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JSS FOCUS ISSUE ON GAN-BASED ELECTRONICS FOR POWER, RF, AND RAD-HARD APPLICATIONS

Optimized Process and Tool Design for GaN Chemical Mechanical Planarization

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In this paper, we present a systematic approach to the gallium nitride (GaN) chemical mechanical planarization (CMP) process through evaluating the effect of crystallographic orientation, slurry chemistry and process variables on the removal rate and surface quality responses. A new CMP process and a complementary tool set-up are introduced to enhance GaN material removal rates. The key process variables are studied to set them at an optimal level, while a new slurry feed methodology is introduced in addition to a new tool set up to enable high material removal rates and acceptable surface quality through close control of the process chemistry. It is shown that the optimized settings can significantly improve the material removal rates as compared to the literature findings while simultaneously enabling a more sustainable process and potential removal selectivity against silica.
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GaN is defined as the silicon of the future based on its adaptability to a wide range of devices including microelectronics and photonics.[1] It is used for the high power, high temperature and high frequency microelectronics device manufacturing due to its wide bandgap energy and high electron mobility including heterojunction field effect transistors (HFETs) and its derivatives (Metal Oxide Semiconductor HFETs-MOSHFET and Metal Oxide Semiconductor Double HFETs-MOSDHFETs) as a barrier layer,[2] high electron mobility transistors (HEMTs) for power switching with AlGaN/GaN stacking as a buffer layer[3] as well as for the applications for heterojunction bipolar transistors (HBTs) and bipolar junction transistors (BJTs).[4] In addition, GaN is suitable for photonics device manufacturing based on its direct bandgap. It is designed into the light emitting diodes (LEDs) and ultraviolet LED (UVLED) manufacturing as an active region.[5,6] These applications require the polishing and optimal planarization of the GaN layers where CMP is the method of choice due to enabling nano-scale smoothness on the wafer surfaces in addition to enabling material and topographic selectivity through advanced slurry formulations.[7] Yet, the main problem in integration of GaN is related to the challenges in its defect free deposition and its hard and brittle nature, which makes it difficult to polish and planarize in an integration scheme without creating surface defectivity, which can be defined as the elevated surface roughness, scratches, local pitting and protrusions, slurry particles and particles from the surroundings that might be left on the wafer surface.

The growth of thick and crystalline GaN films is very challenging due to the formation of the threading dislocations between the selected substrate and the GaN interface that can act as the short-circuit leakage paths.[1] Furthermore, it is also known that GaN films tend to crack above a critical thickness, which can even lead to the film and the substrate to fracture into separate pieces.[8] Many conventional deposition techniques fail to satisfy the defect free deposition of GaN on conventional substrates such as silicon. Therefore, substrates such as ZnO and SiC are experimented which have the same crystal structure as GaN (wurtzite).[9,10] The Ga-rich face of GaN [0001], tends to grow on Zn and C on the ZnO and SiC substrates, respectively.[10] Whereas, the N-rich face $[000\bar{1}]$ grows on the O and Si faces. Between these, SiC is a more suitable substrate for the commercial applications. The crystallographic polarity of the GaN affects the device properties as well as the chemical and mechanical response of the GaN surface in addition to the proper growth of the desired film.[11] It is further reported that the N face GaN tends to be rougher as compared to the Ga-face due to the nucleation mechanism driven by low adatom mobility on the N-face as compared to the Ga-face during the deposition. The utilization of plasma assisted molecular beam epitaxy (PAMBE) deposition was investigated to optimize the surface smoothness of the N-face by implementing additional control parameters.[10] Yet, it is clear that any investigation of the GaN surface treatment, such as CMP, has to take into consideration the polarity of the GaN substrates as well as the proneness of the GaN to fracturing under the applied forces.

Implementation of CMP on GaN planarization has long been studied with much better success as compared to the purely mechanical polishing methods.[12,13] Since the pure chemical dissolution rate of GaN is negligible, the mechanical removal provided by the submicron size abrasive particles was noted to be very critical for GaN removal after a chemically modified layer is formed on the surface.[12–15] The preliminary investigations held by using silica based slurries and KOH or NaOH based pH adjustment have shown improved polishing rates as well as surface quality post material removal. A removal rate of 17 nm/h (2.83 Å/min) was reached with 0.1 nm of average surface roughness (Ra) after 40 hours of polishing.[16] It was noted that the main challenge in reducing surface defectivity is driven by the initial poor surface quality of the substrate as the substrate surface quality strongly depended on the growth conditions. Furthermore, the polarity of the bulk and epitaxial GaN films was also outlined as the difference in driving the variability in the surface roughness post CMP. The Ga face of GaN is altered to form Ga_2O_3 on the surface, which is removed by the relatively harder slurry abrasive particles.[13] By using acidic slurry (pH 2) and adjusting the slurry solids loading, 0.65 nm roughness (Ra) and 165 nm/h (27.5 Å/min) removal rates were obtained on the Ga-side. The removal mechanism on the N-side GaN is also very well described as formation of nitrogen terminated layer with one negatively charged dangling bond on each nitrogen atom, adsorption of hydroxide ions, formation of oxides and dissolution and removal of the oxides.[14,15] Again by using silica-based slurry, the material removal rates were reported to vary from 400 to 1100 nm/h (66.7 to 183.3 Å/min) and the surface roughness values within 0.4–1.1 nm range (RMS-root mean square) were reached.[14] Alumina based slurries in the presence of sodium hypochloride (NaOCl) on the other hand, resulted in residual particles to be left on the surface and required a buff step for post CMP cleaning while removal rates of 50 nm/min (8.3 Å/min) were obtained.[17] More recent study comparing the alumina versus silica slurries in the acidic pH range in the presence of potassium permanganate ($KMNO_4$) as an oxidizer concluded that

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39 nm/hr (6.5 Å/min) and 85 nm/hr (14.17 Å/min) removal rates were obtained with silica and alumina slurries, respectively. The root mean square (RMS) roughness values were reported to be 0.13 nm.[18] By adjusting the slurry flow rate, the authors were able to improve the removal rates to 228–687 nm/hr (380–114.5 Å/min) with a 1 nm RMS roughness obtained.[19] The use of metal (Fe^{+2}) catalyst,[20] as well as conducting GaN CMP under UV light by using photocatalytic titania (TiO_2) nanoparticles as abrasives[21] have been observed to increase the GaN removal rates on the Ga-face of the material.

