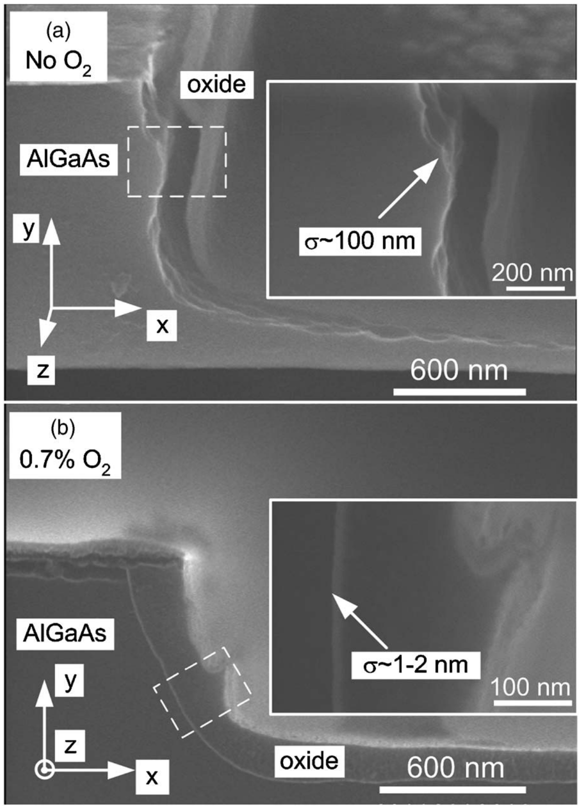

FIG. 1. SEM cross-sectional images of dry-etched $Al_{0.3}Ga_{0.7}As$ ridges after wet thermal oxidation for 20 min at 450 °C in (a) conventional N_2+H_2O and (b) O_2-enhanced (7000 ppm O_2/N_2 ratio) gas ambient. Insets: high-magnification SEM images showing the semiconductor/oxide interface roughness after oxidation. Sidewall roughness is reduced up to 100 fold in (b) relative to (a).

conventional wet oxidation.[16] In general, conventional wet oxides of low Al-content AlGaAs are of inferior quality to those grown via O_2-enhanced wet oxidation, as indicated by greater dissolution in the stain etch seen in Fig. 1(a), much lower refractive index (1.45–1.5 vs 1.65–1.7),[15,17] and poorer adhesion.[16]

While the images in Fig. 1 demonstrate smoothing in a cross sectional view (x-y plane), for ridge waveguide structures the smoothness of the interface along the propagation direction [z axis (into the page on Fig. 1)] determines the scattering loss. Figure 2 shows top view (x-z plane) images of the oxide/semiconductor ($Al_{0.3}Ga_{0.7}As$) interfaces formed (a) without and (b) with [Fig. 2(b)] added O_2. The specimens were prepared by encapsulating the etched and oxidized ridge with 1 μm of plasma-enhanced chemical-vapor deposition SiO_2 to protect the rough outer interface, followed by careful lapping and polishing and subsequent light etch staining as above. As in Fig. 1, Fig. 2(a) shows no smoothing occurring for conventional wet oxidation, while Fig. 2(b) shows that a significant reduction in the sidewall roughness (from $\sigma \sim 100$ nm down to a relatively long period, $\sigma \sim 5-10$ nm modulation) is achieved for O_2-enhanced wet oxidation. The nonuniform AlGaAs surface and particles seen on the oxide in Fig. 2(b) are remnants of the polishing process. The use of a hard dielectric mask (e.g., silicon dioxide or silicon nitride), which generally leads to improved sidewalls over photoresist masking, may make a further reduction in the sidewall roughness possible.

