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The effect of mixed abrasive slurry on CMP of 6H-SiC substrates

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Silicon Carbide (SiC) is a wide band gap semiconductor, being developed for high temperature, high power, and high frequency device applications. Many researchers have studied SiC polishing for the manufacturing of SiC and semiconductor substrates. However, these researchers have faced difficulties with a wafer preparation prior to epitaxial growth due to its hardness and remarkable chemical stability. A smooth and defect-free substrate surface is important to obtain good epitaxial layers. Therefore, a hybrid process, chemical mechanical polishing (CMP) has been proposed as a solution for preparing an epi-ready surface. During the experiments, the material removal rate (MRR) was investigated to learn how long the CMP process continues to remove a damaged layer by mechanical polishing using 100 nm diamond. Furthermore, the dependency of mechanical factors, such as pressure, velocity, and abrasive concentration, were researched using a single abrasive slurry (SAS). The experiments especially focused on the epi-ready surface with a mixed abrasive slurry (MAS). The addition of nanometre sized diamond in the MAS provided a strong synergy between mechanical and chemical effects. Through the experiments, a chemical effect (KOH based) was essential, and the atomic-bit mechanical removal was found to be efficient to remove residual scratches from on MAS. In conclusion, the SiC CMP mechanism was quite different from that of relatively softer materials to gain both high quality surfaces and a high MRR.

Key words: SiC, CMP, epi-ready surface, SAS, MAS

Introduction

Silicon carbides (SiC) are expected to be important for high power and high-temperature electric applications because of their wide energy band gaps, high breakdown voltage, high thermal conductivity, low thermal coefficient of expansion, and high temperature stability [1-6]. Since high level commercial devices using SiC substrates require a surface that is perfect without defects, the CMP process has been regarded as a significant step for the final wafering process. Several studies on the CMP process have already been made using colloidal silica with a variation of pH, chemicals, and abrasives [7, 8]. However, SiC produces many challenges for wafer preparation prior to epitaxial growth due to its remarkable hardness and chemical inertness. A smooth and defect-free substrate surface is important to obtain good epitaxial layers. Mechanical polishing of SiC is typically done through diamond based slurries, where the abrasive size is successively reduced and eventually ends up with a submicrometer slurry to achieve the desired smoothness. Relatively it is not difficult to achieve a surface with a low average roughness value. Although this surface fine polishing damage is detected through AFM (Atomic Force Microscopy). Moreover, a dense network of scratches and defects are revealed after high-temperature thermal

processing prior to, or during the epitaxial growth [9].

During these experiments, the material removal rate (MRR) was investigated to establish the dependency of mechanical factors using a single abrasive slurry (SAS), and in particular gaining an epi-ready surface with a mixed abrasive slurry (MAS).

50 mm diameter 6H-SiC single crystals were grown with a sublimation method [10-14] A 6H-SiC on-axis wafer, crystallized by physical vapor transport (PVT), was used in the experiments. A subsequent wafering process was continued to make SiC wafers. Fig. 1 shows an example of a conventional SiC wafering process used in the experiment.

Experimental

The reaction of wafer surfaces was examined by XPS (X-ray Photoelectron Spectroscopy). SiC CMP was applied using two types of SAS and MAS in order to investigate the relationship between the mechanical and chemical effects. A CMP equipment, POLI-400 (G&P Technology CO.) was used during the experiments. Images of SiC surfaces were measured by an optical microscope (Nikon Model 80i, Korea the scratches. The surface roughness (Ra) and scratch depth (Rv) were measured by an AFM (XE-150, Park System Co.) shown in Fig. 2 (b). Conditions used in these experiments are shown in the Table. 1 and Table. 2.

Generally, the MRR is well controlled under the Preston equation in the CMP process [15]. Using a commercial colloidal silica slurry and the Preston equation on the mechanical factors, the MRR was investigated. The results showed that the MRR increased linearly according to the

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Fig. 2. Measurement system of (a) optical microscope (Nikon Model 80i, Korea Instec Co.) and (b) AFM (XE-150, Park System Co.).

increment of pressure and velocity, which were well matched with the Preston equation.

