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Dislocation analysis in highly doped n-type 4*H*-SiC by using electron beam induced current and KOH+Na_2O_2 etching

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Abstract. Dislocations in highly doped n-type 4*H*-SiC (n^+-SiC, $n>10^{19}$ cm^{-3}) substrate have been studied by means of electron beam induced current (EBIC). Ni/n-SiC/n^+-SiC/Al structure was fabricated in order to simultaneously observe the dislocations in n-SiC epilayer and n^+-SiC substrate. We have found that dark dots in the EBIC image correspond to threading screw dislocations (TSDs) and threading edge dislocations (TEDs) with the former being relatively darker. Short dark lines along off-cut $\langle 11\bar{2}0\rangle$ are attributed to basal plane dislocations (BPDs) in the epilayer; and the randomly oriented long dark lines are caused by the BPDs in the substrate. The classification of the dislocations by EBIC has been examined by wet etching in KOH+Na_2O_2.

Introduction

Electron beam induced current (EBIC) is a powerful tool to reveal the electrically active defects in semiconductors because it can provide a spatial visualization of recombination centers that reduce the diffusion length of minority carriers. Although EBIC has been proved to be effective for identifying dislocations in moderately doped n-type SiC ($n<10^{18}$ cm^{-3}) epilayers [1], or SiC *p-i-n* diodes [2], reports on successful EBIC observation for bulky n^+-SiC substrate have been very few. There are several possible reasons. One of them is the great difficulty in making high quality Schottky junction between n^+-SiC and commonly used metals such as Ni, Ti and Au. The n^+-SiC/metal junction is normally ohmic due to the tunneling effect caused by high electron concentration. As well known, an internal electric field, the depletion layer in the case of Schottky junction, is necessary for carrier collecting in EBIC observation. If the junction is ohmic there will be no EBIC signals. Another reason is a greatly reduced diffusion length of holes in n^+-SiC. If the minority carriers have not enough time to reach the dislocations or enter the electric field region that collects carriers before e^--h^- recombination, EBIC contrast can not form. Moreover, to establish a correspondence between dark spots in EBIC images and the dislocation types, other dislocation mapping techniques are required, e.g. KOH etching or x-ray topography. However, KOH etching loses its effectiveness when used for n^+-samples [3] and other techniques are either inconvenient or not suitable for large-area mapping.

In our work, we have proposed a Ni/n-SiC/n^+-SiC/Al structure to observe the dislocations in both n-SiC epilayer and n^+-SiC substrate. The n-SiC epilayer is lightly n-type doped, grown by chemical vapor deposition (CVD). Using this structure, we have obtained high signal-to-noise (S/N) ratio EBIC images for n^+-SiC substrate. By observing the shape and contrast of the dark spots formed in EBIC images, several types of dislocations in the epilayer and the substrate are identified, including threading screw dislocations (TSDs), threading edge dislocations (TEDs) and basal plane dislocations (BPDs). The EBIC results have been examined by making a side-by-side comparison with the results of a newly developed wet etching technique using molten KOH+Na_2O_2 [4].

Experimental procedure

Figure 1 shows a schematic illustration of the EBIC observation. Commercially available epi-ready n^+-$4H$-SiC (0001) substrate ($n>10^{19}$ cm^{-3}, 8° off towards $\langle 11\bar{2}0\rangle$) was used in this work. 1.8 μm-thick nitrogen doped n-type $4H$-SiC epilayer ($n=7.7\times10^{15}$ cm^{-3}) was grown on the Si-face of the substrate at 1650 °C by CVD. Then the sample was cleaned with aceton, ethanol and deionized water, and etched in HF (5%) for 10 min. After HF etching, top (Ni, Ø=2 mm, 10 nm thick) and bottom (Al, Ø=2 mm, 200 nm thick) electrodes were made by vacuum evaporation. EBIC observation was carried out in a scanning electron microscope (SEM, Hitachi-SU8000) with an accelerating voltage (V_{acc}) of 3~30 kV. After EBIC observation the sample was etched in molten KOH+Na_2O_2 at 510 °C twice, to reveal the dislocations in epilayer and n^+-SiC substrate, respectively. Details about KOH+Na_2O_2 etching is described in Ref [4].

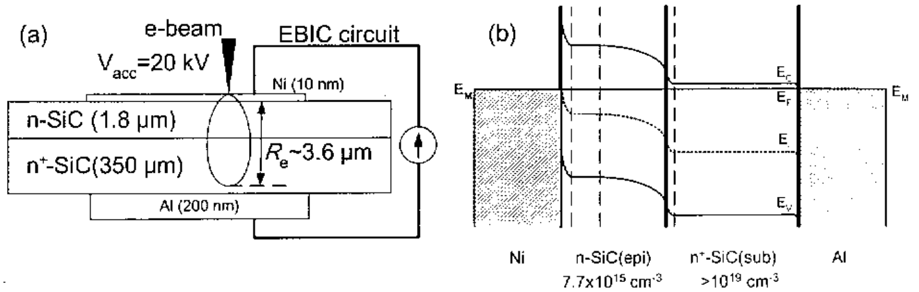

Fig. 1 (a) Sample structure and a schematic illustration of the EBIC observation at V_{acc}=20 kV. R_e: penetration depth of e-beam; (b) Band diagram at equilibrium condition without e-beam excitation

Results and discussion

Figure 2 shows an EBIC image at V_{acc}=20 kV. Dark spots are clearly seen and can be roughly divided into 4 groups according to their shape and contrast: (1) dark dots; (2) less dark dots; (3) short dark lines along $\langle 11\bar{2}0\rangle$; and (4) long dark lines with random orientations (see (1)-(4) in Fig. 2). Note that the contrast difference between (1) and (2) is small. In order to identify the type of dislocations that caused these dark spots, we have made a side-by-side comparison with the wet etching results. Three representative areas are labeled with "①"-"③" in the EBIC image for demonstration.

