

Real-time monitoring of low-temperature hydrogen plasma passivation of GaAs

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By monitoring photoluminescence (PL) in real time and *in situ*, hydrogen plasma operating conditions have been optimized for surface passivation of native-oxide-contaminated GaAs. PL enhancement is critically dependent on exposure time and pressure because of competition between plasma passivation and damage. Optimal exposure time and pressure are inversely related; thus, previous reports of ineffective passivation at room temperature result from overexposure at low pressure. Plasma treatment is effective in removing As to leave a Ga-rich oxide; removal of excess As increases the photoluminescence yield as the corresponding near-midgap-state density is reduced. Passivation is stable for more than a month. These results demonstrate the power of real time monitoring for optimizing plasma processing of optoelectronic materials.

I. INTRODUCTION

The well-known poor electronic properties of oxidized GaAs have stimulated many attempts to passivate the surface, reduce the surface state density, and unpin the surface Fermi level.¹⁻³¹ There are many reasons why it is desirable to reduce the surface state density of III-V compound semiconductors. In particular, for GaAs-based electronic devices, a reduction in surface state density would lead to improved field-effect transistors (FETs) by lowering surface-related leakage currents and by increasing the Schottky barrier height. This would allow the fabrication of digital logic circuits with increased noise margins and relaxed requirements on threshold voltage uniformity of the component FETs. In addition, the unpinning of the surface Fermi level would allow advances in metal-insulator-semiconductor (MIS) capacitor and MISFET technology. For photonic devices, it is important to reduce the surface recombination velocity and increase carrier lifetime for enhanced power output. Passivation may be carried out prior to processing as a cleaning step or after processing to remove process-induced damage.

A variety of wet chemical^{1,2,5,6,8-10,15,17} and photochemical^{12,18-20} treatments have been used to passivate GaAs but controversy remains as to their effectiveness in unpinning the Fermi level.^{5,6,13,14,16,24,27,32} Nonetheless, it is clear that photoluminescence (PL) yield is enhanced by using these surface treatments. In fact, passivation is operationally defined in terms of this enhancement in quantum yield. Although large increases in PL yield have been observed, the enhancements tend to be short lived. We are aware of only one case where the enhancement persisted for more than a few hours.¹⁰ Occasionally, real-time PL monitoring has been used to monitor the effectiveness and kinetics of these wet chemical treatments.^{12,18}

To improve process control and management of environmental hazards, it is desirable to replace wet chemical

processing with dry processing. In particular, plasma methods are effective for not only etching but also deposition, cleaning, and passivation. Recently, H₂ plasmas operated at low pressure have been used to clean GaAs of residual As and As₂O₃.^{21-23,25-27,29,30} Similar plasma treatments have also been reported for InP surface cleaning.³³⁻³⁵ A variety of reactor configurations have been employed: electron cyclotron resonance (ecr) microwave, multipolar, and rf diode. However, in these reports choice of optimal exposure times, which have ranged from 5 s to 30 min, and operating pressures, which have ranged from 10⁻⁴ to 10⁻² Torr are not discussed. Similarly, the consequences of reactor geometry or excitation mode choice have not been addressed. Paccagnella *et al.* studied the properties of Schottky diodes made on hydrogen-plasma-treated *n*-GaAs surfaces as a function of the treatment temperature and concluded that plasma passivation is ineffective for temperatures below 200° C.²⁵ A similar conclusion has been made for hydrogen plasma treatment of InP surfaces.³³ However, as we show here, such conclusions depend on other plasma parameters such as geometry, power, pressure, and exposure time.

