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Wet-based Digital Etching on GaN and AlGaN

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Many advanced III-Nitride devices, such as micro-LEDs, vertical FinFETs and field emitters, require the fabrication of high aspect ratio vertical pillars or nanowires. Two-step etchings combining dry and wet etching steps have been used on vertical devices in the past, but they show poor control in vertical nanostructures with sub-50 nm diameter. In this work, we demonstrate a wet-chemical digital etching on GaN and AlGaN, and apply it to both vertical nanostructure scaling and planar etching along the c-axis. In this digital etching process, a mixture of H_2SO_4 and H_2O_2 is applied to oxidize the III-Nitrides surface, and the oxide layer is then removed by dilute HCl. This digital etching approach can finely sharpen vertical structures and does not require any vacuum or plasma systems, which will enable advanced device structures in the future.

Thanks to their unique material properties, III-Nitride semiconductors are used in applications such as power and high-frequency electronics and solid state lighting. III-Nitride micro-LEDs, vertical FinFETs, field emission devices, and many other devices have dimensions below 100 nm. Unfortunately, the lack of nm-resolution etching technologies for vertical structures, especially for shrinking features with sub-50 nm dimensions, is currently hindering the development of these devices. The orientation-dependent etch of heated tetramethylammonium hydroxide (TMAH)[1,2] and KOH[3] treatments has been used to remove dry-etching-induced damage in III-Nitride materials in the past and to shrink vertical structures. Unfortunately, it is difficult to apply these treatments on vertical nanostructures with sub-50 nm diameter due to microfaceting during the chemical etch. Therefore, etching technologies to finely control and shrink vertical nanostructures following a conventional etching process are still necessary.

Among different vertical nanostructure devices, self-aligned-gate field emitter arrays (SAGFEAs) require especially stringent etching conditions as they need vertical tips with nanometer-level sharpness (i.e., sub-20 nm tip diameter) to reduce operating voltages. This is hard to achieve with a conventional dry etching technology alone. Recently, sharp vertical tapered GaN tips with sub-20 nm tip size and smooth sidewalls, whose slopes are about 78 degree, were demonstrated with a two-step etching process combining both dry etching and hot H_3PO_4 etching[4]. However, the control of this hot H_3PO_4 etching is an issue when uniform nanostructure arrays are necessary. Other technologies, such as digital etching (DE) and atomic layer etching (ALE)[5–11] have, however, mainly focused on the gate recess along the c-axis for HEMT devices and very limited information exists about the etching along other directions for vertical structure sharpening. One common DE approach on III-Nitrides is to first oxidize the surface with an O_2 or N_2O plasma, followed by a removal of the surface oxide layer by dilute HCl[5,6]. DE based on O_2 and BCl_3 plasma has been studied recently,[10,11], but only its etching properties on the c-plane have been studied. Thermal oxidization, followed by KOH wet etching on c-plane AlGaN, has also been investigated[12,13]. Very limited information exists regarding vertical structure scaling based on this method and additional surface roughness could be introduced during thermal oxidization on III-Nitrides[14,15]. Other DE or ALE technologies also feature the use of an anisotropic plasma[7,8], hence, those approaches are not applicable for sharpening vertical structures. A thermal ALE of GaN (0001) surface using XeF_2 and BCl_3 has also been reported[9]. Its plasma-free operation is promising for the vertical structure scaling; however, it requires a custom setup not easily available in most laboratories. To solve these challenges, we recently reported a wet-chemical DE for GaN tip sharpening and we applied it to the fabrication of state-of-the-art SAGFEAs[16].

In this work, we further investigate the wet-chemical DE process for sharpening GaN vertical tips[16] and expand its use to both AlGaN tips and c-plane AlGaN etching. This DE technology can finely shrink the GaN vertical tapered tips without degrading sidewalls, which is beneficial in some applications with dense vertical nanostructure arrays, such as micro-LEDs and vertical power devices. In addition, its plasma-free controllable etch rate on c-plane AlGaN can find numerous applications in HEMT fabrication, including gate and ohmic recess formation.

