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Defect profiling of Al₂O₃-passivated InGaP layers via planar test structures

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Abstract— The electrically active defects in Al₂O₃-passivated InGaP layers, representative of an AlInGaP red micro-light-emitting diodes (μ-LED) sidewall, are characterized by using planar metal/insulator/semiconductor (MIS) capacitors test structures. The use of a planar test structure avoids the complexity of the full μ-LED device analysis and enables the selective InGaP/Al₂O₃ system characterization. The electrically active defects of InGaP/Al₂O₃/Ni MIS test structures, subject to different surface treatments, are investigated by multifrequency capacitance-voltage (CV) and conductance-voltage (GV) curves analysis. Trap levels located in the InGaP and in the Al₂O₃ are detected and profiled. The effect of the surface treatment (either no treatment, wet etching, or dry etching) on the detected traps is reported.

Keywords— Al₂O₃, defect, InGaP, sidewall, trap, μ-LED

I. INTRODUCTION

The μ-LEDs are critical components for the development of next-generation displays, including virtual and augmented reality applications. However, the fabrication of efficient red μ-LEDs remains an elusive technological challenge. Conventional AlGaInP red LEDs, providing internal quantum efficiencies (IQE) exceeding 90% in large-area devices [1], exhibit a dramatic efficiency reduction when scaled to μ-LEDs size (i.e., <100 μm side length) [2-4]. This phenomenon, referred to as “size effect”, is ascribed to non-radiative recombination centers (NRRCs) associated to damage in the vicinity of the mesa sidewalls induced during the μ-LED structure fabrication and passivation [2-4], as depicted in Fig. 1a. The atomic origin of the NRRCs in AlGaInP red LEDs is currently unknown. Profiling the NRRCs involved in the IQE degradation is of paramount importance for the further development of the red μ-LED technology. Previous studies relied on measuring changes electrical or optical characteristics in full μ-LED structures, resulting in non-quantitative and non-material-selective insights on the NRCC [3-6].

In this work, we characterize the electrically active defects in Al₂O₃-passivated InGaP layers, a material system widely considered for μ-LEDs [3,7,8]. Planar MIS capacitors test structures are used to replicate Al₂O₃-passivated InGaP μ-LED sidewalls (Fig. 1b) subject to different surface treatments, possibly including either a wet etching or a dry etching. This approach allows overcoming the complexity of a full μ-LED analysis and enables a material-selective characterization. The electrically active defects are investigated by multifrequency CV and GV curves analysis of the MIS test structures, supported by multiphysics TCAD simulations. This results in an extensive mapping and profiling of trap levels, detected both in the InGaP (close to

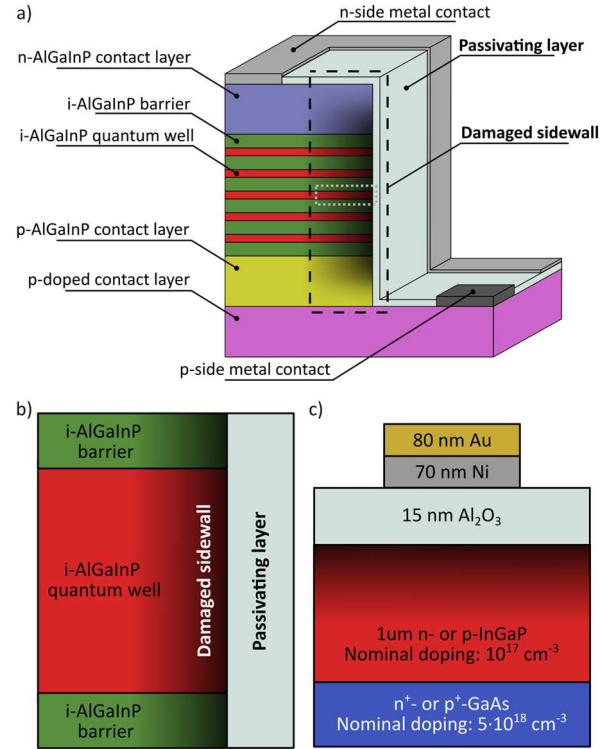


Fig. 1. a) Schematic of a multi-quantum well AlInGaP red μ-LED, showing the damaged mesa sidewall region (shaded) under the passivation layer; b) a magnification of the damaged region highlighted by the grey dotted area in Fig. 1a; c) the planar MIS test structure used to investigate the defects in Al₂O₃-passivated InGaP layer, representative of an AlInGaP red μ-LED quantum well.

the InGaP/Al₂O₃ interface) and in the Al₂O₃ border region, energetically located near the InGaP band edges.

