Chemical mechanical polishing of freestanding GaN substrates*

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Abstract: Chemical mechanical polishing (CMP) has been used to produce smooth and scratch-free surfaces for GaN. In the aqueous solution of KOH, GaN is subjected to etching. At the same time, all surface irregularities, including etch pyramids, roughness after mechanical polishing and so on will be removed by a polishing pad. The experiments had been performed under the condition of different abrasive particle sizes of the polishing pad. Also the polishing results for different polishing times are analyzed, and chemical mechanical polishing resulted in an average root mean square (RMS) surface roughness of 0.565 nm, as measured by atomic force microscopy.

Key words: GaN; chemical mechanical polishing; epitaxial layers
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1. Introduction

As demands increase for highly efficient short wavelength emitters and high power transistor devices, devices using native and bulk GaN substrates have attracted widespread interest. These devices take advantage of the lattice and thermal expansion matching between the substrate and the epitaxial layer, resulting in reduced defect density in the bulk substrate.

Thick freestanding GaN films have been obtained using hydride vapor phase epitaxy (HVPE) and laser-lift off technique, and then processed into substrates for device applications. Mostly, chemical mechanical polishing (CMP) is used to polish sapphire or SiC substrates[1,2]. However, there are very few reports on the CMP of freestanding GaN substrates. CMP can be used to produce surfaces prepared for epitaxial growth[3], and it is an important link in the entire process of preparing high quality GaN substrates. CMP uses a combination of chemical and mechanical reactions to remove the surface structures, leaving a plane and damage free surface.

GaN can be etched in aqueous solution of NaOH or KOH at room temperature[4], so either NaOH or KOH can be used to control PH of the solution. As a result of alkaline etching, numerous pyramids will be formed at the surface of GaN. The surface reaction is kinetically controlled and will persist until GaN surface is covered by the pyramids.

Polishing with a soft diamond polishing pad, which is harder than GaN, under 2–6 kg/cm^2 applied pressure, all surface irregularities are removed[5], including etch pyramids, roughness after mechanical polishing, and growth hillocks on epitaxial layers.

After the CMP process, atomic force microscope (AFM) is used to measure the surface. When optimal CMP is used, atomically flat surface of bulk GaN have been obtained, and its root mean square roughness (RMS) is 0.5 nm.

CMP is a complicated process[6], incorporated both the chemical and mechanical effects[7], and its removal rate is controlled by the slower one. The result of CMP depends on the condition of PH of the solution, abrasive concentration of polishing pad, polishing pressure and slurry flow[8]. In this work, the influences of abrasive concentration of polishing pad and different polishing times are analyzed, in order to point out the mechanism of CMP effect on GaN morphology.

2. Experiment

The GaN bulk crystals were grown using an HVPE process. The thick GaN layers were grown and removed from the underlying sapphire to create freestanding and bulk material. The thickness of the layer is of the order of 900 μm. Before the CMP process, the samples were mechanically polished using a diamond-based platform, in order to remove the thickness difference of samples caused by the HVPE high epitaxial growth rate. In this work, the GaN samples are c-plane substrates.

It is reported that N-face GaN samples exhibit more effective polishing than Ga-face GaN samples[9]. In this work, Ga-face GaN samples were used in the CMP process. KOH solution was used to planarize the Ga-face substrate. The solution was delivered onto the rotating polishing table and the table was covered with a soft embedded diamond polishing pad.

Polishing pads with different abrasive particle sizes were

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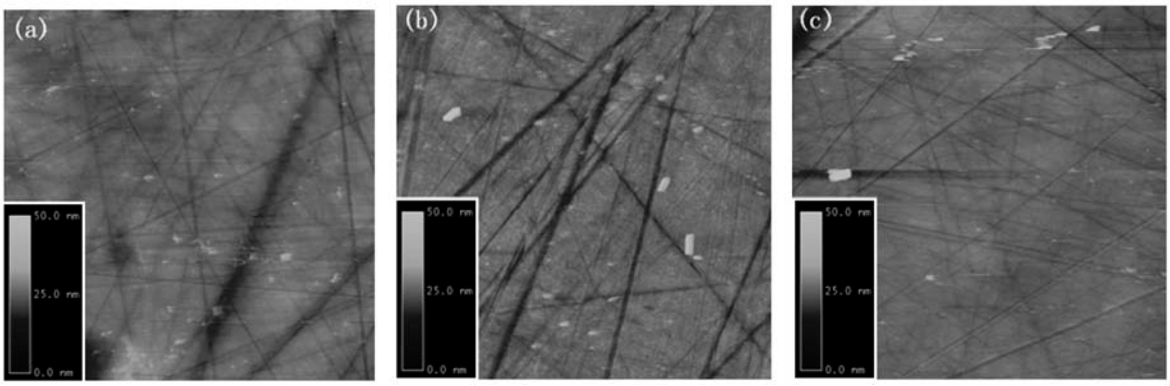

