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Investigation on the surface characterization of Ga-faced GaN after chemical-mechanical polishing

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ABSTRACT

The relationship between the surface characterization after chemical mechanical polishing (CMP) and the size of the silica (SiO_2) abrasive used for CMP of gallium nitride (GaN) substrates was investigated in detail. Atomic force microscope was used for measuring the surface morphology, pit feature, pit depth distribution, and atomic step-terrace structure. With the decrease of SiO_2 abrasive size, the pit depth reduced and the atomic step-terrace structure became more whole with smaller damage area, resulting in smaller roughness. For tiny-sized SiO_2 abrasive, an almost complete atomic step-terrace structure with 0.0523 nm roughness was achieved. On the other hand, in order to acquire higher removal, Pt/C nanoparticle was employed as a catalyst in CMP slurry. The result indicates that when Pt/C catalyst content was reached to 1.0 ppm, material removal rate was increased by 47.69% compared to that by none of the catalyst, and besides, the pit depth reduced and the surface atomic step-terrace structure was not destroyed. The Pt/C nanoparticle is proved to be the promising catalyst to the surface preparation of super-hard and inert materials with high efficiency and good surface.

1. Introduction

Gallium nitride (GaN) and related III–V nitride alloys have demonstrated potential for optoelectronic devices such as blue and ultraviolet light emitting diodes and laser diodes. GaN-based devices have been fabricated from epitaxial films grown on substrates such as sapphire [1–4] and silicon carbide (SiC) [5–8], which are the commonly used substrates in recent years. However, the large lattice and thermal mismatches between epitaxial GaN and the above substrates result in a high dislocation density in the heteroepitaxial layer. Recently, GaN substrate for homoepitaxy has caused the researchers concern owing to its better crystal lattice matching with epitaxial GaN [9], high thermal conductivity, low coefficient of thermal expansion and high temperature stability. GaN layers formed on GaN substrate exhibit electronic properties superior to those formed on foreign substrate.

To use GaN material as a substrate for epitaxy, an atomically smooth and damage-free surface of GaN is desired intensely. However, GaN is categorized as one kind of hard-to-process materials due to its extreme hardness and strong stability against chemicals. Chemical mechanical polishing (CMP) is the effective technology to provide global planarization though mechanical abrading and chemical reaction. S. Hayashi et al. [10] attained material removal rate (MRR) of ∼50 nm/min using slurry including alumina abrasives, and surface roughness (Ra) was achieved to 0.5–0.6 nm using sodium-hypochlorite-based slurry without abrasives. Yan Huaiyue et al. [11] investigated CMP of freestanding GaN substrates with different abrasive particle size, in which, the average root mean square surface roughness was finally achieved to be 0.565 nm. Hideo Aida et al. [12] obtained a low MRR of 17 nm/h and realized an atomically flat surface with Ra = 0.1 nm after 150 h CMP of GaN using slurry with colloidal silica abrasive. Hideo Aida et al. [13] also proved that CMP of GaN is shown to obey Prestonian behavior, and presented that the MRR of Ga_2O_3 is two orders of magnitude higher than that of GaN. Drew Hanser [14] reported surface preparation of substrates from bulk GaN crystals. The MRR and Ra were 500 nm/h and 0.18 nm, respectively, but the details of CMP slurry and process were not illustrated. For all the above investigations, atomic step-terrace structure was not appeared in the final polished surface of GaN. Fortunately, Junji Murata et al. [15] have reported that step-terrace structure of a GaN substrate can be achieved by their chemical planarizing, but their slurry contains no

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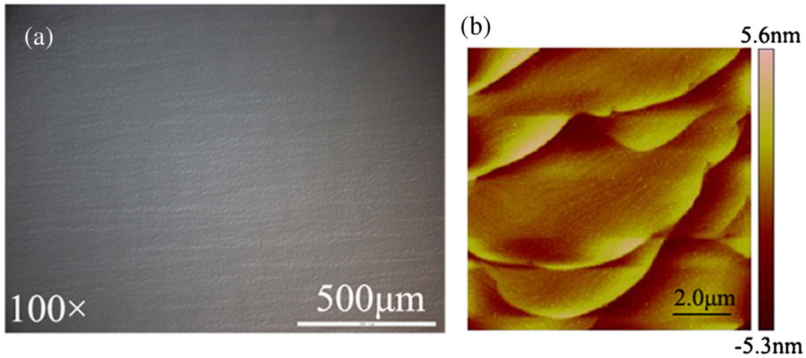

Fig. 1. Initial surface of Ga-face (0 0 0 1) GaN wafer. (a) Microscope image (b) AFM image.

abrasives. Xueping Xu et al. [16] have achieved a step structure on the surface of GaN after CMP process. However, the corresponding detailed information of CMP slurry and process were not presented, resulting in confusions for researchers.