This study evaluated this wet-chemical DE in four different epitaxial structures (Fig. 1). Structure A is an n^+ GaN layer grown by metalorganic chemical vapor deposition (MOCVD) on Si (111) substrate by Enkris, Inc. Structure B is a conventional AlGaN/GaN HEMT structure on Si grown by MIT Lincoln Lab. Structures C and D are AlGaN layers grown by molecular beam epitaxy (MBE) on AlN and GaN templates, respectively. There are two different Al composition ratios in each of structure C and structure D, as listed in Fig. 1. Structures A and C are used for vertical tip sharpening experiments, and structures B and D are used for c-axis AlGaN etching experiments. All samples have metal-polar (0001) surfaces. The wet digital etching (DE) technology studied in this work consists of four steps per cycle, as listed in Fig. 1. The GaN or AlGaN surface is first oxidized by a mixture of H_2SO_4 and 30% H_2O_2 and then rinsed in DI water. The oxidized layer is then removed by dilute HCl and another DI water rinse.

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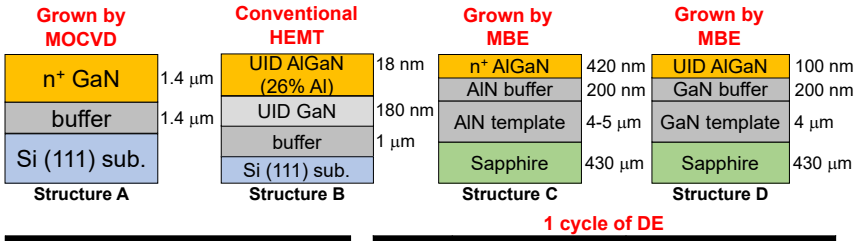

	Al ratio in AlGaN	Sample ID
Structure C	37.6% Al	C1
	65.7% Al	C2
Structure D	30.4% Al	D1
	84.5% Al	D2

1 cycle of DE

Step no.	Solvent	Time
1	1 H_2SO_4 + 1 30% H_2O_2	4 min
2	DI water	30 sec, then DI water rinse
3	1 HCl + 3 H_2O	2 min
4	DI water	30 sec, then DI water rinse

FIG. 1. Epitaxial structures of different films. All samples have metal-polar surfaces. The n^+ GaN and n^+ AlGaN layers of structures A and C are all doped with [Si] $> 10^{19} cm^{-3}$ since these samples are used for SAGFEA devices[16]. The bottom right table shows the different steps in one cycle of DE.

It is important to note that, since the H_2O_2 decomposes over time, the mixture of H_2SO_4 and H_2O_2 is renewed after every three cycles of DE in order to maintain a stable etching rate. Therefore, the fitted etching rates in the following experimental results are all based on the etch depth after 3 full cycles of DE.

The DE was first used to sharpen vertical GaN and AlGaN nanopyramids, formed by plasma dry etching, in order to obtain III-Nitride vertical tips with sub-10 nm tip radius. The process flow is shown in Fig. 2(a). Vertical tips of GaN (A) and AlGaN (C1 and C2) with tip width varying from 30 nm to 70 nm are first formed by Cl_2/BCl_3-based inductively coupled plasma-reactive ion etching (ICP-RIE) with 30-40-nm-thick Ni as hard mask. These tips are defined in arrays with a pitch of 600 nm. The height of the tips is about 400 nm for the GaN tips, and 300 nm for the AlGaN ones. The tip width was checked by scanning electron microscope (SEM) after dry etching (not shown here) and after the Ni hard mask was removed by 10 min piranha etch (Fig. 3(a)). No clear change on the morphology of the GaN vertical tips sidewalls was observed after piranha etch, and the estimated sidewall slope remains about 72 - 75 degree, which is very close to the value reported by the H_3PO_4 work[4]. It should be noted that this slope is based on 60-degree-tilted SEM images rather than from direct cross-sectional images, so a minor error could exist in this estimation. On the other hand, the $Al_{0.66}Ga_{0.34}N$ tips seem to be etched by piranha and the sidewalls become rougher, which indicates that alternative Ni mask etchants should be investigated in the future. After the Ni mask removal, the tips are then sharpened by a few cycles of DE and checked by SEM again (Fig. 3(b), (c), and (d)).