In our earlier work, we have compared the material removal rates (MRR) and the surface quality responses of the Ga-face, N-face GaN coupons as well as a 2" GaN wafer deposited on silicon substrate.[22] The evaluations based on slurry pH and polishing pad type were observed to improve the material removal rates when the chemical component of the CMP process is enhanced through use of softer pads and high pH where the GaN dissolution reaction can be driven toward the products side.[14] In this study, we evaluate the impact of CMP variables on bulk GaN coupons as a function of pH, slurry solids loading and flow rate and downforce by using baseline silica slurry. In addition to these standard CMP variables, slurry temperature and the relative surface energy changes were evaluated as a function of the surface polarity of the GaN coupons. In order to control the surface defectivity (by means of surface roughness evaluation), slurry stability as well as the GaN surface electrical charge values were measured as a function of pH. We also introduced new slurry feeding strategy and polisher set-up, which promotes the GaN polishing rates significantly.

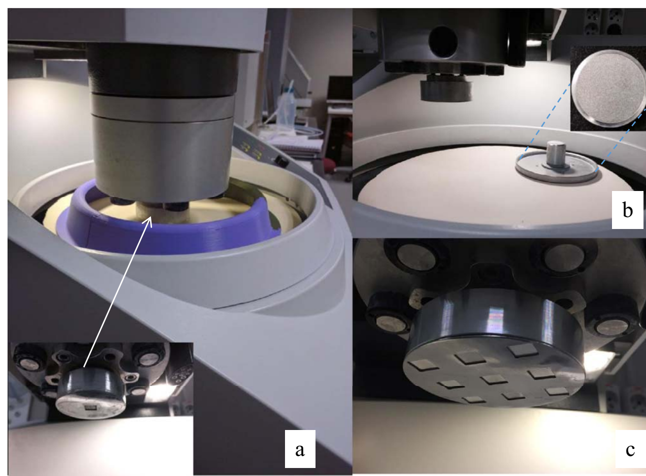

Figure 1. Desktop Struers Tegrapol-31 polisher (a) with the standard single wafer holder and slurry pool adopted (b) platen and the conditioning disk for the standard CMP testing and (c) multi-wafer holder designed to reduce pressure by adjusting the downforce through changing the number of wafers held during the polishing experiments.

Materials and Methods

Materials.—CMP evaluations were performed on bulk GaN coupons with 10 × 10 mm dimension and 1 mm thickness. Bulk GaN coupons with (0001) orientation were selected on purpose to prevent any defectivity driven by the dislocation threads originated from the GaN/substrate interface. Both Ga-face and the N-face were available on the bulk substrates. A baseline silica slurry with uniform particle size distribution and 0.1 μm mean particle size provided by BASF SE, Germany was used for the CMP experiments. Slurry pH was stable at pH 6.42 and it was adjusted by adding HCl and NH_3 as needed. Slurry stability was checked through particles size measurements by using static light scattering technique with Coulter LS 13 320 Instrument Universal Liquid Module. Background DI water pH and temperature were adjusted as needed to maintain the slurry conditions during the particle size measurements and evaluate the stability precisely. IC 1000/Suba IV stacked pads were used after aging for 100 hours to accomplish a soft pad/wafer interface and promote removal rates as per our observation in the earlier studies.[22]

Methods.—*Chemical mechanical polishing experimental.*—CMP experiments were conducted on a tabletop Struers Tegrapol-31 polisher. Figure 1a illustrates the polisher set up with the modifications made to form a slurry pool (which is utilized as a part of the new slurry feeding methodology) and the single wafer holder. Figures 1b and 1c show the standard polishing platen with 3M conditioner and the multi-wafer coupon holder to adjust the applied pressure by changing the number of wafers placed during the polishing experiments. CMP tests were conducted at 30 N downforce, which is equivalent to ∼43 psi pressure on 10 × 10 mm sample size. To adjust the pressure to lower values, the multi-wafer holder was utilized and 3.3, 6.2, 8.7, 14.5psi polishing performances were also evaluated. GaN samples were polished for 5 minutes and the material removal rates were reported as Å/min through weighing the samples pre and post polish by Swiss Made ES125SM model precise scientific balance (five digits after the decimal point, 0.01 mg accuracy). All samples were cleaned in ultrasonic bath with pH-adjusted water at pH 9 for 5 minutes and dried with nitrogen gas before they were characterized. Slurry pH was evaluate at pH 3, 6 and 9, solids loading was tested at 5,10, 15, 20 and 25 wt%, and the flow rate values of 20, 50 and 100 ml/min were experimented to optimize the material removal rates.

Surface characterization experimental.—GaN wafers were characterized on both Ga and N-faces thoroughly in order to evaluate their CMP performances in terms of material removal rates as well as the post polishing surface quality. Both crystallographic faces were tested for wettability, surface energy and work of adhesion in addition to the FTIR/ATR surface scans and surface charge values which were correlated to their CMP behavior as a function of slurry pH as a preliminary evaluation.

Wettability characterization.—Surface wettability analyses were performed by using a KSV ATTENSION Theta Lite Optic Contact Angle Goniometer using the sessile drop method. Contact angles of five drops of DI water were measured on each sample and the averages were reported with the standard deviation values. The drop images were stored by a camera and an image analysis system calculated the contact angle (Θ) from the shape of the drops. Surface free energy (SFE) and the work of adhesion (W_a) were calculated by using acid-base method after measuring contact angles with liquids of different polarity as given in the literature (ethylene glycol, formamide, glycerol and DI-water).[23] The surface free energy and the W_a are helpful in determining the propensity of particle adhesion on the wafer surfaces as well as the material removal potential based on surface chemical modification.

Surface topography and roughness characterization.—The surface topographies of the GaN coupons were examined by Nanomagnetics Atomic Force Microscope (AFM) using contact mode. Surface roughness values were recorded on 5 × 5 μm scan area consistently and reported as an average of minimum three measurements taken on the samples. In order to determine the root mean square (RMS) surface roughness values properly, the line scans were also performed by excluding the major surface defectivity such as particles stacked on the surface or deep scratches when needed. Yet, the full AFM scans are presented since the surface slurry particles left on the wafers are related to the insufficient surface charge, which is also studied as delineated in the following section.

Surface charge analyses.—Surface charge and zeta potentials of GaN samples for both Ga and N faces were measured by using streaming current measurement technique with SurPASS Instrument by Anton Paar. For the analysis of the zeta potential, 1mM KCL solution was used as an electrolyte while 0.05 M HCl was used for the pH titration. The 10 mm^2 GaN samples were mounted to the adjustable gap cell by orienting two coupons with the same crystalline orientation to face each other within the required gap for the flow conditions.