Real-time monitoring of PL from GaAs during hydrogen plasma passivation clearly provides the means to investigate the effects of processing conditions such as exposure time, pressure, and temperature. In addition, the kinetics of plasma-surface reactions can be monitored by measuring real-time changes in PL yield.³⁶ In general, PL yield is sensitive to changes in surface recombination velocity (*S*) and band bending.³⁷⁻⁴⁰ Clearly, PL yield increases as *S* decreases. In addition, if the bands are bent and a space-charge field exists near the surface, separation of electrons and holes can lead to a reduction in radiative recombination efficiency. Thus, a reduction in band bending can increase PL yield. However, if *S* is reduced substantially, PL yield can increase despite an increase in band bending.^{5,6,13,14,16,21,27,32} In *n*-GaAs, for example, passivation treatments tend to remove excess As from the surface and thereby reduce the As antisite defect density that pins the Fermi level near midgap. The

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Fermi level shifts to lower energy because the density of Ga antisite defects remains constant or even increases. Thus, the band bending increases. Ordinarily this would cause a decrease in PL yield as carriers are separated by the space-charge field. However, reduction of the near-midgap As antisite defect density decreases the nonradiative recombination rate causing an increase in PL yield.

In this work, we use PL to monitor the effectiveness of and optimize processing conditions for hydrogen plasma passivation of native-oxide-contaminated GaAs. A preliminary report of this work has recently been published.⁴¹ We find that previously reported pressures and exposure times for rf diode reactors are far from optimal^{25,33} and that low-temperature passivation is feasible. The effects of varying pressure, exposure time, flow rate, rf power, and surface temperature are examined, but only pressure and exposure time are found to be critical. In addition to monitoring PL intensity, we have fabricated Schottky diodes from passivated *n*-type material and measured *I-V* characteristics to deduce the surface barrier height. Treated surfaces are also examined using x-ray photoelectron spectroscopy (XPS) to determine the chemical consequences of the passivation treatment. Finally, the long-term stability of the treatment is evaluated.

II. EXPERIMENT

Samples are either semi-insulating or *n*-type (10^{17} cm^{-3}) GaAs (100) substrates. Initially, these samples were inserted into the plasma reactor without any preprocess treatment. However, we soon discovered that the magnitude of the PL enhancement and its long-term stability are widely variable from wafer to wafer, although for a given wafer the reproducibility is excellent; within a wafer lot, reproducibility is better than between lots. These variations are attributed to differences in native oxide thickness and composition from wafer to wafer and lot to lot. Thus, when wafers are first dipped in concentrated (30%) HF or NH_4OH for 30 s, excellent PL yield reproducibility and long-term stability are observed. All results presented here correspond to samples first treated by dipping for 30 s in HF.

The diagnostic arrangement is shown in Fig. 1. A pulsed N_2 pumped dye laser (Moletron) beam is injected into a fiber-optic cable so that the beam can be transported from one optical table to another. After passing through a polarizer to select *p*-polarized light, the output from the fiber is focused onto the sample to a spot size of about 0.15 cm^2 (1 cm long \times 0.15 cm wide). The angle of incidence is $\sim 83^\circ$ from normal which is close to Brewster's angle so that most of the light is efficiently coupled into the substrate. The laser pulse energy is typically $100 \mu\text{J}$ of 500-nm light.

PL is collected, collimated, and then focused onto the entrance slit of a scanning monochromator (Spex 1269, 1.25 m) equipped with a photomultiplier tube (PMT) (RCA C31034A or Hamamatsu 7102). The output of the PMT is monitored using a gated integrator (SRS model 250) which in turn is sampled by a Digital Equipment Corporation 11/23 computer.

Pulsed laser excitation is preferable to continuous-wave excitation since the PL signal is superlinear with laser pulse

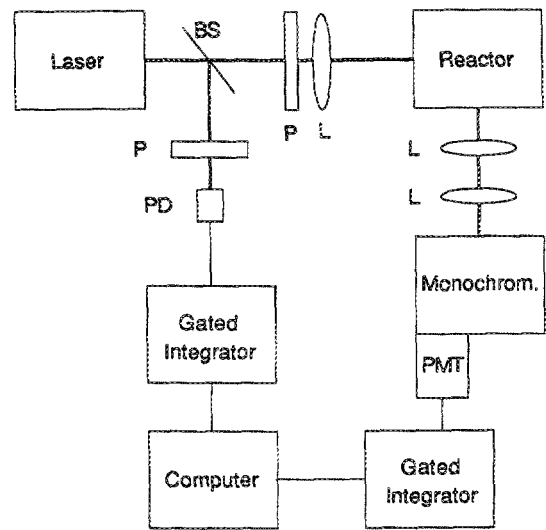


FIG. 1. Schematic of experimental arrangement. BS: beam splitter, P: polarizer, L: lens, PMT: photomultiplier tube, PD: photodiode.

