Proceedings of the E-MRS Fall Meeting, Symposium H: Warsaw, Poland, September 19–23, 2011

Detection of Shallow Dislocations on 4H-SiC Substrate by Etching Method

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Correlation between dislocation types in epitaxial 4H-SiC and etch pit types on the 4H-SiC wafer surface were investigated by etch pit method and transmission electron microscope. Shallow dislocation on the wafer was found to form round pit without core. The shallow dislocation was estimated half-loop type in wafer and this estimation explains that step-flow growth converts half-loop dislocation into complex dislocation composed by threading dislocation and basal plane dislocation.

PACS: 61.72.uj, 81.65.Cf, 68.37.Lp

1. Introduction

4H-SiC is a promising material for fabricating high-power, high-temperature and high-frequency electric devices. However dislocations in SiC degrade device performance in life, yield and break down voltage. Generally, devices are integrated in homoepitaxial film deposited on wafer. Therefore, improvement of crystal quality of the epitaxial film brings the advanced function of SiC based electric devices. Most of dislocations in the epitaxial films are formed by dislocation propagation from the wafer.

Dislocations of wafer are composed from deep dislocations caused by crystal growth and shallow dislocations by mechanical treatments. The deep dislocations were well analyzed by X-ray topography and were threading edge, threading screw and basal plane dislocations [1]. However, the surface dislocations remained unclear in spite of Zhang et al. pointed out its importance [2] because the density of the shallow dislocations was much smaller than the density of deep dislocations. However, the recent remarkable developments of SiC crystal increased its importance of shallow dislocations. Recently we reported that round pit formed by molten KOH etching seems to indicate the shallow dislocation [3].

In this paper we investigated the correlation between dislocation types in epitaxial 4H-SiC and etch pit types on the 4H-SiC wafer surface by etch pit method and transmission electron microscope. It is indicated that half-loop type shallow dislocations were detected as round pits without core. We discussed how the round pit forms by half loop dislocation.

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2. Experimental method

Commercially available 0.01–0.05 Ω cm n-type 4H-SiC (0001) wafers made by a sublimation method were used. The (0001) plane was 8° off toward the [11–20] direction. About 8 μm undoped homoepitaxial films were deposited by cold-wall chemical vapor deposition using CH_3SiH_3 and CH_4 as source gases for detecting the dislocation propagation from wafer to epitaxial film. Etching was performed using molten KOH and/or molten KOH added Na_2O_2 (KN) [4, 5] at about 500 °C. The pits were observed using a laser microscope. The dislocation structure under pit was observed by transmission electron microscopy (TEM). The measured areas were picked up using a focused-ion-beam (FIB) microsampling technique.

3. Results

3.1. Etch pit

Figure 1 shows the correlation of etch pits between epitaxial film and the surface of wafer. The image of Fig. 1a was taken after etching the surface of epitaxial film. Figure 1b shows the etched wafer surface after removing the epitaxial film by polishing. Both images were taken from the same area. Large and small hexagonal pits, sea-shell pits and pairs of small hexagonal and sea-shell pits indicated by arrows were observed on the epitaxial film (Fig. 1a). The large and small hexagonal pits can be identified as threading screw dislocations (TSD) and threading edge dislocations (TED), respectively [6–10]. The sea-shell pit was BPDs [6–10]. In the pit pairs, the hexagonal pits were always placed at upper position as shown in Fig. 1a. The periods between the core of hexagonal pit and the tail of sea-shell pit were almost constant in the observed area and consisted in the epitaxial layer thickness divided by tan 8° as shown in Fig. 1c. It shows that TED and boundary phase dislocation (BPD) cross at the interface of the epitaxial film and the wafer. Therefore they can be estimated as complex dislocation of TED and BPD [2, 11]. They were explained by imperfection of epitaxial growth or decomposition of dislocation at the wafer surface [2, 11].