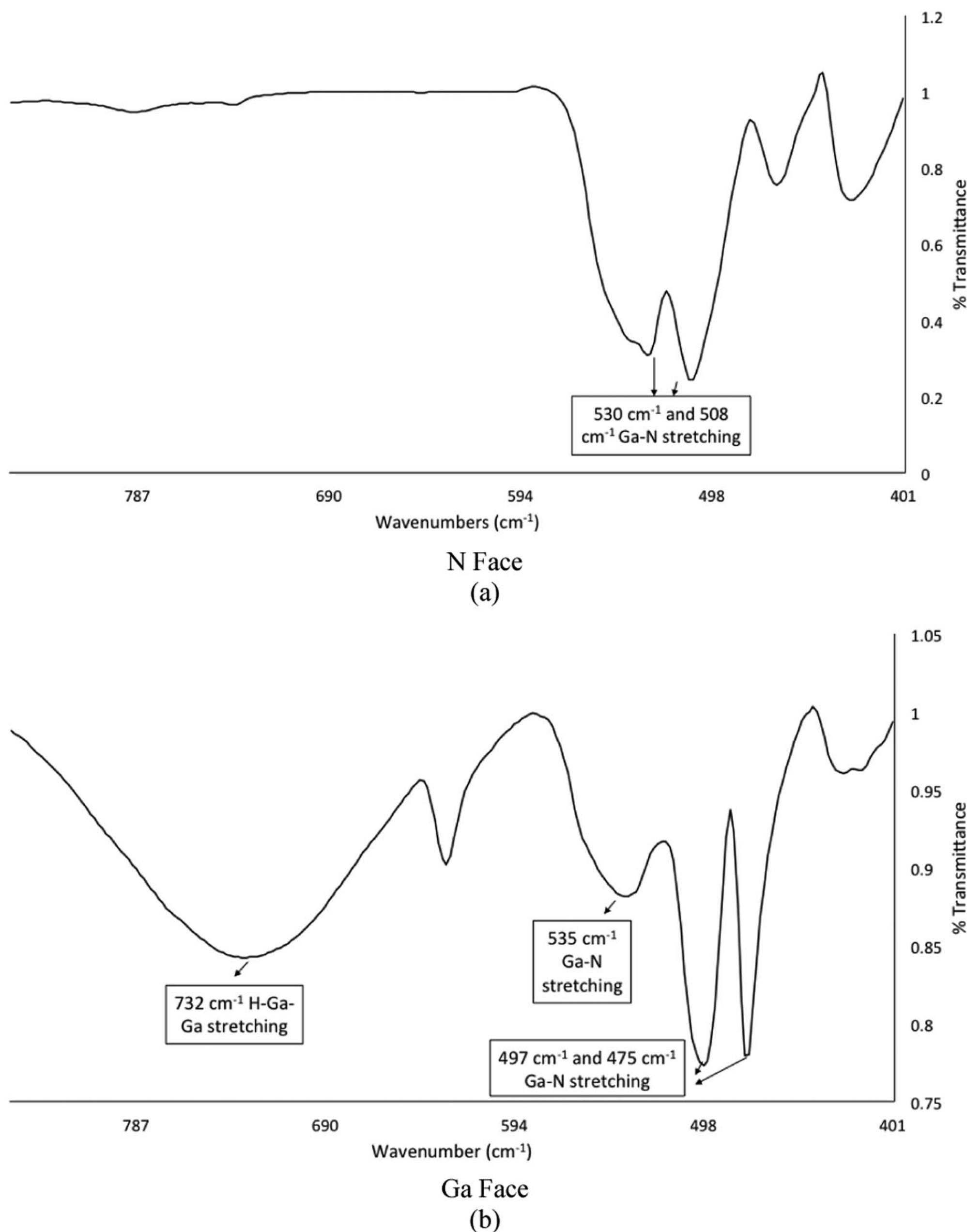

Figure 2. Surface FTIR analyses of (a) N Face and (b) Ga Face, based on crystallographic orientation.

Surface chemical composition analyses.—The crystalline nature of the GaN surfaces was studied through FTIR analyses by using an ATR crystal to be able to focus onto the top surface film. A Bruker Tensor 27 FTIR was used to detect the GaN related peaks on both the Ga-face and the N-face of the bulk coupons. Furthermore, pre and post CMP FTIR analyses were conducted as a function of pH to determine the relative changes in the chemical bond intensities and the impact of polishing on the chemically modified top film during the CMP operations.

Results and Discussion

The preliminary experimental evaluations were conducted on the bulk GaN coupons by analyzing the effect of crystallographic orientation of GaN to the surface wettability, electrical charge and surface energy properties. Following these analyses, CMP optimization was performed by testing the slurry pH, solids loading, flow rate, temperature and applied downforce by means of improved material removal rates and the surface quality. Based on the optimal settings, a new CMP process and tool set-up is suggested which brings the synergy in reducing slurry consumption for polishing of bulk layer of GaN films while effectively enhancing material removal rates and protecting the surface quality by adjusting the tool and process configurations.

Characterization of the crystallographic faces of the GaN and related surface properties.—In order to understand the effect of crystallographic orientation of bulk GaN coupons on CMP performance, initially the FTIR spectra were taken on both sides of the $\langle 0001 \rangle$ crystallographic orientation to identify the Ga-rich face [0001], and the N-rich face $[000\bar{1}]$ of the bulk coupons.[11] Figure 2 shows the FTIR spectra of the N-face and Ga-face GaN with the primary peaks identifying the atomic vibrations related to the specific crystalline orientation. It can be seen in Figure 2a that the main peaks of G-N vibration are detected at 530 and 508 cm^{-1} wavelengths

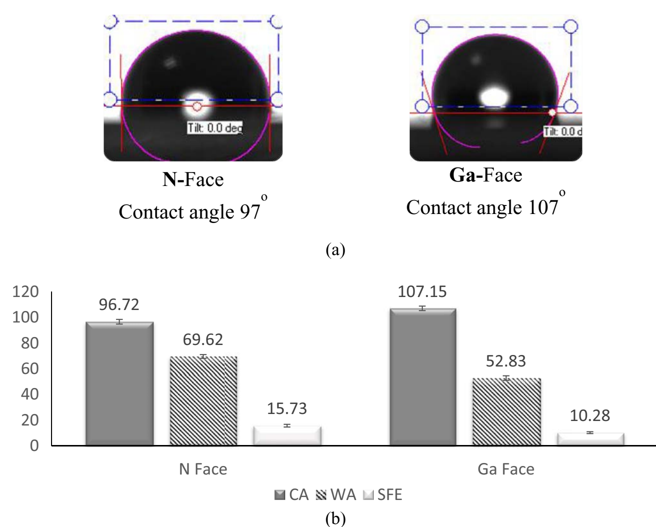

Figure 3. Surface characterization of the N-face and Ga-face of the GaN coupons through (a) contact angle measurement showing the DI-water droplets on the surfaces and (b) contact angle, work of adhesion and surface free energy values calculated by measuring the contact angle with liquids of varying polarity.