II. DEVICES AND SIMULATIONS

The planar InGaP/Al₂O₃/Ni MIS test structures considered in this study, depicted in Fig. 1c, allow reproducing the cross-section of a μ-LED layer (Fig. 1b), composed of an InGaP layer and an insulating passivation layer. The additional metal electrode, not present in the μ-LEDs, provides an electric contact for the measurements. The InGaP layer is intentionally doped to tune its electrical behavior and to ensure the test structures' reproducibility. By applying the same surface preparation used in the full μ-LED fabrication, the resulting MIS test structures reproduce the atomic defects distributions of the replicated μ-LED region.

The InGaP/Al₂O₃/Ni MIS test structures were fabricated on heavily doped (5·10¹⁸ cm⁻³) GaAs substrates with both n-

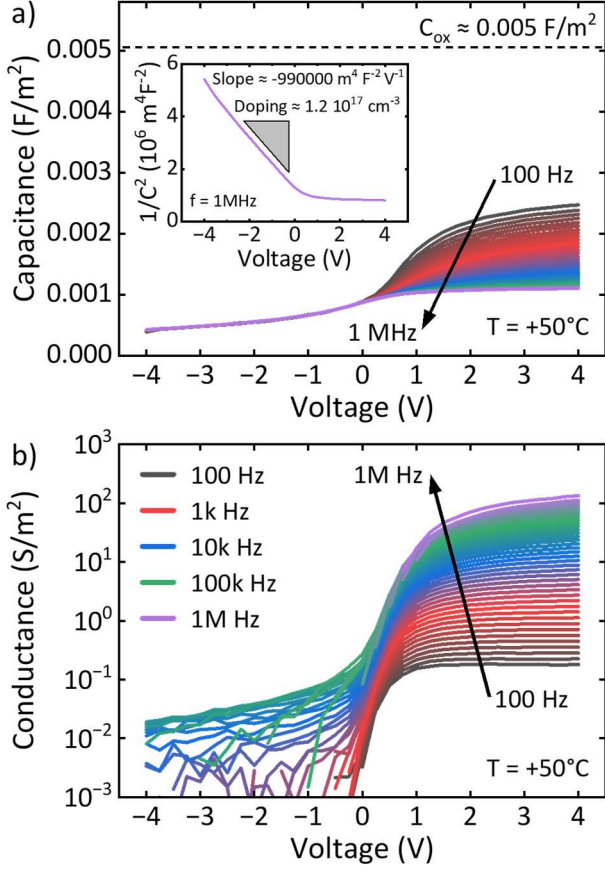


Fig. 2. Experimental CV (a) and GV (b) of a n-InGaP/Al₂O₃/Ni MIS test structure with no surface treatment at +50°C. The inset in (a) shows the $1/C^2$ plot used to extract the n-InGaP doping according to Eq. 1.

type and p-type polarity. A 1 μm lattice-matched In_{0.49}Ga_{0.51}P epilayer, Si-doped (n-type) or Mg-doped (p-type), was deposited by metal-organic vapor-phase epitaxy on a GaAs substrate of the corresponding polarity. The target nominal doping for the epilayers was 10^{17} cm^{-3} . Following growth, an optional surface treatment, either a wet etching or a dry etching, was applied to the InGaP surface to simulate the μ -LED sidewall processing. A 15 nm Al₂O₃ insulating layer, replicating the μ -LED sidewall passivation layer, was deposited by thermal atomic-layer deposition at 250 °C using trimethylaluminum and deionized water. A 70/80 nm Ni/Au electrode was deposited by e-beam evaporation, patterned by a photolithography and lift-off process.