energy (Fig. 2). The nonlinearity in Fig. 2 is believed to result from high-level injection and flattening of the bands. Thus, short-pulse, high-intensity excitation with gated electronic detection eliminates the plasma background glow. In addition, a low duty cycle, pulsed laser deposits less energy into the sample and thereby minimizes heating and photodegradation.⁴² Note that for the level of excitation employed here, the experiment is relatively insensitive to changes in band bending caused by the passivation process or doping level (10^{17} – 10^{19} cm^{-3}).

Samples are plasma passivated in a parallel-plate, capacitively coupled reactor described in detail previously.⁴³ Briefly, we use a six-way Pyrex cross with 7.6-cm-diam anodized Al, water-cooled electrodes, and a gap of 3 cm. The

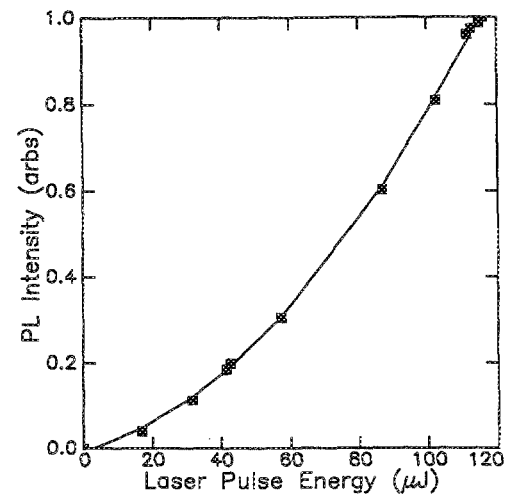


FIG. 2. Semi-insulating GaAs band-edge (868 nm) photoluminescence intensity as a function of 8-ns laser pulse energy. Approximate beam area is 0.15 cm^2 .

wafer is attached to the grounded, bottom electrode with heat-sink compound (Wakefield 120-2) and the top electrode is connected to a 13-MHz power supply (Tektronix FG504 oscillator, ENI 350L amplifier, and pi matching network). The plasma is gated on and off by using a control voltage generated by the computer (D/A converter) to 100% modulate the output from the oscillator. Power measurements are routinely made using a Bird 4410 meter connected between the amplifier output and the matching network input. Power is also estimated by digitizing instantaneous current (measured in the ground lead) and voltage waveforms and taking their averaged product over a single rf cycle. These two estimates agree to within a factor of 2, with the Bird meter always being larger. The matching network is pretuned by running H_2 plasmas under the conditions of interest for the subsequent passivation. H_2 (Spectra Gases 99.999%) flow rate and pressure are controlled using MKS 260 series mass flow controllers and an MKS 257 throttling valve, respectively.

III. REAL-TIME PHOTOLUMINESCENCE MONITORING

When GaAs is exposed to a H_2 plasma, H atoms created in the plasma react with native oxides and reduce the surface As concentration and thereby increase the PL yield. The plasma also produces energetic ion bombardment of the surface and can create damage that increases nonradiative recombination and decreases PL yield. Both of these effects are evident when GaAs PL is monitored as a function of time. In Fig. 3(a), a GaAs substrate is first dipped in HF to thin the native oxide and then exposed to a moderately low-pressure H_2 discharge. When the plasma is gated on, the PL yield first