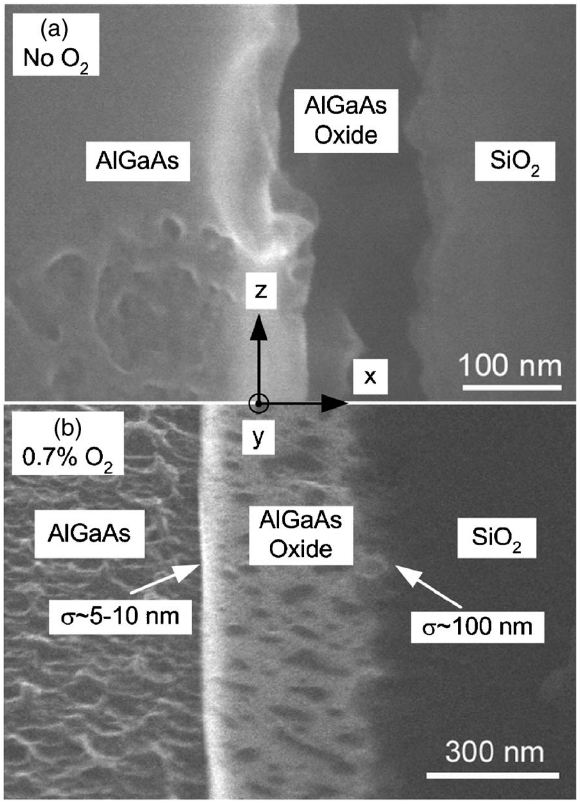

FIG. 2. Top-view (x-z plane) SEM images of oxide/semiconductor interface resulting after wet oxidation (a) without and (b) with 7000 ppm added O_2, showing 10–20 times roughness reduction along an etched $Al_{0.3}Ga_{0.7}As$ ridge. The SiO_2 encapsulant layer serves to preserve the original outer oxide/air interface during polishing.

The native oxides of $Al_{0.3}Ga_{0.7}As$ studied here have been found to be soluble in a dilute buffered HF (BHF) solution with good selectivity against unoxidized $Al_{0.3}Ga_{0.7}As$.[18,19] SEM images in Fig. 3 show (a) an unoxidized dry etched $Al_{0.3}Ga_{0.7}As$ ridge, (b) after 20 min O_2-enhanced wet oxidation at 450 °C with 7000 ppm O_2, and (c) after etching off the oxide in 1:10 BHF:H_2O for 15 min. The rough outer $Al_{0.3}Ga_{0.7}As$/air interface shown before oxidation in Fig. 3(a) remains rough in appearance as the outer oxide/air interface shown in Fig. 3(b) following oxidation. However, the inner $Al_{0.3}Ga_{0.7}As$/air interface observed in Fig. 3(c) after the oxide is selectively removed shows that the O_2-enhanced oxide growth has led to substantial sidewall roughness reduction along the entire ridge. The spongelike features on the $Al_{0.3}Ga_{0.7}As$ surface in Fig. 3(c) are believed to be remnants of unremoved oxide.

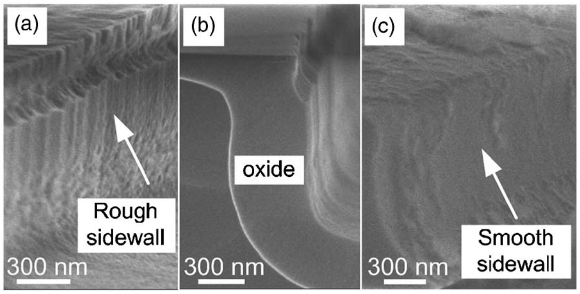

FIG. 3. SEM images of dry-etched $Al_{0.3}Ga_{0.7}As$ ridge structure (a) before oxidation, (b) after 20 min, 450 °C O_2-enhanced wet oxidation (7000 ppm O_2), and (c) after selective oxide removal in 1:10 BHF:H_2O solution for 15 min to reveal oxidation-smoothed interface.