The test structures' multifrequency CV and GV characteristics (100 Hz–1 MHz) were measured on a Cascade probe station with a Keysight E4980 LCR meter. The measurements were performed by using a single voltage sweep, sweeping the full frequency range at each analyzed bias point. This avoids artifacts due to trap charging for subsequent voltage sweeps (e.g., horizontal voltage shifts).

Device simulations used the Ginestra™ TCAD software [9]. Ginestra™ self-consistently models the stack electrostatics and all the main carrier transport mechanisms, including the charge capture/emission processes related to atomic defects. The capture and emission rates are computed according to the multi-phonon trapping theory [10–12] providing an accurate and physically grounded description of the two processes. This allows accounting for the carrier-phonon coupling, the lattice relaxation during the carriers' capture and emission, and the possible tunneling into traps.

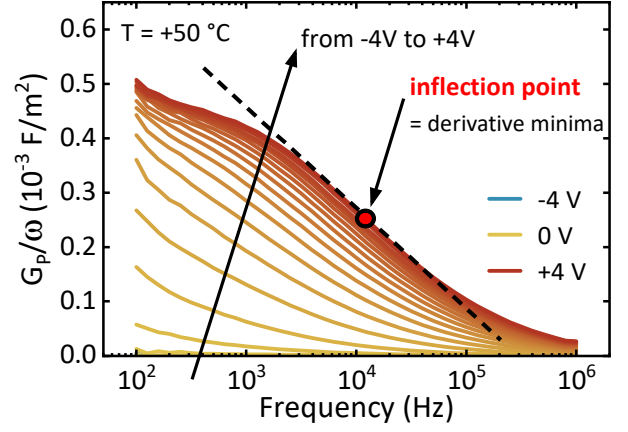


Fig. 3. The G_p/ω plot associated to the n-InGaP/Al₂O₃/Ni MIS GV curves of Fig.2b (no surface treatment), showing the emergence of a peak at positive bias. Even if the peak is not fully resolved, its temperature dependence can be tracked by its inflection point (identified by the derivative minima).

III. RESULTS AND DISCUSSION

The n-InGaP/Al₂O₃/Ni MIS test structures with no surface treatment are first considered to present the used methodology. The same methodology is then applied to p-InGaP/Al₂O₃/Ni MIS test structures with no surface treatment to prove its generality. Finally, surface-treated n-InGaP/Al₂O₃/Ni MIS test structures were analyzed to investigate the impact of surface treatments (i.e., a wet etching and a dry etching) on the traps' distributions.

A. n-InGaP/Al₂O₃/Ni MIS – no surface treatment

At negative bias, the pristine n-InGaP MIS test structures exhibit a deep-depletion trend associated to a negligible minority carriers' generation, thus to a low mid-gap traps' density. An active doping concentration $N_D \approx 1.2 \cdot 10^{17} \text{ cm}^{-3}$ is calculated according to [13]:

$$N_D = 2 \left(q \varepsilon_0 k_{\text{InGaP}} \frac{dC_m^{-2}}{dV} \right)^{-1} \quad (1)$$

where q is the elementary charge, ε_0 is the vacuum permittivity, k_{InGaP} is the InGaP relative permittivity, and C_m is the measured capacitance per unit area.

At positive bias, CV (Fig. 2a) and GV (Fig. 2b) curves of the pristine MIS clearly exhibit non-ideal features. The CV remains significantly lower than the nominal oxide capacitance $C_{\text{ox}} \approx 0.005 \text{ F/m}^2$, indicating that true majority carriers' accumulation is not reached. This behavior is consistent with interface and near-interface traps pinning the surface potential and enforcing a persistent depletion region in the InGaP [14,15]. The CV and GV frequency dispersion trend at positive bias is consistent with Al₂O₃ border traps [16,17]. The pinning traps' AC response is not clearly detected from the experimental data of Fig. 2, suggesting their extremely slow dynamics in the probed frequency and temperature ranges. The high-frequency measured capacitance (per unit area) C_{HF} , exhibited in the limit of no frequency dispersion (e.g., at 1 MHz), provides information on the MIS stack electrostatics. Since the MIS stack operates either in deep-depletion or in depletion over the considered voltage range, C_{HF} follows:

$$C_{\text{HF}}^{-1}(V) = C_{\text{ox}}^{-1} + C_D^{-1}(V) = \frac{t_{\text{ox}}}{\varepsilon_0 k_{\text{ox}}} + \frac{W_D(V)}{\varepsilon_0 k_{\text{InGaP}}} \quad (2)$$