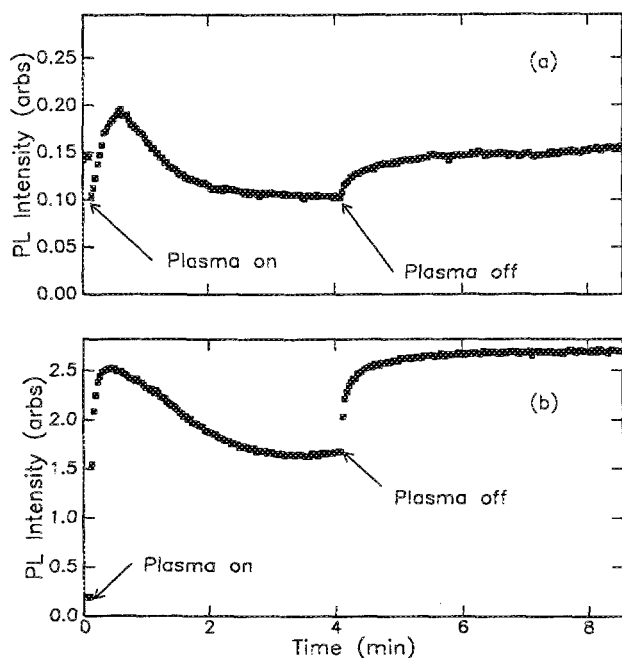


FIG. 3. GaAs PL intensity vs time for (a) 0.2-Torr and (b) 4-Torr plasma treatments. Other plasma parameters: 13 MHz, 20 W, 20 °C, 2 sccm H_2 . Note difference in scales between (a) and (b).

decreases as the surface charges negatively,⁴⁴ then increases rapidly as As is removed from the surface, (see next section) and finally plummets to a steady-state level below the initial value as the plasma irreversibly damages the surface. When the plasma is gated off, a slight increase in PL yield occurs and is attributed to discharging of the surface.⁴⁴

Similar results are obtained over a range of substrate temperatures (10–50 °C), flow rates (0.4–40 sccm), and rf powers (4–20 W). Only pressure has a substantial effect on the magnitude and time dependence of the PL enhancement [compare Figs. 3(a) and 3(b)]. At higher pressures, sheath voltages are smaller, sheaths are more collisional, ion energies are smaller, and damage does not readily occur. Changes in pressure could also change the atomic H concentration and thereby affect passivation kinetics. Although the atom production rate by electron impact should increase with pressure, recombination rates should also increase and the overall change in atom concentration with pressure is difficult to predict. In any case, PL enhancement is much larger at higher pressure and the decrease in PL that occurs on overexposure is attributed solely to reversible charging of the surface by the plasma. Exposure time at pressures ≥ 2 Torr is not critical as can be seen by comparing final PL levels in Figs. 3(b) and 4. Similar results are expected for processing downstream from the glow discharge or, in general, for reactor geometries and excitation modes that minimize energetic ion bombardment.

For plasma treatments under the conditions of Figs. 3(b) and 4—high pressure, short times, low temperature—only the surface should be passivated (i.e., $< 100 \text{ \AA}$). Bulk passivation is not likely to play an important role in enhancing the PL yield. For a peak voltage of 380 V, typical of our operating conditions, the time-averaged sheath voltage that ions can respond to in being accelerated to the surface is only $\sim 120 \text{ V}$.⁴⁵ At pressures of 4 Torr, ions experience a significant number of charge exchange collisions as they traverse

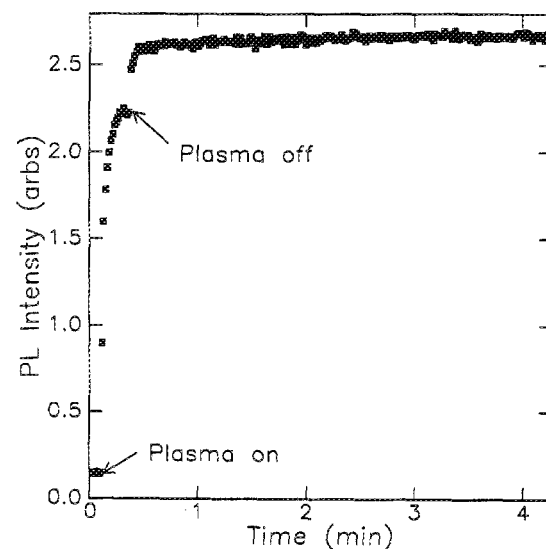


FIG. 4. GaAs PL intensity as a function of time [same conditions as in Fig. 3(b)]. Plasma turned off as PL intensity reaches a maximum. Despite the overexposure in Fig. 3(b), the final PL levels are comparable.