Fig.1. Surface morphology of the substrates after polishing with different abrasive particle sizes: (a) 1 μm; (b) 0.5 μm; (c) 0.1 μm.

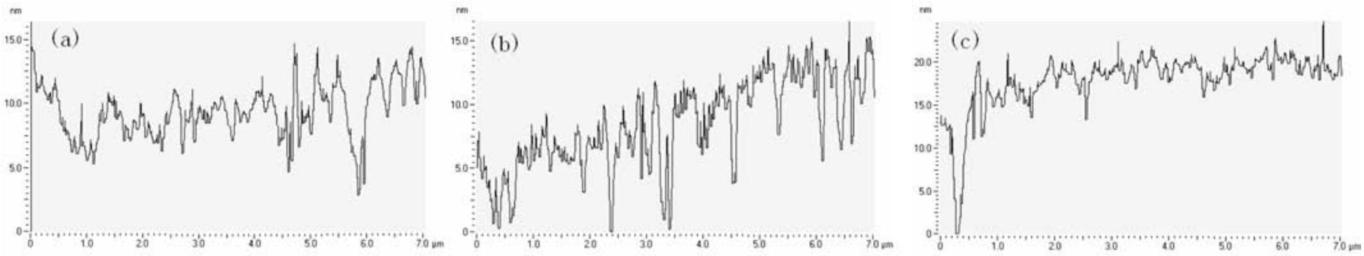

Fig.2. Cross section of Fig.1: (a) Abrasive particle sizes of 1 μm; (b) Abrasive particle sizes of 0.5 μm; (c) Abrasive particle sizes of 0.1 μm.

used in the experiment, including 0.1, 0.5, and 1 μm. After the CMP, AFM (Nanoscope III) was used to characterize the surface morphology. The influence mechanism of abrasive particle sizes of polishing pad and different polishing times is analyzed.

3. Results and discussion

3.1. Influence of different abrasive particle sizes

Three GaN samples were polished using polishing pad of different abrasive particle sizes, which are 0.1, 0.5, and 1 μm, respectively. The PH of KOH solution is 13, and the polishing times are all 1 h.

Figures 1 (a), 1 (b), 1 (c) are the AFM images of three GaN samples which were polished by polishing pad with different abrasive particle sizes after 1 h. The area of each image is $5 \times 5\ \mu m^2$.

AFM scans on the surface of the samples after CMP using different polishing pads reveal the changes in the surface morphology and roughness. The surfaces root mean square (RMS) roughness of Figs.1 (a), 1 (b), 1 (c) are 2.523, 2.722, 2.554 nm, respectively. Also Z range of Fig.1 was calculated. Z range is the maximum vertical distance between the highest and lowest data points in the image, which means the thickness difference of the sample surface. Z ranges of Figs.1 (a), 1 (b), 1 (c) are respectively 42.554, 48.595, 100.38 nm. So the RMS roughness of the surface reveals no significant difference under the given CMP condition. But Z range changed a lot after CMP process using polishing pad of different abrasive particle sizes. This means that using polishing pad of abrasive particle size of 1 μm has a highest remove rate, and so the thickness difference of the surface can be quickly removed.

In order to show the thickness and scratch change on the samples surface after polishing, a cross section of Fig.1 is shown in Fig.2. In Figs.2 (a), 2 (b), and 2 (c), the mean depths of scratches are respectively 8.5, 7, and 5 nm. This means that deeper scratches are induced as a result of using polishing pads with large particle sizes.

In order to get smooth and damage-free freestanding GaN substrates, a process of using polishing pads of different abrasive particle sizes should be taken into consideration. Polishing pad of large abrasive particle sizes has a higher thickness remove rate, and it could be used to remove the surface uneven thickness difference quickly at first. After that process, polishing pad of small abrasive particle sizes will induce smaller scratches, and it could be used to remove local roughness difference.