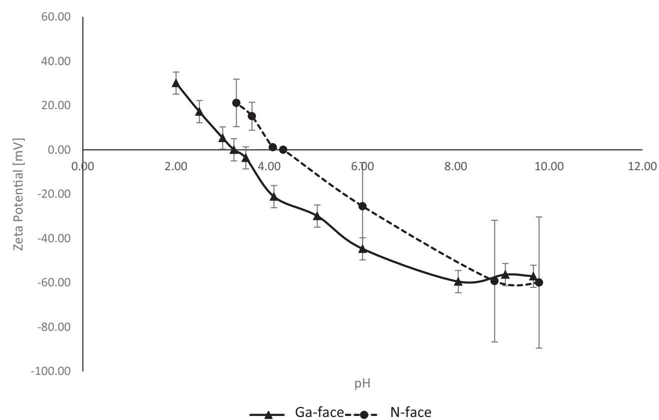

Figure 4. Surface charge measurements on the Ga-face and N-face of the GaN bulk coupons.

corresponding to the N-face,[24] whereas the Ga-face shows the intense H-Ga-Ga peak at 732cm-1 wavelength that is missing in the N-face spectra.[24] The additional Ga-N stretching is also seen on the Ga-face at 535, 497 and 475 cm^{-1} wavelengths in agreement with the literature findings.[24–26]

Once the Ga-rich and N-rich surfaces were identified, wettability, surface energy and work of adhesion values were evaluated as can be seen in Figure 3. The image of the water droplet illustrated in Figure 3a shows a higher contact angle with the Ga-face, which is 107° and 10° higher than the N-face GaN. This statistically significant difference between the contact angles of the two faces was found to be a useful tool in verifying the crystallographic orientations when needed. Although there is a difference between the wettability of the different sides, it is clear that the GaN shows a contact angle of 90° and higher that is an indicator of its hydrophobic nature. Furthermore, the work of adhesion (W_a) and surface free energies (SFE) were also measured (as per the procedure given by Bargir and co-workers[23]) for both the orientations and reported in Figure 3b. The W_a values are beneficial in estimating the propensity of the surface to adhere other species and can be utilized in predicting the post CMP cleaning efficiency of the selected crystallographic orientations. In this case, the Ga-face had a lower Wa value (52.83 J/m^2) as compared to the N-face (69.62 J/m^2) indicating that the N-face is more prone to the attachment of particles post CMP. This characteristic was further verified by the surface charge measurement results as reported in Figure 4. The iso-electric point (IEP) of Ga-face was measured to be at pH 3.24, whereas the N-face was detected to be at pH 4.30. Hence at the given slurry pH of 6.4 the Ga-face is more negatively charged (−46 mV) as compared to the N-face (−23 mV). Consequently, the higher negative charge on the Ga-face results in more repulsive forces with the negatively charged silica particles (IEP of 2.2) and can be cleaned more easily. Furthermore, the surface energy values of 10.28 and 15.73 J/m^2 calculated on Ga-face and N-face, respectively indicate that the Ga-face is more stable as compared to the N-face, which is in agreement with the literature[10,11] indicating that the N-face decomposes more easily as compared to the Ga-face during the GaN growth. This observation is also consistent with the high standard deviations observed on the surface charge measurements of the N-face GaN as compared to the Ga-face.

CMP performance evaluations.—*CMP performance as a function of pH on Ga-rich and N-rich faces of GaN.*—In order to evaluate the CMP performances of the Ga-rich and N-rich faces, preliminary polishing analyses were performed as a function of pH. Table I summarizes the material removal rate (MRR) results on the N-face and Ga-face of GaN coupons at 30 N downforce and 10 ml/min slurry flow rate at pH 3, 6 and 9 without using a conditioner. It can be seen that the increase in pH results in increasing MRR values on both sides of GaN. However, the Ga-rich side has removal rates that are at least an order of magnitude higher as compared to the N-rich side. This can be attributed to the nature of material removal on the Ga-rich face, which occurs through formation of Ga_2O_3 on the wafer and its removal by the relatively harder slurry abrasive particles.[13] On the other hand, the N-side GaN requires the formation of a nitrogen terminated layer with one negatively charged dangling bond on each nitrogen atom followed by the adsorption of hydroxide ions leading to the formation of oxides and dissolution and removal of the oxides.[14,15] Therefore, the CMP performance optimization studies were focused on the N-rich face where the MRR is limited and needs to be improved mainly for the applications in LEDs.

Figure 5 summarizes the material removal rates and the surface roughness values of the N-face GaN at pH 3, 6 and 9. It can be seen that the increasing pH promoted the MRR values as it helps providing additional hydroxide ions required for activating the material removal on the N-face. The MRR values increased from 47 Å /min at pH 3 to 110 and 142 Å/min at pH 6 and pH 9, respectively. As the material removal is enabled by increased pH, the surface roughness was also observed to decrease as can be seen in Figure 5b. The increase in the chemical activity resulting in GaN removal was further verified with the FTIR spectra collected pre and post CMP as a function of pH as it can be seen in Figure 6. While the FTIR spectra exactly overlapped for the GaN coupons pre and post CMP polished at pH 3 (Figure 6a), the Ga-N vibration peaks at 530 and 508 cm^{-1} wavelengths were observed to decrease post CMP at pH 6 and pH 9 indicating removal of GaN.

In terms of surface quality performance, the decrease in the surface roughness with the increasing pH can be explained based on two phenomena. First of all, the decrease in the slurry pH results in agglomeration due to the reduction of the surface charge of the silica particles (IEP of pH 2.2). Indeed the % volume based particle

Table I. Material removal rate results on the N-face and Ga-face of GaN coupons at 30 N downforce and 10 ml/min slurry flow rate as a function of pH.