In this paper, the relationship between the surface characterization after CMP and the size of the silica (SiO_2) abrasive used for CMP of GaN substrates was investigated in detail. The surface morphology, pit feature, pit depth distribution, and atomic step-terrace structure were measured by Atomic force microscope. As SiO_2 abrasive size reduced, the pit depth decreased and the atomic step-terrace structure became more whole with smaller Ra. For tiny-sized SiO_2 abrasive, an almost complete atomic step-terrace structure with 0.0523 nm roughness was achieved. Moreover, Pt/C nanoparticle catalyst was added into the CMP slurry for acquiring higher removal. The results indicated that material removal rate was increased and pit depth was reduced effectively compared to that by none of the catalyst, and besides, the atomic step-terrace structure was not destroyed.

2. Experimental

HVPE grown n-type epilayer GaN (50 μm thick) on 2 inch sapphire wafer (Suzhou Nanowin Science and Technology Co., Ltd) was used in this study. The dislocation density was less than 1×10^8 cm^{-2}. Ga-face (0 0 0 1) GaN wafers were processed through CETR CP-4 bench-top polishing tester. The experiments were performed for 2 h with SUBA 800 polishing pad under the conditions of the pressure 5.6 psi, slurry flow rate 70 mL/min, up plate rotating speed 120 rpm, and down plate rotating speed 160 rpm. After CMP, the wafers were cleaned by liquid cleaner and deionized water, and then dried off by air spray gun for measurements.

The slurries contain 30 wt% SiO_2 abrasives, 0–2 wt% H_2O_2, 1 wt% phosphoric acid (P), 0–3 ppm platinum–carbon (Pt/C) catalyst, and deionized water. Three kinds of SiO_2 abrasives were used, and their mean particle sizes were measured by Malvern Zetasizer Nano SZ Particle Size Analyzer as 110 nm, 40 nm, and 10 nm, respectively. The first two SiO_2 particles (110 nm and 40 nm) were the commercial silica sol, and the tiny-sized SiO_2 particle (10 nm) was prepared by ion exchange method, and the detailed description was shown in Ref. [17]. The Pt(40%)/C catalyst was Pt particles with the average size of 5 nm supported on plate shaped C particles with the average size of 80 nm.

Material removal rate (MRR) of GaN is calculated as below:

$$\mathrm{MRR} = \frac{\Delta m}{\rho_{\mathrm{GaN}} \pi r^2 t}$$

Δm is the removal weight of GaN wafer by professional electronic balance (OHAUS DISCOVERY), whose resolution is 0.00001 g, ρ_{GaN} is 6.1 g/cm^3, r is the radius of GaN wafer, t is polishing time. The MRR value used in this paper was the average of four individual polishing tests. The initial and processed surfaces were observed by Leica DM2500 optical microscope. The surface topography, surface roughness (Ra), and surface pit density were evaluated by atomic force microscopy (AFM, Bruker Dimension Icon). For AFM measurement, super-sharp probes with tip radius of only 2 nm were used for observation of high-definition atomic step-terrace structure of polished surface. For every polished sample, four different positions were measured, and the average value of Ra was used in this paper. The variation of coefficient of friction (COF) in the polishing process was investigated by CETR CP-4 polishing machine. All the experiments were carried out at room temperature.

3. Results and discussion

3.1. *The CMP performance on Ga-face (0 0 0 1) GaN wafer by using SiO_2 abrasives with different sizes*

Fig. 1 shows the rough initial surface of Ga-face (0 0 0 1) GaN wafer. From the scale bar in Fig. 1(b), we can also see that the distance between the lowest and highest points is ~11 nm, and the surface roughness (Ra) is 1.03 nm on 10 μm × 10 μm scan area. The obvious wavy surface of Ga-face GaN cannot meet the requirement for further homoepitaxy, it needs to be polished until the wafer surface is global flat and reach an atomic smooth level.

Firstly, Ga-face (0 0 0 1) GaN wafers were polished by CMP slurries with 110 nm and 40 nm SiO_2 abrasive, respectively. The corresponding material removal rates were calculated to be 52.17 nm/h and 23.70 nm/h, respectively. After 2 h of polishing, both the surfaces appeared to be flat and planar from optical microscope, and no defects could be seen. There was no difference between the two polished surfaces. In order to observe the surface morphology clearly, AFM images were measured, as shown in Fig. 2(a) and (b). The surface Ra values reduced to be 0.967 nm (110 nm SiO_2) and 0.321 nm (40 nm SiO_2) on 30 μm × 30 μm scan area. However, though the whole surfaces became flat, several pits were observed in AFM images. The average densities of these pits, calculated from five scan areas of 30 μm × 30 μm were ~0.813×10^8 cm^{-2} and ~0.887×10^8 cm^{-2} for 110 nm and 40 nm SiO_2, respectively. Specially, these two values were near the value of dislocation density of GaN sample used in this paper, which reveals that the pits may caused by dislocation created during crystal growth of GaN. Ref. [18] has demonstrated that AFM counting of the CMP polishing pits can be a convenient and accurate way to measure the dislocation density at the Ga-surface of GaN wafers.

Besides, Ref. [19] has indicated that pit formation was observed to be specific to silica particles and likely caused by interaction of silica particle with the GaN surface. Here, during CMP process, the GaN material was changed to Ga_2O_3 firstly due to the oxidizing agent of CMP slurry, and then the Ga_2O_3 was removed by SiO_2