the ~ 0.5 -mm-thick sheath and their average energy is reduced further to ~ 50 V. With such low ion energies, the penetration of ionic hydrogen into the bulk beyond 100 \AA can be neglected.⁴⁶ Similarly, at the low temperatures employed here ($\leq 50^\circ\text{C}$), diffusion of atomic hydrogen beyond 100 \AA can be neglected.⁴⁷ These conditions are quite different from those typically employed for bulk dopant or defect passivation—long exposure, high temperature, energetic ion bombardment.⁴⁸ Examination of the low-temperature PL spectrum confirms that the passivation process described is surface ($< 1000 \text{ \AA}$) specific: no significant differences are observed between passivated and unpassivated samples.

IV. X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)

To more fully understand the consequences of hydrogen plasma passivation, we examined the surfaces using XPS. The results are shown in Figs. 5 and 6. Samples were transferred either after air exposure or via an Ar-purged glove bag into a load-lock chamber. The results are indistinguishable. It is clear from Figs. 5 and 6 that the concentration of As_2O_3 is reduced after H_2 plasma passivation. This is consistent with previous *in situ* XPS studies and indicates that the surface is depleted of As.²¹ The Ga_2O_3 peak increases after treatment but this is difficult to interpret. Based on the *in situ* work of Friedel and Landesman,²¹ it seems unlikely that Ga_2O_3 is reduced by the H_2 plasma treatment at these low temperatures; however, we cannot rule out the possibility that the oxide has been reduced and has simply reformed rapidly despite the glove bag transfer. Note that the resolution in these experiments is insufficient to detect the presence of hydrogen on or below the surface.

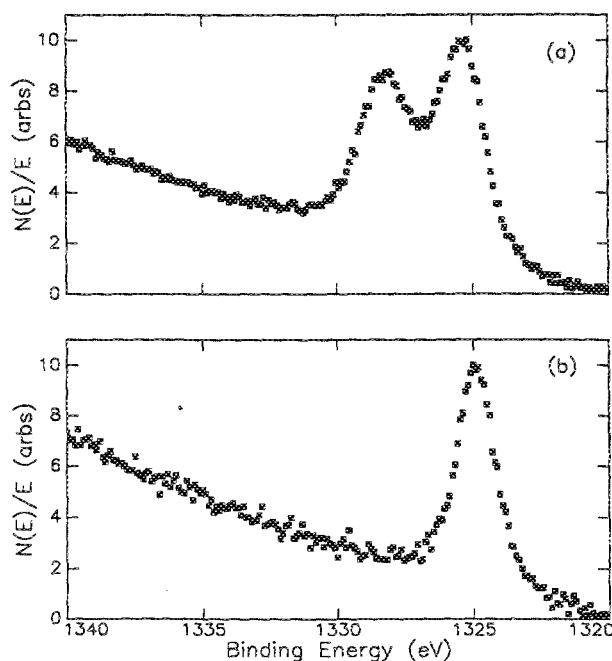


FIG. 5. As ($2p_{3/2}$) photoelectron spectrum (a) prior to and (b) after plasma treatment. In both cases, the GaAs wafers are preprocessed by dipping in concentrated HF for 30 s. Plasma conditions same as reported in Fig. 3(b).

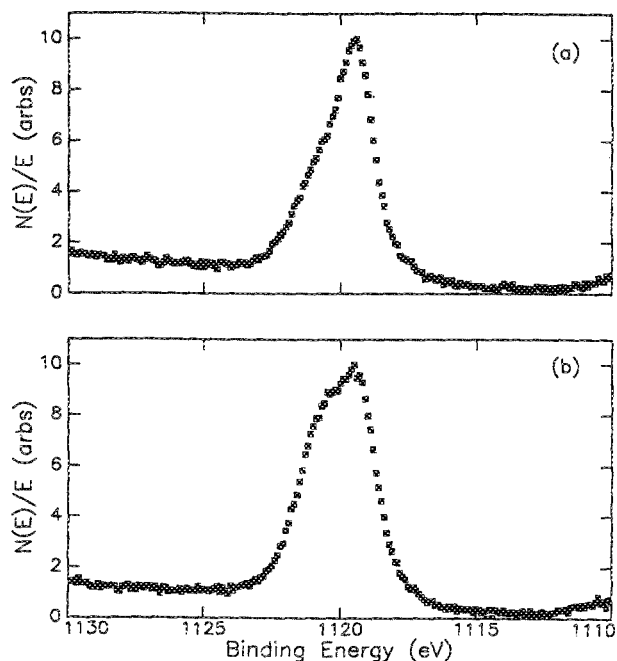


FIG. 6. Same as Fig. 5 except Ga ($2p_{3/2}$) peak.