The tip width before and after DE was measured by SEM and the results are summarized in Fig. 2(b). The tip shrinking due to DE is very stable for n^+ GaN tips. It is more difficult to estimate the tip width for n^+ AlGaN tips because of SEM charging effects once the size is below 20 nm. The sharpest

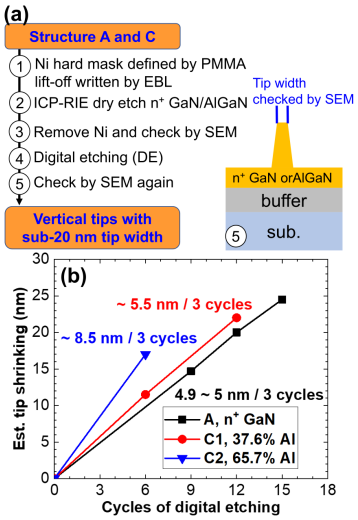

FIG. 2. (a) Process flow used in the vertical tip shrinking experiments (structure A and C) and (b) estimated tip shrinking through DE on n^+ GaN and n^+ AlGaN assessed through SEM measurements. We only do 12 cycles of DE for sample C1 and 6 cycles for sample C2 since their tip diameters are below 20 nm after those etching conditions and it becomes challenging to estimate the sizes of sub-20 nm tips in the SEM.

GaN and AlGaN tips are estimated by SEM to be sub-20 nm width, that is sub-10 nm tip radius. High-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) would be necessary in the future to fully confirm the

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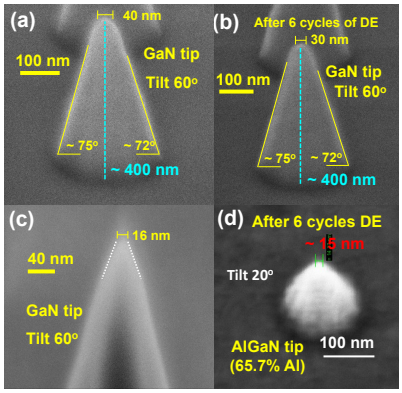

FIG. 3. Tilted SEM images of GaN tips (a) after Ni mask removal (i.e., no DE cycles) and (b) after 6 cycles of DE. (c) GaN and (d) AlGaN tips with estimated sub-20 nm tip width (sub-10 nm tip radius) after multiple cycles of DE. The GaN tip in (c) is different from the ones in (a) and (b).

tip radius[4]. The shrinking rates on AlGaN (C1 and C2) are higher than GaN (A), and the sidewall surface of the AlGaN tips becomes rougher after piranha clean and after DE (Fig. 3(d)). The mixture of H_2SO_4 and H_2O_2 seem to not only oxidize the AlGaN sidewalls formed by dry etching but also to directly etch them. However, at this point, it is not clear whether this etching is the result from the plasma damage or from the different exposed lattice orientations. Although, it is expected that the shrinking of the III-N structures during this DE should remove the plasma damage induced during the initial tip formation, in a way similar to TMAH and KOH treatments[1–3], photoluminescence (PL) measurements are needed in the future to assess the extent of sidewall damage[3,17].

The DE technology was also tested in the planar etch of c-axis AlGaN. A controllable and low-damage etching is critical for fabricating normally-off HEMTs. Our wet-based DE could be beneficial for these devices since there will be no plasma-induced defects.