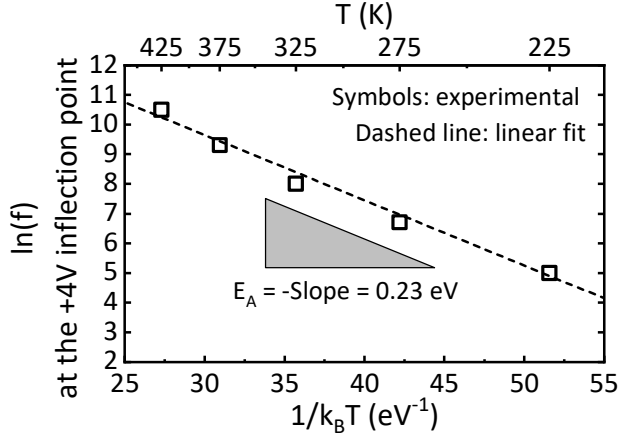


Fig. 4. Arrhenius plot tracking the position of the G_p/ω peaks of the n-InGaP/Al₂O₃/Ni MIS of Fig.2b (no surface treatment) at different temperatures. Tracking the position of the G_p/ω peaks through the G_p/ω inflection point an activation energy $E_A \approx 0.23$ eV is extracted.

where V is the bias voltage, C_{OX} is the oxide capacitance per unit area, C_D is the depletion layer capacitance per unit area, t_{OX} is the oxide thickness, k_{OX} is the oxide relative permittivity, and W_D is the depletion width.

Noticeably, the high-frequency capacitance remains constant at approximately 0.001 F/m² over the full positive bias range, confirming the surface potential pinning. This capacitance value is explained, using Eq. 2, by a persistent depletion width $W_D \approx 70$ nm. The frequency dispersion, providing information on the oxide traps response, is analysed using the conductance method [13] by using the quantity G_p/ω :

$$\frac{G_p}{\omega} = \frac{\omega G_m C_{OX}^2}{G_m^2 + \omega^2 (C_{OX} - C_m)^2} \quad (3)$$

where $\omega = 2\pi f$ is the AC signal angular frequency with f being its frequency, G_p is the parallel conductance per unit area

associated to the InGaP substrate, and G_m is the measured conductance per unit area.

The G_p/ω plot associated to the GVs of Fig.2b, calculated as in Eq. (3) and shown in Fig. 3, reveals a peak emerging at positive bias, even if not fully resolved. The saturation of the G_p/ω peak at high bias also confirms the surface potential pinning. The central frequency of the G_p/ω peak f_{PK} is related to the interacting traps' (i.e., the oxide traps) time constant τ_T as [11,18]:

$$\tau_T^{-1}(T) = 2c_n(T)n_s(T) = 2\pi f_{PK}(T) \quad (4)$$

where c_n is the electron capture coefficient, n_s is the surface electron density, and T is the temperature.

The rate τ_T^{-1} , being proportional to the surface carrier density n_s , is in turn affected by the surface barrier ϕ_S [18]:

$$\tau_T^{-1}(T) = 2c_n(T)N_D \exp\left(-\frac{\phi_S}{k_B T}\right) \quad (5)$$

Under pinned conditions, the surface barrier is enforced by the pinning traps' thermal ionization energy $E_{TH}^{pinning}$ (i.e. $\phi_S = E_{TH}^{pinning}$):

$$\tau_T^{-1}(T) = 2c_n(T)N_D \exp\left(-\frac{E_{TH}^{pinning}}{k_B T}\right) \quad (6)$$

This shows that, although the detected G_p/ω peak is associated with the oxide traps, its temperature dependent frequency shift provides information on the pinning traps' depth. It's worth noting that, even if Eq. (5) and (6) are exact only for interface traps, they also hold for traps close to the interface (verified by simulations).