Fig. 3 shows XPS spectra of SiC substrate submerged into the KOH based slurry for 24 hours. The result of the peak fitting performed on the Si 2p spectrum for the as-received SiC shows, a Si peak at 99.5 eV. The intensity of the Si peak dropped, and a peak of SiO₂ at 102.5 eV is observed after SiC was dipped into the KOH based slurry. In accordance with the XPS spectral results, the SiC surface is chemically reactive in the KOH based slurry. Thus, a standard slurry was used with a KOH based colloidal silica slurry.

Fig. 3. XPS spectra of : (a) Survey and (b) Si 2p from SiC surface of as-received and dipped in KOH based slurry.

Results and Discussions

First of all, the difference between the SAS with single abrasive and the MAS with colloidal silica mixed with nanodiamond abrasive was studied (Table 1). The roughness and scratches were evaluated using an optical microscope and an AFM after SiC CMP, as shown in Fig. 4 The average roughness value (Ra) before SiC CMP was 8 Å, and the greatest scratch depth (Rv) was 6 nm. The Ra and Rv, measured by the AFM, increased up to 10.5 Å and 12 nm, in the case of Table 3 (b) (SAS-I), respectively. However, the Ra of a defect-free region was down to 2.5 Å, because the shallow scratches, which are induced by the mechanical

Table 1. Experimental conditions (First)

Wafer	50 mm silicon carbide wafer (6H-SiC on-axis)					
Pad	Felt type pad (Suba 800)					
Velocity	Head 120 rpm / Platen 120 rpm					
Pressure	1.2 kg/cm^2					
Slurry	SAS-I: 20 wt% Colloidal silica (KOH base, 120 nm)	SAS-II: DIW + Nano-diamond (0.8 g/l)	MAS-I: 10 wt% Colloidal silica + Nano-diamond (0.8 g/l)	MAS-II: 20 wt% Colloidal silica + Nano-diamond (0.8 g/l)		
Flow rate	125 ml/min					
Process time	2 hours					

Table 2. Experimental conditions (Second)

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Wafer	50 mm silicon carbide wafer (6H-SiC on-axis)				
Pad	Felt type pad (Suba 800M2)				
Velocity	Head 100 rpm / Platen 100 rpm				
Pressure	1.2 kg/cm^2				
Slurry	SAS_no.: 20 wt% Colloidal silica (KOH base, 120 nm) : 5 run MAS_1: Colloidal silica + Nano-diamond (25 g/l) : 1 run	MAS_2: Colloidal silica + Nano-diamond (25 g/l)			
Flow rate	120 ml/min				
Process time	Each run: 1 hours	1 hours			



Fig. 4. Optical microscope and AFM images : (a) after MP, and after CMP using (b) SAS-I (20 wt% colloidal silica), (c) SAS-II (DIW + Nano-diamond (0.8 g/l)), (d) MAS-I (10 wt% colloidal silica + Nano-diamond (0.8 g/l)), and (e) MAS-II (20 % colloidal silica + Nano-diamond (0.8 g/l)).

 Table 3. Measurement results after CMP of each condition (First)

	MP(a)	SAS-I(b)	SAS-II(c)	MAS-I(d)	MAS-II(e)
MRR (mg)		0.6	0.1	0.6	0.5
Ra (Å)	8	10.5	24.8	15.5	2.4
Rv (nm)	6	12	23	12	1.2