The process flow followed for the characterization of the etch rate along the c-direction is shown in Fig. 4(a). After the samples are cleaned by a 10 min Piranha and DI water rinse, a 100-nm-thick SiO_2 hard mask is deposited by plasma enhanced chemical vapor deposition (PECVD). After that, this SiO_2 is patterned by buffer oxide etch (BOE) to expose the AlGaN surfaces. The thickness of SiO_2 layer and surface roughness of AlGaN layers are then measured by atomic force microscope (AFM) (Fig. 4(b) and (c)). After that, the samples are etched by a few cycles of the wet-based DE. The depth of patterned SiO_2 and etched AlGaN, and the surface roughness are then checked again by AFM. All AFM measurements were performed on similar regions for each sample to prevent additional variations across the sample (about 1 cm × 1 cm size). Results are summarized in Fig. 5. The etching rate of UID AlGaN along the c-axis is slower on AlGaN with

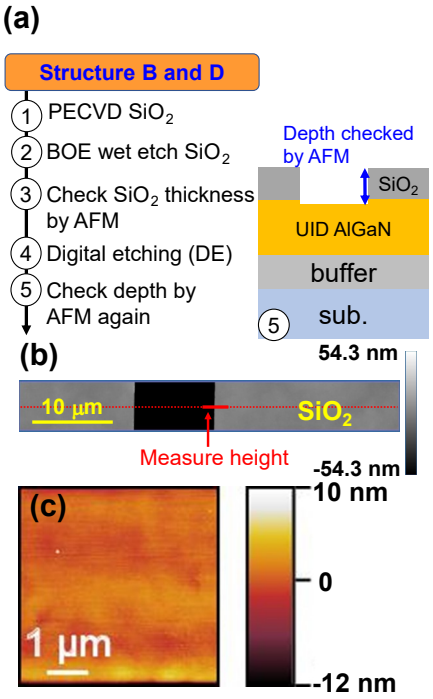

FIG. 4. (a) Process flow for digital etching along c-axis of UID AlGaN (structure B and D), and (b) depth and (c) surface roughness measured by AFM. The surface roughness is measured across 5 $\mu m \times 5\ \mu m$.

high Al ratio (sample D2). Besides the different etching rates, surface roughness does not degrade after at least 18 cycles of digital etching on all 3 samples, making this DE a good technology candidate for gate recess process in normally-off HEMTs. It should be noted that crack-type defects exist in structure D2 prior to etching due to the large lattice mismatch between AlGaN (84.5% Al) layer and the GaN buffer. After multiple cycles of DE, some areas of this high-Al AlGaN film are peeled off. One possible explanation of this phenomenon is that crack defects provide an additional path for chemicals to reach the AlGaN/GaN interface and etch undercut the AlGaN layer. During AFM measurement, those defect areas are avoided. This phenomenon is not observed in samples B and D1.

Experimental results demonstrate that the proposed wet-based DE is applicable for both vertical structure scaling and lateral device fabrication (Fig. 2 and Fig. 5). The etching rates are stable and reproducible, and the AlGaN (0001) surface roughness does not degrade after at least 18 cycles of DE. However, the DE behaves differently in planar and vertical etching. In tip shrinking experiments, the AlGaN tips

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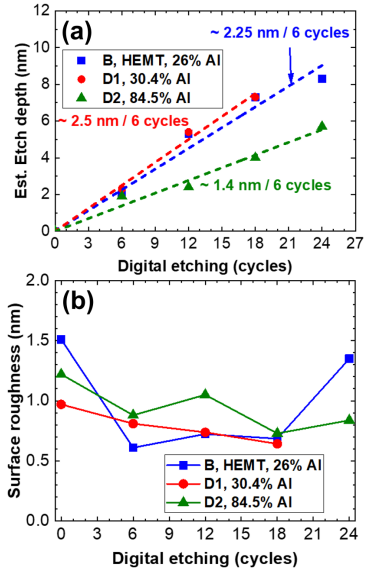

FIG. 5. (a) Estimated etching depth of UID AlGaN along c-axis and (b) surface roughness after different number of cycles of DE. Surface roughness is measured across 5 $\mu m \times 5\ \mu m$.