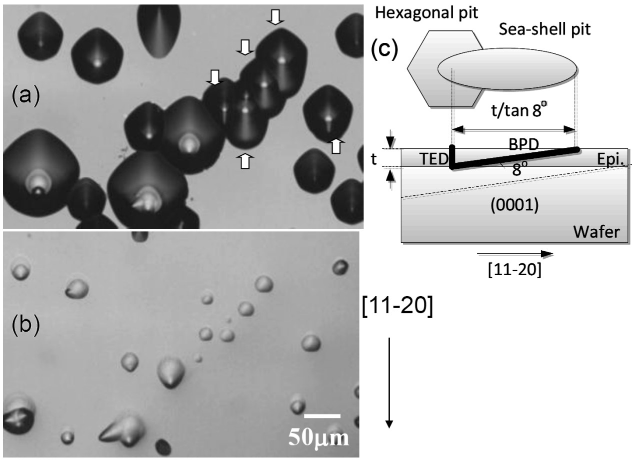

Fig. 1. Etch pit images of epitaxial film (a) and wafer surface (b) at the same area. Etch pit pairs are indicated by arrows. Schematic shows the dislocation structure which forms etch pit pair (c).

Figure 1b shows the etch pits on the SiC wafer of the same area. As the etchant was molten KOH, the etch pits shapes were deformed, however, etch pits under typical dislocations (TED, TSD and BPD) have cores as described in Ref. [3] but under complex dislocations they were round pits without cores. In our study, all observed dislocations that form the round pits without core in SiC wafer transformed to the TED–BPD complex.

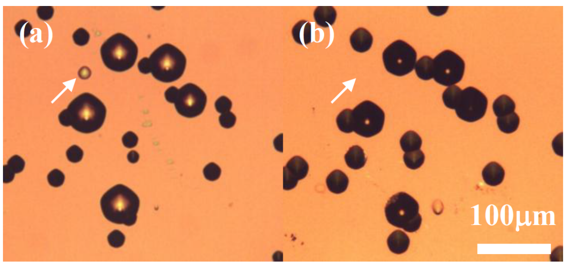

Fig. 2. Etch pit images at (a) the surface and (b) 3 μm under the surface of epitaxial film at the same area.

Similar round pits were observed on commercially available epitaxial wafer. A round pit was observed in Fig. 2a that is the image of etch pits at the surface of a 10 μm epitaxial wafer. However, the round pit was not observed inside the epitaxial wafer as shown in Fig. 2b. The image was taken after etching following 2 μm polish. Therefore, the dislocation related with the round pit seems to shallow.

It should be noted that round pit detection was not dependent on the etching solution. Round pits were also observed after KN etching that was developed for detecting dislocations on highly N-doped n-type SiC. Therefore, molten KOH and KOH+Na_2O_2 are useful for detecting round pits.

3.2. TEM observation

Observation of dislocation under round pit was tried by TEM. Plan-view TEM sample was formed by picking up a 10 μm × 10 μm area 1–2 μm under the bottom of round pit as shown in Fig. 3 using focused-ion-beam microsampling technique. Cross-section type sample was not selected for dislocation observation as encompassing the dislocation in the thin (200 nm) cross-section sample is difficult. On the other hand, the plan-view type sample was suitable because dislocation loss never occurs if dislocation is deep enough to reach the sampling area.

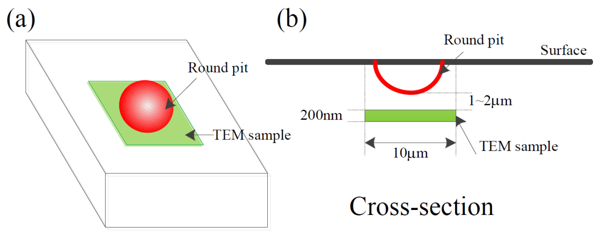

Fig. 3. Schematic diagram of TEM sampling: (a) a bird's-eye view and (b) a cross-section.

However, we found no dislocations on plan-view TEM sample. It shows that dislocation that forms round pit was shallow and never propagates to the inside of SiC wafer.