Slurry pH Value	N-Face MRR [Å/min]	Ga-Face MRR [Å/min]
3	47.2	1313.9
6	110.1	1887.6
9	141.6	2269.6

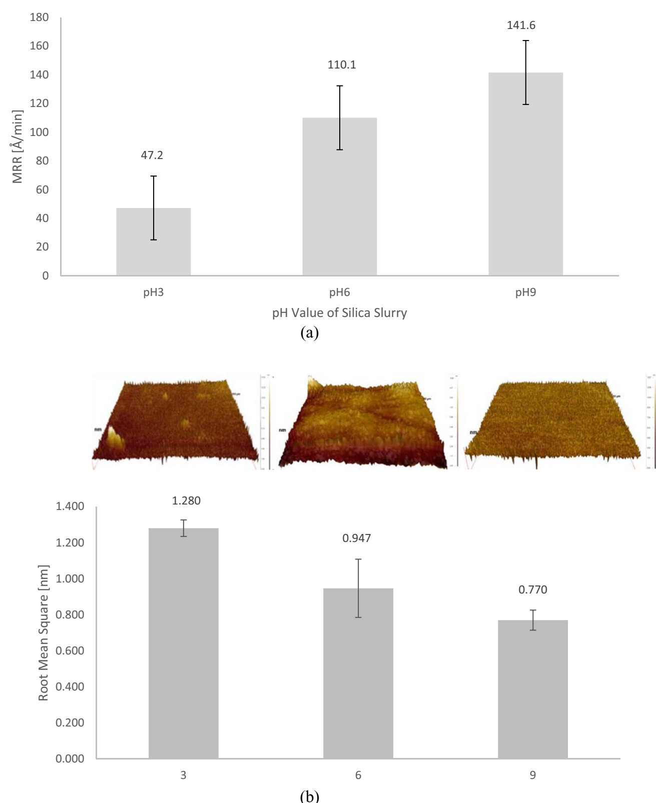

Figure 5. CMP performance of N-face GaN as a function of pH (a) material removal rate and (b) surface roughness with 10 wt% silica slurry at 30 N downforce.

size analyses conducted on the slurries prepared for CMP at pH 3, 6 and 9 measured a mean particle size of 106, 105 and 74 nm, respectively. These results confirm the formation of soft agglomerates (due to coagulation) in the slurry, based on larger mean particle size of the slurries at lower pH, which tends to mechanically abrade the surface and generate higher levels of defectivity.[27] Furthermore, Figure 7 compares the surface free energy and the work of adhesion values pre and post CMP conducted at pH 3, 6 and 9. It is clearly seen that, polishing in general increases both the surface free energy and work of adhesion due to the opening of fresh surface layers. Yet, the work of adhesion is the highest when the CMP is conducted at pH 3 meaning that the propensity of the slurry particles to adhere on the surface increases. The particles remaining on the surface can be seen in the bottom left corner of the AFM micrograph corresponding to the CMP performed with the slurry at pH 3 in Figure 6b. Therefore, both the slurry agglomeration and the increase in the work of adhesion result in higher surface roughness in the case of CMP conducted with the slurry adjusted to pH 3.

CMP performance optimization as a function of mechanical components of the process.—In order to improve the material removal rates on the N-face GaN, the mechanical components of the CMP process were evaluated at the optimized slurry pH value of 9 and using a 3M pad conditioner in-situ. As the mechanical forces are transferred to the wafer surface as a function of pressure per particle, the slurry solids loading, flow rate and the applied downforce were varied to change the pressure applied on the abrasives during the polishing operation.

The slurry solids loading was experimented at 5, 10, 15, *20* and 25 wt% as illustrated in Figure 8a. It was observed that the highest removal rate was achieved at 10 wt% slurry solids loading, which was 220 Å/min at 30 N downforce. The use of pad conditioner also increased the MRR value significantly from 141.6 Å/min (Figure 5a) up to 220 Å/min. Beyond 10 wt%, the MRR values tend to decrease, which can be attributed to the surface saturation of particles resulting in the rolling action of the abrasive particles on the surface and hence decreasing the efficacy of the mechanical abrasion, known as the contact area based material removal mechanism promoting chemical activity and demoting mechanical abrasion.[7] Following the slurry solids loading optimization, the slurry flow rate impact was evaluated at 20, 50 and 100 ml/min as seen in Figure 8b. It was also observed that beyond 20 ml/min, the excessive slurry flow rate did not affect the material removal rate and even decreased the MRR at 100 ml/min of slurry flow. This trend can also be explained based on the contact area based material removal to become more pronounced as observed in the effect of slurry solids loading. Finally, Figure 8c illustrates the impact of downforce which was experimented by using10 wt% slurry solids loading at 20 ml/min slurry flow rate by using the multiple-wafer sample holder illustrated in Figure 1c. In this case, it was observed that the highest removal rate (346 Å/min) was obtained at 10N downforce, which corresponded to 14.5psi pressure. The effect of pressure on the removal rate response can be explained in two ways; (i) the higher the pressure, the load per particle increases which promotes the mechanical action of the abrasives on the surface and expected to promote the removal rates, and (ii) the higher the pressure it becomes more difficult for the abrasive particles (slurry) to be fed in between the sample and the polishing pad, reducing the chemical activity and the total surface area of particles in contact with surface, demoting the removal rates. Hence, the high pressure tends to result in more mechanically driven removal mechanism reducing the number of particles engaged in polishing by reducing the total contact area. In this case, 10 N downforce seems to be the optimal to promote both the chemical and the mechanical actions and increase the removal rates. Based on the mechanical force adjustments, the optimal level of abrasion was obtained at 10 N downforce with 20 ml/min slurry flow rate and 10 wt% solids concentration.

CMP performance optimization as a function of chemical components of the process.—The chemical reaction that defines the GaN dissolution for the N-rich surface is given in Equation 1 as described by Weyner and co-workers.[12] Based on this reaction, the chemical decomposition of GaN results in formation of ammonia (NH_3) in the products. Although the high pH values obtained by adjusting slurry pH to 9 by NH_3 addition helps increasing the material removal rates by promoting the hydroxyl ion formation, the resulting NH_3 in the products tend to push the reaction backwards resulting in a decrease in the material removal rates. Therefore, a series of experiments were conducted intending the neutralization of the ammonia in the reaction products.