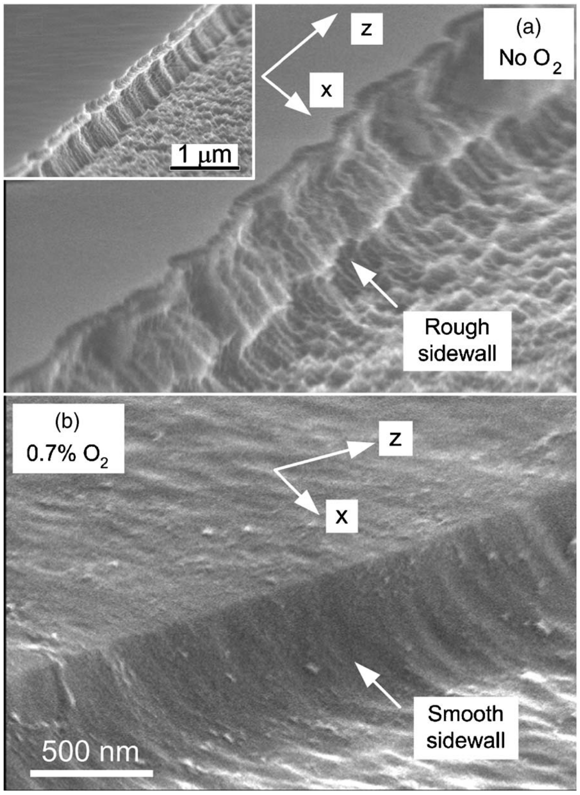

FIG. 4. SEM images of shallow dry-etched $Al_{0.3}Ga_{0.7}As$ ridge structures wet oxidized at 450 °C for 45 min (a) without and (b) with added O_2 (7000 ppm), shown after selective oxide removal in 1:10 BHF:H_2O for 75 min. The inset shows 500 nm tall ridge structure formed via RIE before oxidation. Image (a) demonstrates the absence of smoothing in the BHF solution.

In order to verify that the observed smoothing is solely due to the O_2-enhanced wet oxidation process and not isotropic smoothing of the AlGaAs by the dilute BHF solution, four $Al_{0.3}Ga_{0.7}As$ samples with surfaces intentionally roughened via RIE are left in the dilute BHF for 5, 30, 45, and 75 min, respectively. No surface improvement (i.e., smoothing) or degradation (i.e., roughening) is observed in SEM images (not shown), eliminating the possibility that the BHF might play a role in the observed smoothing shown in Fig. 3(c). This is also confirmed by the SEM images in Fig. 4 which show shallow-etched $Al_{0.3}Ga_{0.7}As$ (inset) oxidized at 450 °C for 45 min by (a) conventional oxidation and (b) O_2-enhanced wet oxidation, following immersion in the dilute BHF for 75 min to completely etch off the oxide. Because the sidewall roughness cannot be reduced except through the participation of O_2 during wet oxidation, the rough outer sidewall in Fig. 4(a) remains unchanged from that of the original outer surface roughness (inset), confirming the absence of smoothing by the BHF. A dramatically different result is observed in Fig. 4(b), where substantial sidewall oxidation smoothing has occurred during the O_2-enhanced growth of ~520 nm of oxide. The flat and smooth GaAs cap layer in Fig. 4(a) also shows the lack of any reaction of the GaAs cap layer to the conventional oxidation ambient and subsequent dilute BHF wet etch. In contrast, the similar top and sidewall surfaces in Fig. 4(b) show that the relatively nonselective O_2-enhanced wet thermal oxidation process[15] also converts the GaAs cap layer into an oxide which is then removed by the BHF solution.

In conclusion, we have demonstrated that substantial isotropic smoothing of sidewall roughness in etched AlGaAs ridge structures is achievable via an O_2-enhanced wet thermal oxidation process. Further analysis and modeling are required in order to understand why O_2 participation during wet oxidation enables the interface smoothing to occur. The process has significant potential for advancing the integration of GaAs-based photonic circuits by enabling the fabrication of high-index-contrast waveguides simultaneously possessing both low bending and low scattering losses.

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