Fig. 4 shows the Arrhenius plot resulting from the G_p/ω peaks tracking in the -50°C to +150°C temperature range. Since the G_p/ω peaks were not fully resolved over the considered frequency/voltage/temperature ranges, the peaks'

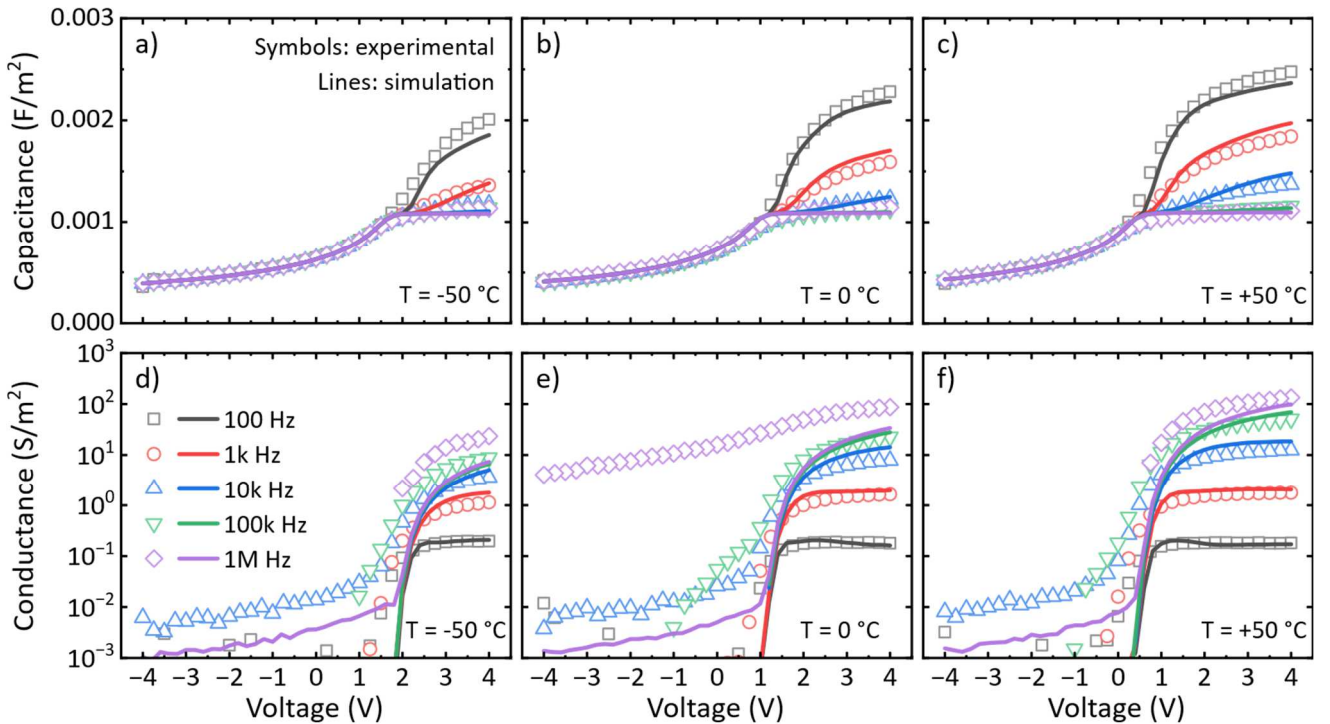


Fig. 5: Experimental and simulated CVs (a-c) and GVs (d-f) of a n-InGaP/Al₂O₃/Ni MIS test structure with no surface treatment in the -50°C to +50°C range. The simulations accurately reproduce both the frequency and the temperature dependences.