The removal of As from the surface is further seen by examining XPS signals as a function of photoelectron escape angle. From such data we deduce for HF-treated samples that the Ga:As atomic ratio is approximately 1.1. After plasma treatment, this ratio is approximately 2.0. Removal of As from the surface is consistent with the large increases in PL intensity; the As antisite defect lies near midgap and is therefore an efficient recombination center.³² As pointed out above, reduction in this defect state density does not imply that the Fermi level is unpinned.

V. SCHOTTKY DIODE CHARACTERISTICS

To further evaluate the consequences of low-temperature H_2 plasma passivation, we treated *n*-type GaAs with an alloyed Au-Ge-Ni ohmic contact already in place on the back side of the wafer. On the front side, Schottky diodes were fabricated both prior to and after plasma treatment. No change was observed in the characteristics of the diodes formed prior to plasma treatment. It had been hoped that reduction of the surface recombination velocity adjacent to the diode might have improved the diode performance but this was not observed. However, for diodes fabricated on passivated areas of the same wafer (i.e., after plasma treatment), the barrier heights showed small but significant increases (Table I). Consistent with a reduction in surface state density, we found barrier heights to depend slightly on the metal used to make the contact. The barrier heights are also close to those reported in the literature for clean GaAs prepared under UHV conditions. Note that such clean surfaces are still thought to be "pinned."

The increases in Schottky barrier height shown in Table I are significant when compared to the values for untreated

TABLE I. Barrier heights (ϕ) and ideality factors (n) for Schottky n -GaAs diodes.^a

Metal	n	ϕ (eV)	ϕ (eV) (clean GaAs) ^b
Au	1.06	0.89	0.88
Ti	1.01	0.84	0.83
Pt	1.02	0.85	...

^aFor untreated n -GaAs: $n = 1.02$ and $\phi = 0.75$ eV.

^bE. H. Rhoderick and R. H. Williams, *Metal-Semiconductor Contacts* (Clarendon, Oxford, 1988), p. 70.

control samples and provide increased noise margin in MES devices. The increases are also consistent with an increase in band bending as the Fermi level shifts to a lower pinning position closer to the Ga antisite defect. Evidently, the increase in band bending is more than compensated for by the reduction in the near midgap As antisite defect state density.

VI. LONG-TERM AND THERMAL STABILITY

A natural question to ask when dealing with passivated GaAs surfaces is how stable is the treatment. With few exceptions, most treatments reported in the literature do not exhibit good long-term stability. In most cases, PL intensity degrades within hours after treatment. For hydrogen plasma treatments, this issue does not appear to have been addressed. As the data in Fig. 7 show, the long-term stability of the treatment described is excellent. Within the first few days, the PL intensity drops between 10% and 30% of its peak value (which is usually more than 10 times the initial value). After this time period, the PL intensity remains constant within our experimental precision for at least 40 days.

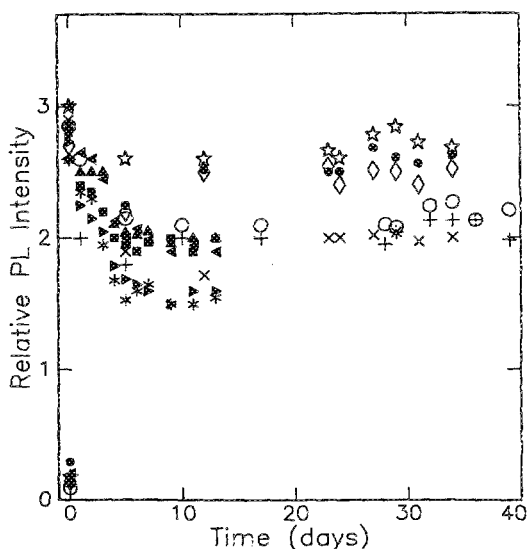


FIG. 7. Passivated GaAs PL intensity data for 12 different samples over a period of more than 30 days. After an initial decrease of 10%–30% of the enhanced value, the PL intensity remains constant indefinitely.