with high Al ratio are shrunk more quickly than GaN tips (Fig. 2(b)), while the etching rate of high-Al-ratio AlGaN along the c-axis is slower than the one of low-Al-ratio AlGaN (Fig. 5(a)). Though the reason for this deviation is not clear yet and requires future study, there are at least two possible explanations:

First, the dry etch used to define vertical structures may induce more plasma damage in high-Al AlGaN sidewalls than in GaN. The piranha treatment could then etch the plasma-damaged AlGaN surface much faster than GaN. Prior work[7] has shown that KOH etches Ar-plasma damaged GaN regions more easily than pristine materials, though the similar study on AlGaN is limited. DE experiments on AlGaN vertical structures formed by wet etching[18] can help rule out the effects of plasma damage in the future.

Second, the etching rates are expected to vary with the exposed lattice planes. For example, the reversed polarization between Ga-polar and N-polar surfaces is responsible for selective etch by KOH of N-polar GaN since the spontaneous polarizaiton in Ga-polar surface prevents the OH^- ions from reacting with Ga atoms to form gallium oxide[19]. Similar mechanism can explain that the semi-polar surface is more reactive to chemicals than the (0001) c-plane because of the weaker spontaneous polarization[20]. Therefore, the Al_2O_3 could be formed on the semi-polar high-Al AlGaN sidewalls and be etched by the mixture of H_2SO_4 and H_2O_2[21], while the strong polarization of the c-plane (0001) AlGaN surface limits the oxidization and thus the etching rate. The increasing roughness on the sidewalls of AlGaN vertical tips could indicate microfaceting due to orientation-dependent etching rates[4,22,23] or the probable segregation in AlGaN alloys[24,25]. DE experiments on semi-polar or non-polar AlGaN materials in the future would allow to further investigate the hypothesis of lattice-plane-dependent etching rates.

In summary, a wet-chemical digital etching is demonstrated on GaN and AlGaN materials. Vertical GaN and AlGaN nanopyramids, formed by plasma dry etching, can be sharpened by this technology. While the deviation of etching rates with Al composition requires future investigation, the proposed wet DE technology demonstrates a simple approach to do a nm-level and well-controlled etching without any vacuum or plasma system. III-Nitride vertical nanostructures can be shrunk by this technology and used to improve the performance of vertical devices, including III-Nitride vertical SAGFEAs[16]. The surface of (0001) AlGaN after multiple cycles of DE does not degrade (Fig. 5(b)), which also suggests that this technology could be beneficial for plasma-free gate recess or relevant process when fabricating III-Nitride lateral devices. The use of this type of digital etching is expected to render III-Nitrides devices with less etching damage than in plasma-based etching technologies.

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The data that support the findings of this study are available from the corresponding author upon reasonable request.

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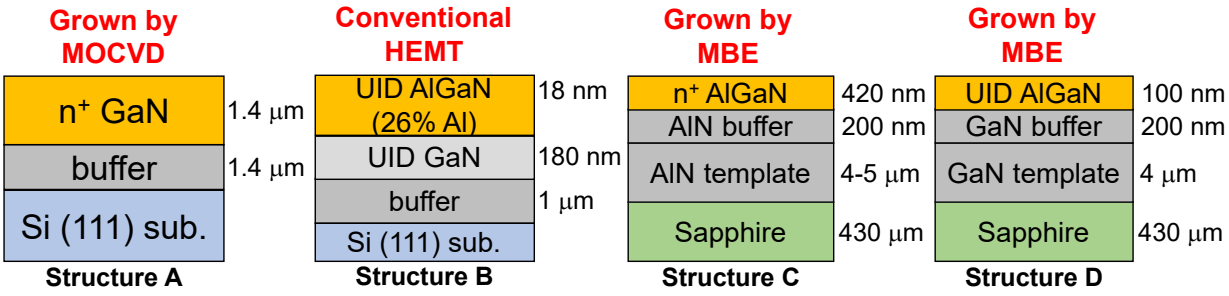