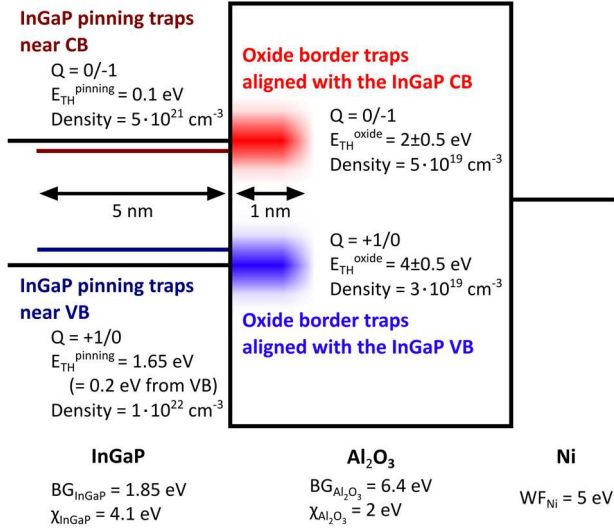


Fig. 6. Trap profiles extracted from the InGaP/Al₂O₃/Ni MIS test structures with no surface treatment. The n-InGaP/Al₂O₃/Ni MIS allowed detecting a shallow InGaP trap level close the conduction band ($E_C - E_T = E_{TH}^{pinning} = 0.1$ eV), and oxide border traps aligned with the InGaP conduction band (shown in red). The p-InGaP/Al₂O₃/Ni MIS allowed detecting a shallow InGaP trap level close the valence band ($E_T - E_V = BG_{InGaP} - E_{TH}^{pinning} = 0.2$ eV), and oxide border traps aligned with the InGaP valence band (shown in blue). Note: flat-band condition is shown for convenience. In practice, flat-band is not achieved over the examined bias range due to the strong surface potential pinning enforced by the InGaP pinning traps.

positions were tracked using their upper inflection points (identified by the G_p/ω derivative minima, see Fig. 3). The Arrhenius plot clearly shows a temperature dependent shift of the peaks, revealing an activation energy $E_A \approx 0.23$ eV. The extracted activation energy accounts for both the temperature dependence associated with the pinning level depth (i.e., the exponential part of Eq. 6) and the possible temperature dependence of the capture coefficient $c_n(T)$, including both effects in a single exponential as:

$$\tau_T^{-1}(T) \approx A \exp\left(-\frac{E_A}{k_B T}\right) \quad (7)$$

where A is a temperature independent prefactor.

Comparing Eq. 6 and Eq. 7, it's clear that the extracted E_A provides an upper limit for $E_{TH}^{pinning}$ (i.e., $E_{TH}^{pinning} = E_A$ for a temperature independent capture coefficient c_n , $E_{TH}^{pinning} < E_A$ otherwise). The information provided by the CV and GV curves at different temperatures, and the Arrhenius plot of

Fig. 4 were used to reproduce the experimental results. A robust fit reproducing both the frequency and temperature dependences is obtained, resulting in the simulations shown in Fig. 5. The considered traps' distributions are depicted in Fig. 6 (in red), including a shallow pinning level ($E_{TH}^{pinning} = 0.1$ eV, with density = $5 \cdot 10^{21} \text{ cm}^{-3}$) extending for 5 nm in the InGaP bulk and a 1 nm border trap distribution in the Al₂O₃, aligned with the InGaP conduction band ($E_{TH}^{oxide} = 2 \pm 0.5$ eV, with density = $5 \cdot 10^{19} \text{ cm}^{-3}$). Noticeably, an $E_{TH}^{pinning}$ lower than the extracted E_A is used to account for the temperature dependence of the capture coefficient c_n introduced by the lattice relaxation of the Al₂O₃ border traps [16,17,19,20].

B. p-InGaP/Al₂O₃/Ni MIS – no surface treatment

The same methodology presented in Section IIIA was applied to p-InGaP MIS test structures with no surface treatment (Fig. 7a) to investigate the traps' distributions near the InGaP valence band. Interestingly, a defect landscape similar to the n-InGaP described in Section IIIA is revealed (Fig. 6, in blue): a shallow pinning level ($E_{TH}^{pinning} = 1.65$ eV, with density = $1 \cdot 10^{22} \text{ cm}^{-3}$) extending in the InGaP bulk and a 1 nm border trap distribution in the Al₂O₃ aligned with the InGaP valence band ($E_{TH}^{oxide} = 4 \pm 0.5$ eV, with density = $3 \cdot 10^{19} \text{ cm}^{-3}$).