This provides more than enough time to complete the processing sequence for all types of GaAs-based electronic and photonic structures and suggests that devices made using such a passivation should have enhanced performance indefinitely.

The XPS data in Fig. 8 help us to understand how the passivated GaAs surfaces degrade initially. No changes are observed in Ga $2p$, Ga $5d$, or As $5d$ XPS data, but the As $2p$ peaks show a component growing in after 2 days. The chemical shift for this peak is consistent with its being As_2O_3 . After 2 days, the magnitude of this peak is relatively stable. The growth of As_2O_3 instead of As_2O_5 is consistent with a surface deficient in As. Its formation may result from As leaching out from the bulk or oxygen diffusing through the thin Ga_2O_3 (hydride) layer. A second plasma treatment after more than 20 days results in complete recovery of the maximum PL signal.

Another important question to ask is how stable the passivated surfaces are to heat treatment. We find the PL intensity degrades by two times when passivated samples are heat treated for 30 s at either 250 or 400 °C. However, after such heat treatment, the PL intensity remains stable indefinitely at this reduced level and is still enhanced by at least five times relative to untreated material. In particular, this implies that the hydrogen plasma surface treatment may be performed either before or after ohmic contact alloying, allowing considerable latitude for its insertion into the processing sequence.

VII. CONCLUSIONS

We have shown that real-time, *in situ* monitoring of photoluminescence from GaAs is essential for optimizing plasma passivation of the native-oxide-contaminated sur-

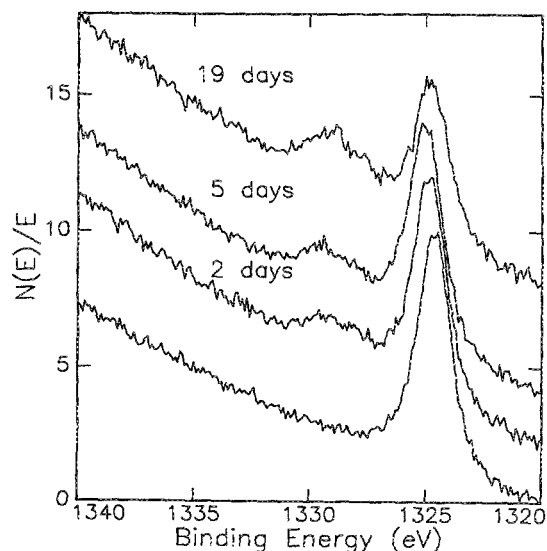


FIG. 8. As ($2p_{3/2}$) XPS peak as a function of time exposed to the ambient. A higher binding-energy peak attributed to As_2O_3 appears after the first two days and coincides with the initial decrease in PL intensity shown in Fig. 7. After 19 days, the surface is contaminated with C and the primary As peak is anomalously attenuated.

face. In particular, pressure and exposure time are critical parameters in rf diode treatments because of the competition between passivation and damage. At low pressure, timing is critical as overexposure of the wafer to the plasma results in substantial damage and a reduction in quantum yield. At higher pressures, exposure time is no longer a critical parameter and the processing window is large and forgiving.

From XPS measurements, the passivation mechanism is attributed to removal of excess As on the surface and formation of a stable Ga oxide. Removal of As presumably reduces the As antisite defect density and thereby the surface recombination velocity. For *n*-type material, the Schottky barrier heights increase, although the Fermi level is likely still pinned. This increase in barrier height is clearly useful for reducing leakage currents in reverse-biased devices.

The low-temperature passivation described shows excellent long-term stability and resistance to short heat treatments. As such, it should be useful for preprocess cleaning and postprocess damage removal.

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