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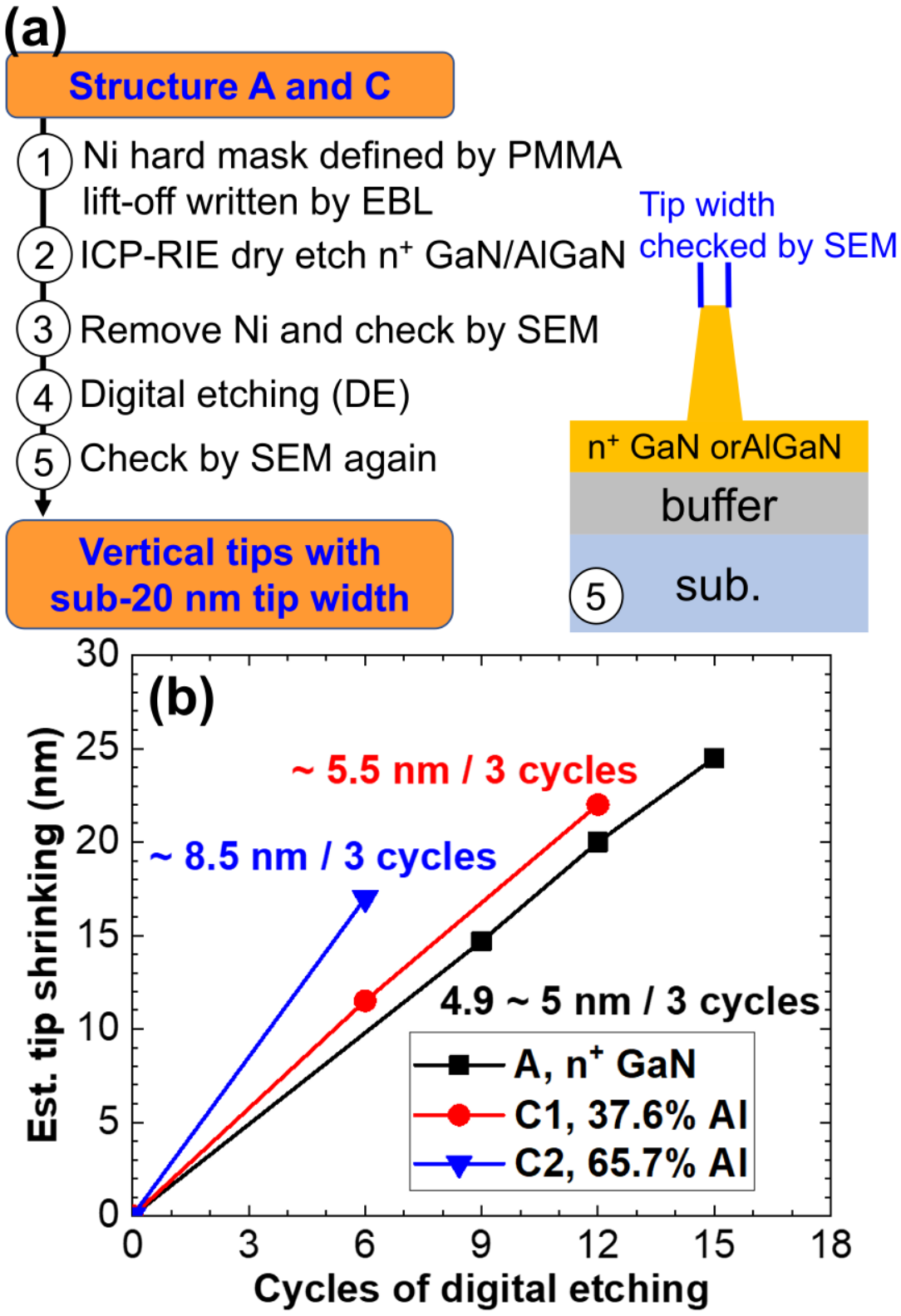