3.2. Surface morphology after different polishing times

Under the condition of the PH of KOH solution is 10, the rate of solution flow is about 90 mL/min, and the applied pressure on the substrate surface is 2 kg/cm^2, we polished the GaN substrate and analyzed its surface morphology after different polishing times using AFM. Before this experiment was done, the GaN substrate was mechanically polished using polishing pad of a large abrasive particle size as an original planarization step.

Figure 3 is the AFM surface morphology of GaN substrate after CMP for 0.5 h, and the area of the image is $5 \times 5\ \mu m^2$. Z range of the GaN substrate is 42 nm and the RMS roughness of the surface is 5.396 nm. As shown in Fig.3, after CMP for 0.5 h, numerous pyramids are formed on the surface of *c*-plane GaN, and these pyramids are randomly distributed over the surface.

Weyher *et al.*[5] reported that, after a short time etching in alkaline solution (about 10 min), numerous pyramids with a

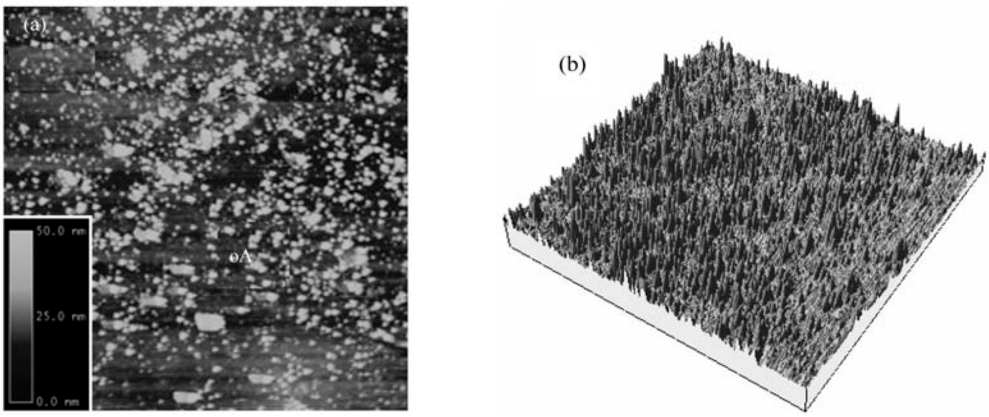

Fig.3. Surface morphology of GaN substrate after CMP for 0.5 h: (a) 1D AFM image; (b) 3D AFM image.

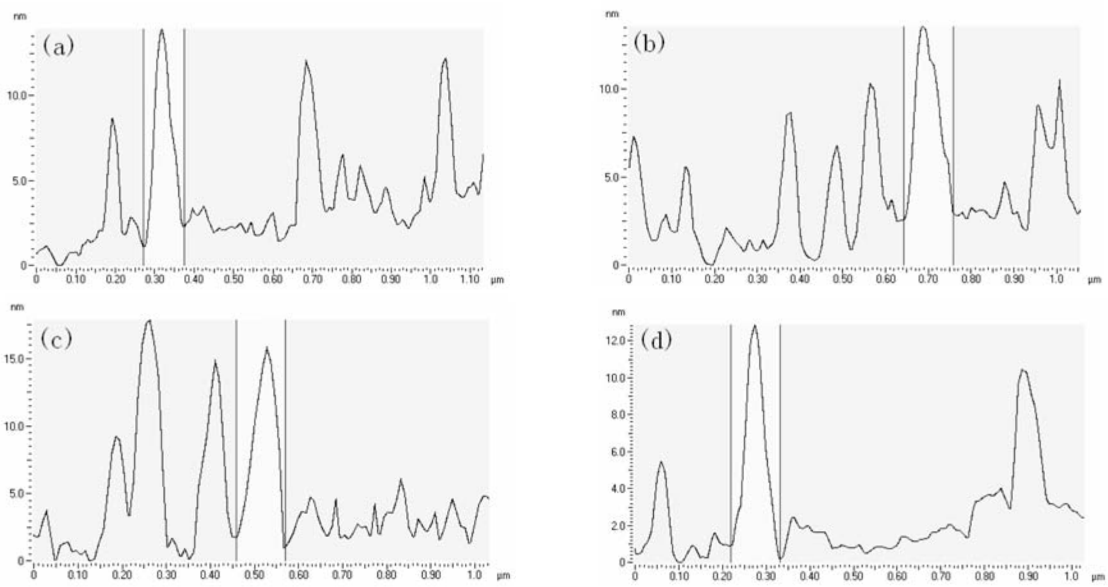

Fig.4. Four different cross sections across point A.