$$GaN + OH^- + 3\,H_2O \rightarrow Ga\,(OH)_4^- + NH_3 \qquad [1]$$

Figure 9a illustrates the material removal rate response for the slurry adjusted to pH 9 by NH_3 addition as compared to the in-situ neutralization of NH_3 on the pad surface by adding 0.1 M HCl through the secondary slurry flow line (20 ml/min slurry flow at 30 N downforce without conditioner). It can be seen that the MRR value of 157 Å/min increased to 189 Å/min by in-situ neutralization of the NH_3 in the products pushing the dissolution reaction forward. Any ammonium chloride (NH_4 Cl), which may be forming out of this reaction may slightly increase the ionic strength as a salt that is highly soluble in water. In order to prove the concept, the same experiment was conducted by using a HCl adjusted acidic slurry at pH 3 and adding NH_3 through the secondary slurry flow line as can be seen in Figure 9b. However, the MRR values further decreased when the neutralization was performed by using a base in the acidic slurry. Hence, it can be concluded that the initial impact of NH_3 is necessary to form the hydroxyl ions on the surface as a reactant in Equation 1, whereas, the addition of HCl on the pad surface can neutralize the excessive NH_3 and further help pushing the reaction forward. Furthermore, the same CMP tests were also conducted on the silica wafers yet resulted in no material removal rate indicating that this chemical promotion is specific to the GaN polishing. However, this

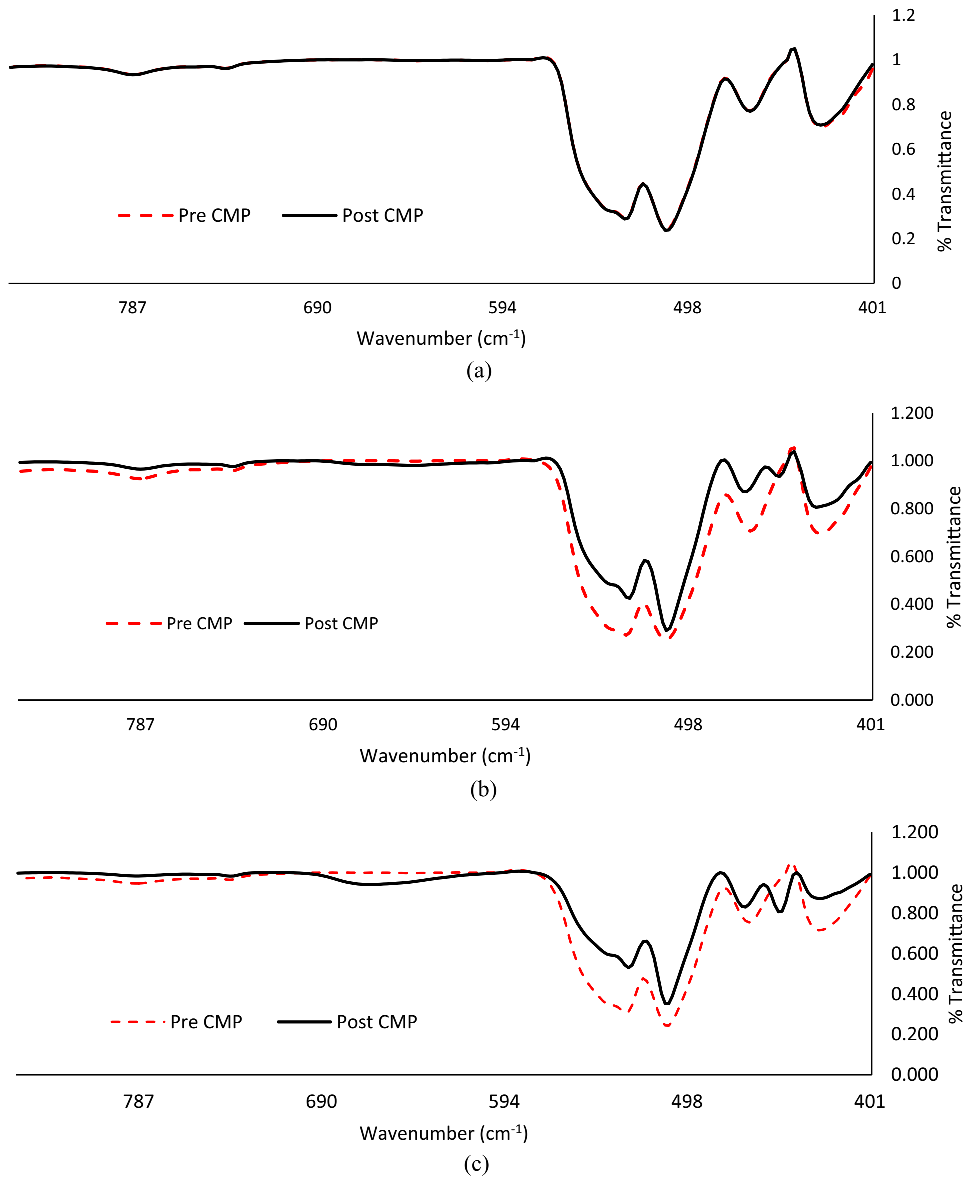

Figure 6. FTIR spectra of pre and post CMP application as a function of pH (a) pH 3 (b) pH 6 and (c) pH 9.

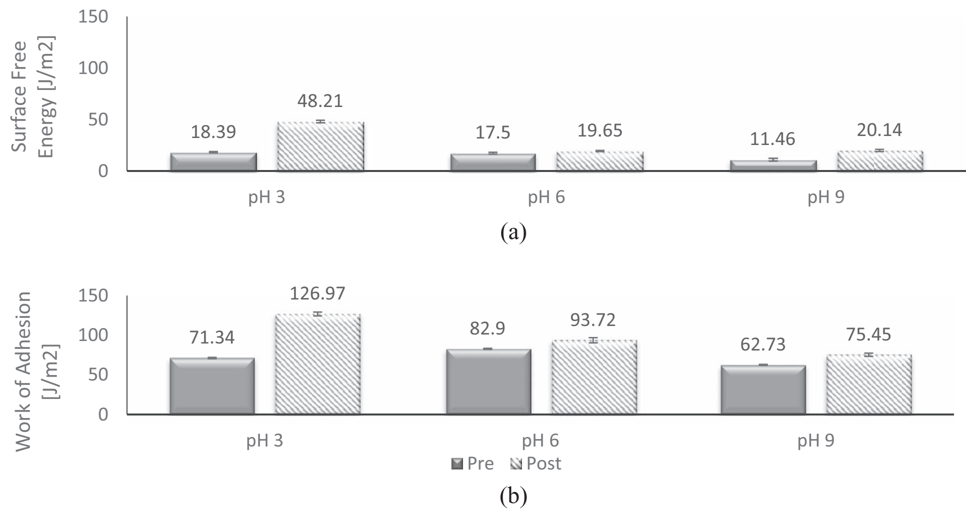

Figure 7. Characterization of (a) surface free energy and (b) work of adhesion on N-face GaN coupons pre and post CMP application at pH 3, 6 and 9.