C. n-InGaP/Al₂O₃/Ni MIS – wet etching

In n-InGaP MIS test structures subject to the wet etching step before the Al₂O₃ deposition (Fig. 7b), the extracted trap profile is largely unchanged relative to the untreated case. The high-frequency capacitance in the pinned range remains approximately 0.001 F/m², indicating that the InGaP pinning traps' density is essentially unaffected. However, fitting the wet-etched sample requires a thinner border trap layer (~ 0.5 nm). This suggests the wet etching is only affecting the InGaP surface with partial removal of the native oxide.

D. n-InGaP/Al₂O₃/Ni MIS – dry etching

In n-InGaP MIS test structures subject to the dry etching step before the Al₂O₃ deposition (Fig. 7c), the InGaP trap profile is significantly different from the untreated case. The reduced high-frequency capacitance over the pinned region indicates a stronger surface potential pinning, thus a higher pinning traps' density. Moreover, inversion is observed instead of the deep-depletion trend, indicating an increased mid-gap traps' density. Fitting the wet-etched sample requires an even thinner border trap layer (< 0.2 nm), suggesting the complete removal of the native oxide.

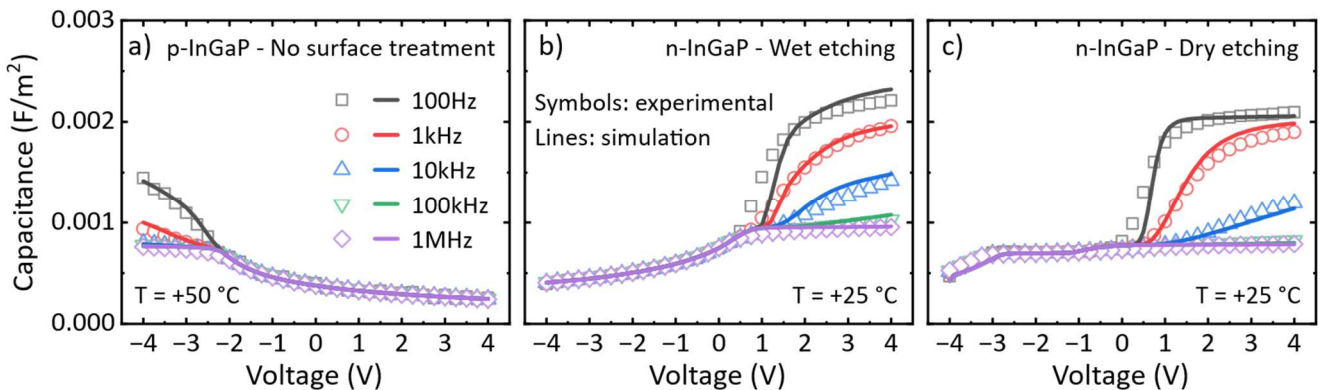


Fig. 7. Experimental and simulated CVs for different InGaP/Al₂O₃/Ni MIS test structures: a) p-InGaP/Al₂O₃/Ni MIS with no surface treatment (1 nm oxide border traps); b) n-InGaP/Al₂O₃/Ni MIS with wet etching surface treatment (0.5 nm oxide border traps); c) n-InGaP/Al₂O₃/Ni MIS with dry etching surface treatment (0.1 nm oxide border traps).

IV. CONCLUSIONS

InGaP/Al₂O₃/Ni MIS test structures were used for investigating sidewall-related defects in an Al₂O₃-passivated InGaP layers, representative of AlInGaP red LED. The presented analysis revealed a high-density of trap states in InGaP near both the band edges and tracked the etching-induced changes in the trap profiles in both InGaP and Al₂O₃. Neither of the etching treatments remove the shallow InGaP trap level at $E_{\text{TH}}^{\text{pinning}} = 0.1$ eV. Wet etching only partially modifies the InGaP surface, whereas dry etching affects both the surface and the near-surface bulk, substantially increasing the InGaP-related traps' densities (both shallow and mid-gap states). The presented methodology enables the profiling of shallow states near both band edges and the detection of mid-gap states, thus providing a valuable tool for investigating near-interface trap distributions in passivated semiconductor layers. Its application is readily extendable to the full AlInGaP alloy composition range and to other material systems currently studied for the fabrication of high-efficiency μ -LEDs, including AlInGaP alloys. It is also suited for profiling near-interface traps in MIS stacks, particularly in the presence of a strong surface potential pinning.

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