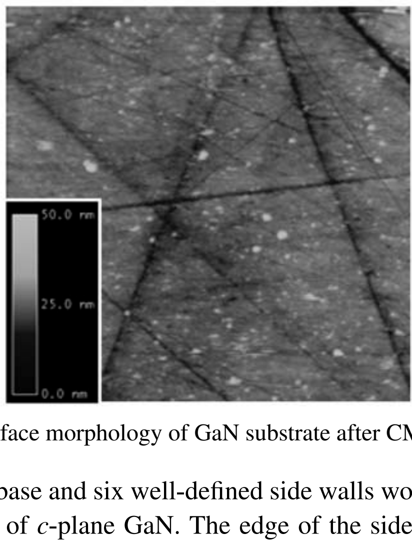

Fig.5. Surface morphology of GaN substrate after CMP for 3 h.

hexagonal base and six well-defined side walls would form on the surface of *c*-plane GaN. The edge of the side walls were parallel to the $[10\bar{1}0]$, and the inclination of the side walls of pyramids is about 60° to the (0001) plane. From Fig.3, it is shown that the base of the pyramids became conical. In order to attest the conclusion, across point A marked in Fig.3 (a), four cross sections of the surface along different orientations were made in Fig.4.

In Fig.4, the width of the base of the pyramid in point A was marked. It can be measured that all the widths in Figs.4 (a), 4 (b), 4 (c), and 4 (d) are about 0.11 μm. It means that the width of the base of the pyramid in point A is the same along four different orientations. The result attest that the base of the pyramids became conical after a longer etching time. It indicates that some dissolution occurs slowly at the edge of the pyramids.

The rate of solution flow should be well controlled. The solution flow could wash away the remains of polished GaN material from polishing pad, and also affect the rate of chemical reaction on the surface of the substrates at the same time. Compared with the rate of the chemical reaction, the rate of removal of reaction products must be higher than it, so that the mechanical polishing could remove the pyramids caused by chemical etching in time. In this work, the rate of the alkaline solution flow was controlled above 90 mL/min, and an optimal polishing result was obtained.

Figures 5 and 6 show the AFM images of the GaN surface morphology after CMP for 3 and 4 h, and the area of the image is 5 × 5 μm^2, 4 × 4 μm^2, respectively. There are still some etching pyramids in Fig.5 after CMP for 3 h. The RMS roughness is 1.704 nm, and Z range thickness is 34.234 nm.

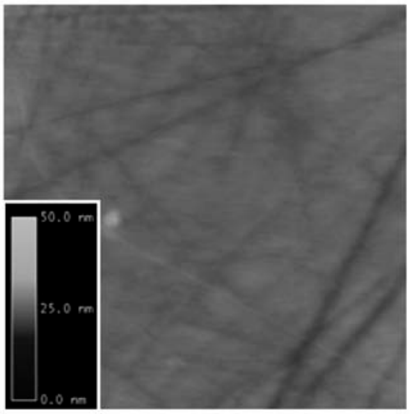

Fig.6. Surface morphology of GaN substrate after CMP for 4 h.

In this case, the damages from polishing process revealed in the image are mainly the scratches that remained after the final mechanical polishing using polishing pad of large abrasive particle sizes and the pyramids on the surface caused by the chemical etch. Figure 6 is the AFM image after CMP for 4 h. The RMS roughness is 0.565 nm, and Z range thickness is 0.4 nm. The figures show that the CMP process was effective at removing all the surface irregularities. A smooth GaN substrate was obtained.

4. Conclusion

In this work, the CMP method of freestanding GaN substrates was studied. CMP was successfully employed to prepare GaN surfaces for epitaxial growth. The influence mechanism of different abrasive particle sizes of polishing pad and different polishing times was analyzed. In order to obtain a quick and good CMP result, polishing pad of large abrasive particle sizes could be used at first to remove the thickness difference quickly. And then small abrasive particle sizes polishing pad could be used to remove local roughness difference on the surface. For the GaN surfaces, CMP resulted in an average RMS surface roughness of 0.565 nm, and it satisfied the need for further epitaxial growth.

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