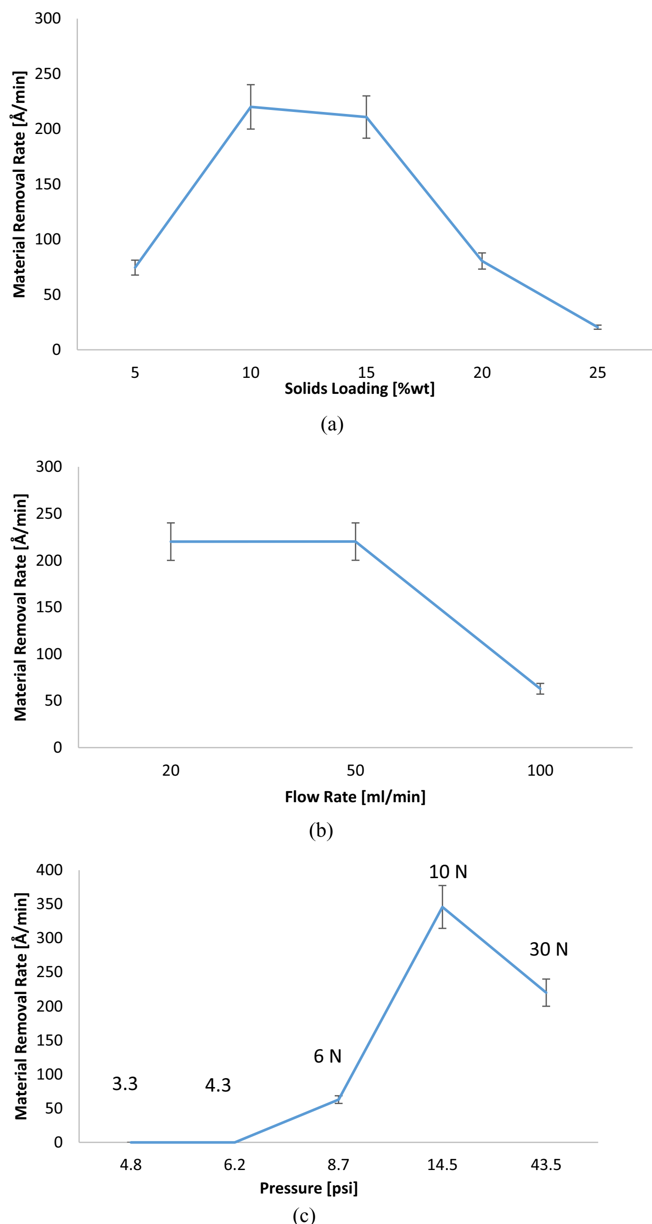

Figure 8. Material removal rate evaluations as a function of (a) slurry solids loading (b) slurry flow rate and (c) applied downforce (pressure) at pH 9.

is an indicator that GaN can be selectively removed against silica by using this specific process.

As an additional chemical modification, the effect of temperature was also evaluated on the material removal rate responses. Table II compares the material removal rates of the wafers polished with CMP slurry at pH 9 with and without the addition of 0.1 M HCl to the pad surface at 4°C and 20°C (ambient). It can be seen that the lowered temperatures helped increasing the material removal rates by 2 to 3 times. As the GaN CMP generates heat as a reaction product, the

Table II. Effect of temperature on material removal rates (Å/min).

Slurry Chemistry	Material Removal Rate (Å/min) 4°C	20°C
pH 9	475.0	158.5
pH 9 + 0.1 M HCl	503.2	188.7

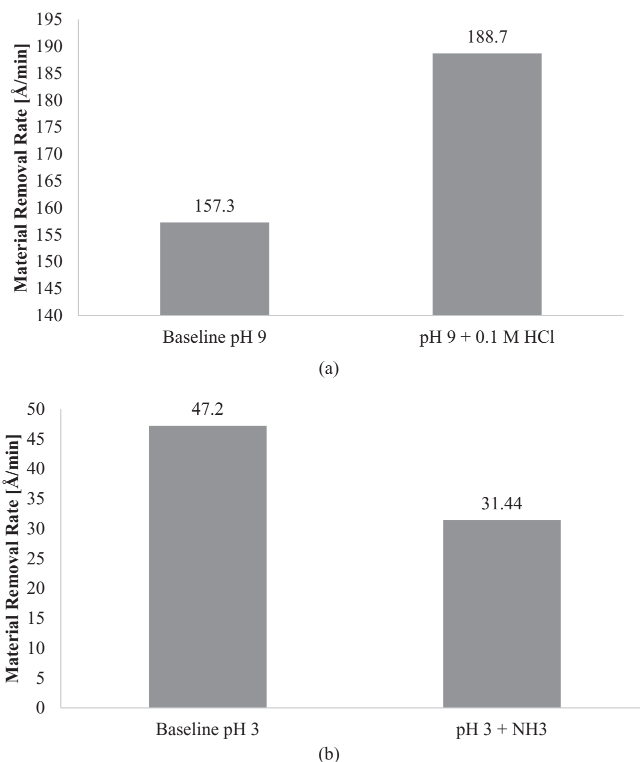

Figure 9. Material removal rate evaluations as a function of slurry chemistry by pushing the chemical reaction forward through (a) neutralizing the basic slurry with an acid and (b) neutralizing the acidic slurry with a base.

cooling of the platen surface also helps promoting the removal rates by pushing the reaction forward. Consequently, MRR values of ∼500 Å/min were achieved on the N-face GaN coupons by using a slurry at 4°C in combination with the HCl neutralization in-situ. However, the particle size analyses conducted with Coulter LS 13 320 (by using volume% distribution) to detect any agglomeration have shown that slurry neutralization with HCl increased the mean particle size. This is suspected to be due to agglomeration of the particles with the decreased pH. A further increase through more severe agglomeration was observed at 4°C slurry temperature as can be seen in Figure 10. Therefore, the surfaces were found to be more defective when lower temperature slurry was used for the processing as illustrated in Figure 11, although the removal rates were improved significantly. Hence, the trade-off between improving the removal rates and maintaining the surface quality still remains with configurations in process set-up required.

Optimized CMP tool set-up and process configurations.—Based on the improvement of the material removal rates of the N-face GaN with the in-situ addition of HCl, a new tool configuration was also experimented by adopting a slurry collector pool around the polishing platen as can be seen in Figure 1a. In this configuration, the pool was filled with the slurry (200 ml) and the CMP was performed with and without flowing 0.1M HCl solution at 20 ml/min flow rate. By using the slurry pool configuration, it was observed that the removal rate without the HCl addition was 94.35 Å/min as the generated NH_3 forced the dissolution reaction to go backwards. However, when the HCl flow was on, MRR value of 157.22 Å/min was reached which was equivalent to the polishing with just the pH 9 slurry by continuously flowing the slurry at 20 ml/min flow rate and at 30N downforce. This configuration may help conserve slurry for the bulk GaN polishing, which can be followed by a buff process by using fresh slurry.