4. Discussion

As described above, the dislocation that forms round pit can be estimated as a half loop dislocation. If we accept this estimation, the formations of complex dislocation in epitaxial film and round pit are reasonably explained. If there is a half loop dislocation open to the surface, two dislocations that have the same Burgers vector intersect the surface. Repulsive force rises between these two dislocations according to the same Burgers vector (Fig. 4a). In the chemical vapor deposition (CVD) process, epitaxial growth progresses by step flow mode. Therefore, possible propagation direction of dislocation is limited between parallel and perpendicular to the surface as shown in Fig. 4b. One dislocation propagates as a threading dislocation (TD) and another one propagates as a BPD that has the largest period of the two dislocations in possible configurations (Fig. 4c). Thus complex of TD and BPD in epitaxial film is the most probable feature of dislocations that propagate from the half-loop dislocation on SiC wafer.

The round pit formation also can be explained. At first step, two small pits were formed at the surface. The

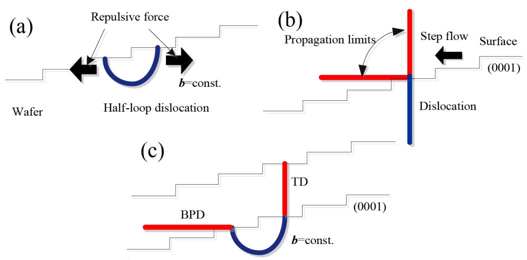

Fig. 4. Schematic diagrams of (a) repulsive force in half-loop dislocation, (b) propagation limits of dislocation in step flow growth and (c) stable dislocation structure that is started from half-loop dislocation and formed in step flow growth.

pit shape can be speculated nearly hexagonal according to the dislocation geometry (Fig. 5b, c). With the progress of etching, an etching area reaches the nearly basal plane dislocation region. At this stage dislocation is almost parallel to the surface, an oval pit without core was formed and hexagonal pits were rounded by absence of threading type dislocations (Fig. 5d). The two rounded hexagonal pits and one sea-shell pit compose one large round pit without core as shown in Fig. 5d.

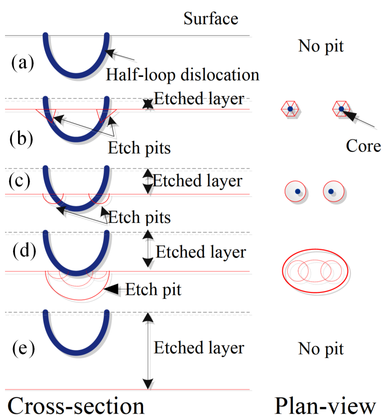

Fig. 5. Schematic diagrams of an etch pit formation. With the progress of etching, the etch pit shape changes from (a) to (e).

Finally, we should discuss the detection accuracy of the shallow dislocation density by round pit density. If half-loop dislocation is much shallower than etched thickness, round pit detection became hard (Fig. 5e), because formed small round pits in early stage disappeared by prolonged etching after the loss of dislocation. If the depth of half-loop dislocation was similar to the etched thickness (Fig. 5d), we can detect half-loop dislocation as a round pit. On the contrary, a pit pair will be detected if the half-loop dislocation was much deeper than the etched thickness. Therefore, only half-loop dislocations that have comparable depth with etched thickness are detected as round pits. However, we observed clear correlation between the round pit density and the density of half-loop induced dislocation in epitaxial film, thus high round pit density on wafer surface results in high complex dislocation density. The round pit detection by etch pit method is useful for estimating crystal quality of wafer surface but accurate density estimation is difficult.

5. Conclusion

Shallow dislocation on the SiC is detected as round pit without core by molten KOH and molten KOH+Na_2O_2 etching. The shallow dislocation is speculated half-loop type. The estimation well describes round pit formation by etching and complex dislocation (threading dislocation and basal plane dislocation) formation in epitaxial film. Accurate density detection by round pit is difficult as the possibility of dislocation detection depends on magnitude correlation between the depth of half-loop and etched thickness. However the density of complex dislocation has tendency to increase with round pit density on the wafer.

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