polishing, are removed by the silica abrasive. The region around the deeper scratches reacted with the chemical solution; the width and depth of scratches were increased by the silica abrasive. The Ra and Rv of Table 3 (c) (SAS-II) were dramatically increased. The reason is that the specific slurry had a strong mechanical effect only on the material removal. Thus, the MRR was very low. As a result, the chemical effect was found to be essential to the SiC CMP. When the SiC wafer was polished by the mixed abrasive slurry, which is a high mechanical and chemical condition, the Ra and Rv values were 2.4 Å and 1.2 nm respectively in MAS-II. Also, Fig. 4(e) shows that the scratching was effectively reduced. The values show an outstanding result compared to the other experimental conditions. It is likely that the diamond abrasives easily remove the reacted layer around deeper scratches in the KOH based slurry, and the silica abrasive remove the shallow scratches. However, the AFM image shows that the surface was improved, but that the scratches were not completely removed. It is presumed that the small amount of the nano-diamond that was included, had an insignificant effect. The MRR was lower than that of SAS-I, because the pressure per unit particle was decreased as the abrasive concentration was increased. However, the MAS-I treatment could not improve the surface roughness due to its limited ability to remove shallow scratches according to a decrease of the silica abrasive. In case of the MRR, however, the addition of the diamond abrasive could cover lower mechanical effects by lowering the concentration of abrasives.

Through the experiments, the importance of the concentration of colloidal silica and diamond abrasive was confirmed. Thus, another experiment was performed by increasing the concentration of diamond abrasive and mechanical energy (Table 2). Fig. 5(b), (c) show the surface was improved up to the third run of the CMP using SAS, and the roughness and scratch depth increased thereafter. Likewise, the MRR was 0.4 mg/h for the first run, but the rate decreased after the first run (Fig. 5(a)). However, the result of the CMP using MAS for the sixth run shows an improvement in every aspect including the surface roughness, scratch depth, and the MRR (Table 4). The reason for the increase of the MRR is that as the concentration in the slurry increases, the effects of increasing mechanical energy appear to be stronger than the effects of decreasing the pressure per unit particle. Fig. 6 shows the result of the CMP process using the MAS after the MP. As seen on the image, no scratches could be found. After measurement, the values of the surface roughness and the scratch depth were lowered (Table 4).



Fig. 5. Results of CMP using SAS (20 wt% colloidal silica) and MAS (20 wt%colloidal silica + Nano-diamond (25 g/l)): (a) MRR, (b) Surface roughness, (c) Scratch depth.

 Table 4. Measurement results after CMP of each condition (Second)

	MP	SAS_1	SAS_2	SAS_3	SAS_4	SAS_5	MAS_1	MP	MAS_2
MRR (mg)		0.4	0	0.1	0	0.1	0.2		0.6
Ra (Å)	3.8	14.9	14.1	9.8	10.8	13.8	3.2	4.7	2.7
Rv (nm)	3	10	10.4	7.4	8	9.3	1.4	3	1.8



Fig. 6. AFM images: (a) after MP and (b) after CMP using MAS (20 wt% colloidal silica + Nano-diamond (25 g/l)).

Therefore, the balance among the abrasive concentration, abrasive type, and chemical component in the slurry are important to ensure the material removal rate and increase the possibility of obtaining a fine surface for the SiC CMP process.

Conclusions

In order to create a surface on a SiC wafer, which when finished with the mechanical polishing process has a fine surface, the CMP process was conducted.

(1) When the pressure, velocity, and concentration of an abrasive were increased, the MRR also increased using a colloidal silica slurry. Also, a valid reaction between the SiC surface and KOH was verified with XPS.

(2) However, the deeper scratches could not be removed, and the surface roughness was not improved. In order to solve these problems, SAS (only colloidal silica and nanodiamond abrasive) and MAS (colloidal silica + nanodiamond) were used for the SiC CMP.

(3) When MAS was used, both mechanical and chemical energy were raised; thus, the surface roughness was improved up to 2.4 Å. However, the scratches were not completely removed, and the MRR was decreased as the abrasive concentration (0.25 mg/h) was increased. The reason for such a result is presumed to be the addition of a small amount of nano-diamond abrasive.

(4) The concentration of nano-diamond abrasive for the MAS was increased for another experiment. As a result, the surface roughness and the condition of scratches were improved, and it was confirmed from AFM images that the scratches were removed from the surface. Likewise, the MRR was increased up to 0.6 mg/h as the concentration was increased. As such, the addition of nano-diamond abrasive results in an improvement of surface roughness, removal of scratches, and a reduction of polishing time.

(5) Through the experiments, the significance of a balance between mechanical and chemical energy was confirmed and verified. Further research regarding the influence of the diverse mixed conditions will be continued.

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