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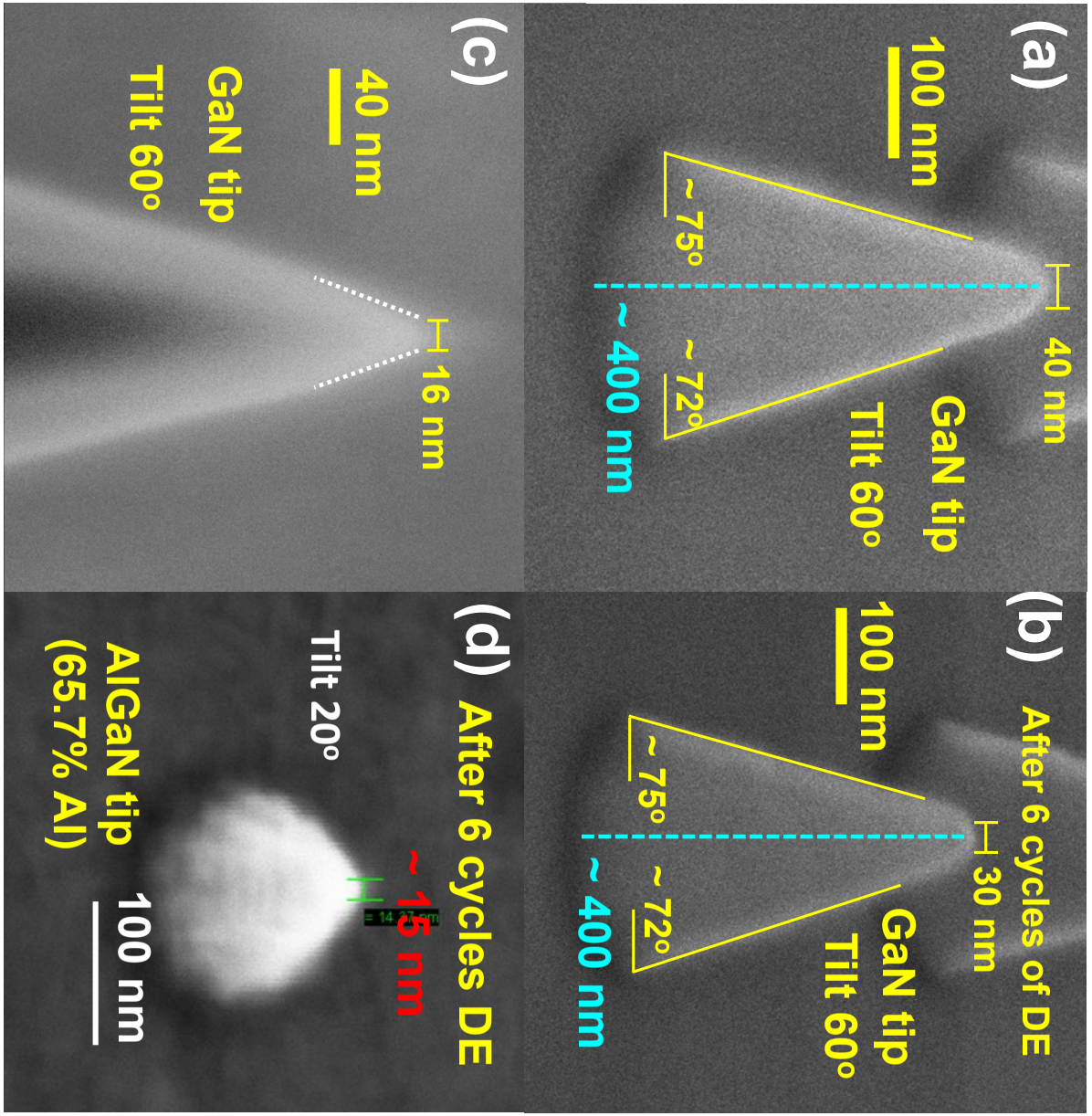