In order to finalize the optimization, the best processing conditions including the mechanical and the chemical improvements were tested

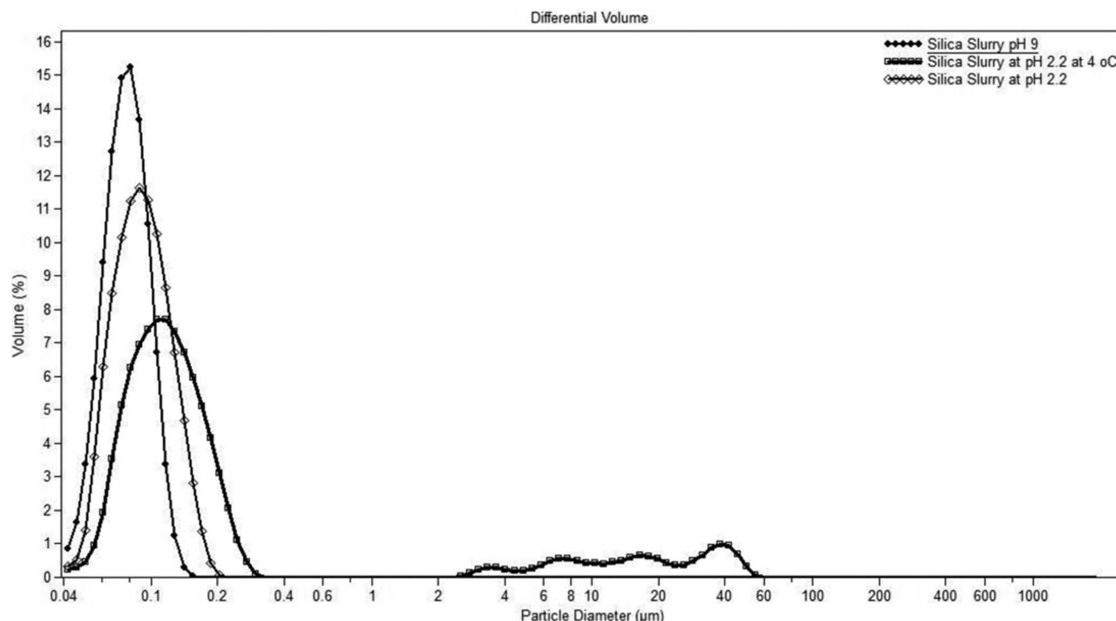

Figure 10. Particle size distribution analyses by volume% for the silica slurry at pH 9, silica slurry after addition of 0.1 M HCl into a pH 9 slury (changing the pH to 2.2) and HCl added slurry at 4°C.

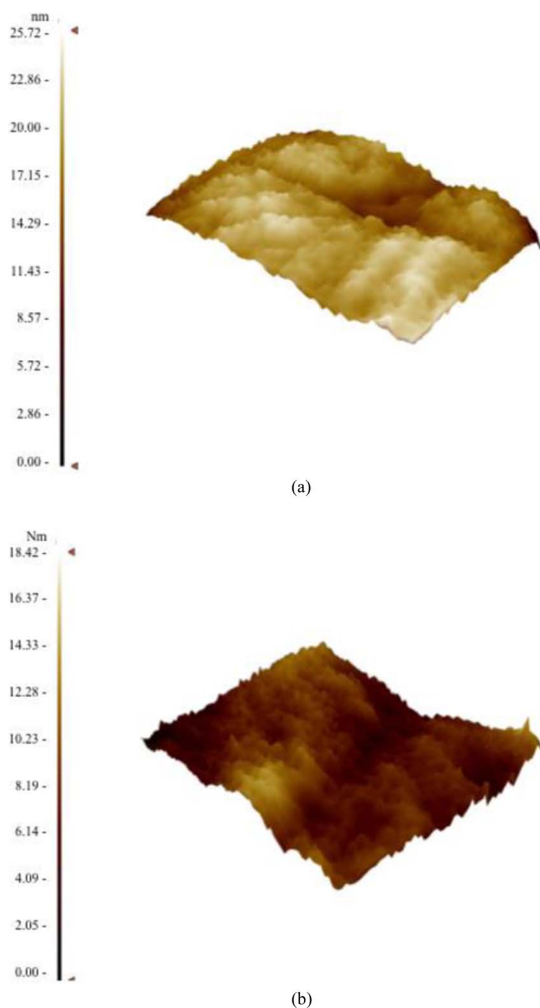

Figure 11. Surface quality analyses through AFM images for the GaN N-face coupons polished with slurry at pH 9 with 0.1 M HCl addition at (a) 20°C and (b) 4°C.

on the N-face GaN coupons. By using 10 wt% slurry solids loading at pH 9 and 4°C temperature and 20 ml/min flow rate with 0.1M HCl flown through the secondary slurry feeder at 10N downforce and in-situ conditioning CMP experiments were conducted. The tests resulted in 880 ± 110 Å/min MRR and 0.8 ± 0.4 nm surface roughness value.

Conclusions

In this study we introduced a systematic optimization of CMP process for GaN as a function of the crystallographic orientation by considering both the chemical and the mechanical factors affecting the process performance. The N-face GaN was studied more in detail due to its limited removal rate response. Beyond determining the optimal slurry flow rate, solids loading and applied downforce, chemical promotion of GaN removal by in-situ neutralization of the ammonia generated as a reaction product and adjusting slurry temperature were analyzed. Consequently, a new CMP process set-up as well as a tool design was suggested which are synergistically promoting the material removal and controlling the surface quality while conserving slurry for bulk removal of GaN.

In conclusion, the new process and tool configuration can be implemented for GaN CMP based on the desired outcome of the particular process set-up and the process integration requirements. The basic optimization approaches detailed in this study can be further investigated for high volume manufacturing with a significant advantage in enhancing the material removal rates of GaN. This study also sheds light into why there is a inconsistency in the literature findings on the CMP performance of the GaN. As detailed throughout the paper, GaN surface crystallographic orientation as well as the surface nature (in terms of surface free energy, wettability and charge), slurry and operational variables tend to play a critical role on CMP performance. Therefore, unless all these factors are kept equivalent, it is not justifiable to compare the different CMP process results.

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