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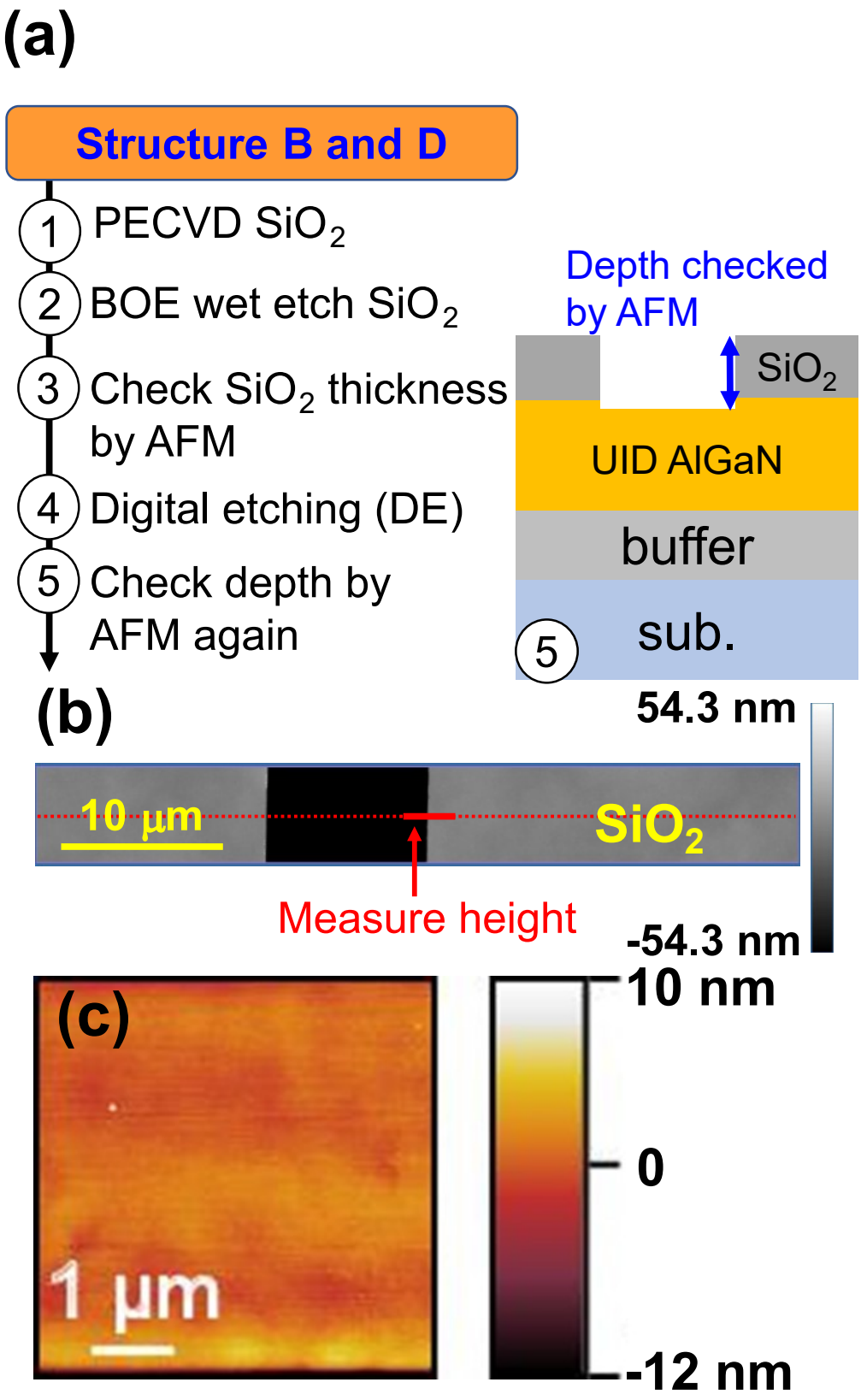

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