Figure 3(a)-(c) presents a comparison of EBIC image, 1st-time etching and 2nd-time etching of the areas chosen from Fig. 2. The duration of 1st etching was controlled to remove the topmost 1 μm from the epilayer, thus the etch pits indicate the dislocations in the epilayer. After that the 2nd etching was carried out to completely remove the epilayer and reveal the dislocations in the substrate. Therefore, we are able to make a correlation between the EBIC pattern and the dislocations in epilayer and/or substrate. In Fig. 3(a), a short dark line along $\langle 11\bar{2}0\rangle$ in the EBIC image can be found, and it is attributed to a BPD in the epilayer, as the etch pit after the 1st etching is of seashell shapes; Two other BPDs of the same type can also be found in Fig. 3(c). In Fig. 3(b), a typical TSD is seen at the center. The EBIC contrast of TSD dark dots is normally darker than that of TED dark dots. This is in agreement with Chen *et al.*'s report [1]. The etching results have suggested that TSDs can easily propagate from substrate into epilayer. Another important feature in Fig. 3(b) is a long dark line with one end attaching to a dot. This dot, as indicated by the 1st etching, is caused by a TED in the epilayer. After the 2nd etching, the hexagonal etch pit of TED disappeared due to the removal of the whole epilayer and the BPD in the substrate emerged, which was the origin of the long dark line in the EBIC image. This EBIC pattern suggests a BPD-to-TED conversion that likely occurred at the epi/sub interface. Two more examples of this kind can also be found in Fig. 3(c). Note that the orientations of the seashell-shaped pits after the 2nd etching are consistent with those of the long dark lines.

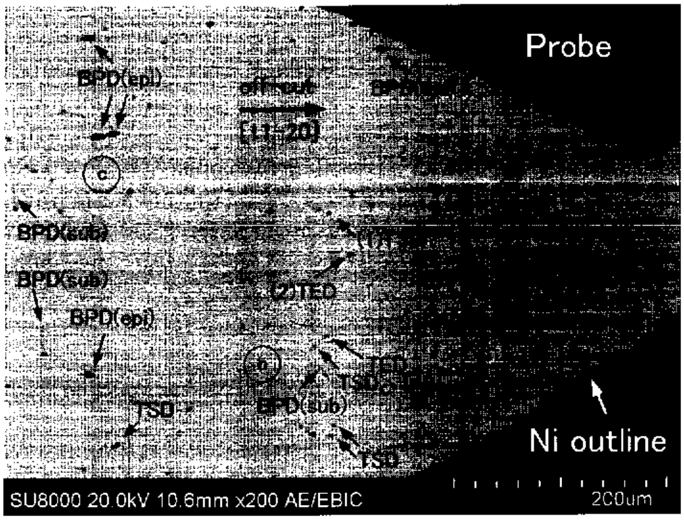

Fig. 2 EBIC image taken at room temperature with V_{acc}=20 kV and V_{bias}=0 V. Three representative areas are labeled with "ⓐ"-"ⓒ" for comparison with the results of wet etching, as shown in Fig. 3

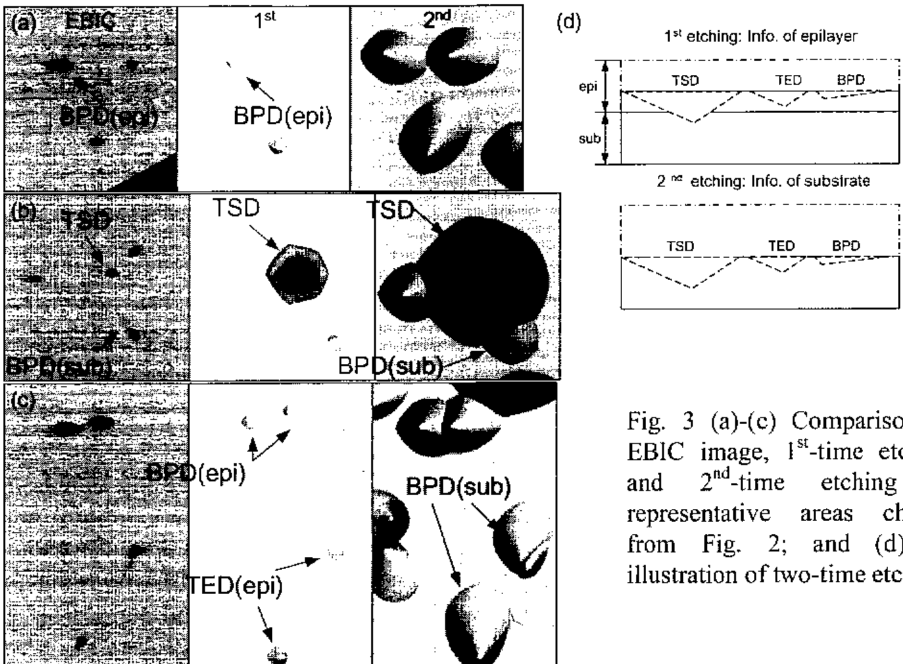

Fig. 3 (a)-(c) Comparison of EBIC image, 1st-time etching and 2nd-time etching of representative areas chosen from Fig. 2; and (d) an illustration of two-time etching.

Figure 4 presents the line profiles of the greyscaled brightness of each type of dislocations. We found that the sizes of the dark dots for TSDs and TEDs are similar, but TSDs are darker than TEDs on average. This contrast difference is small, indicating the carriers have similar recombination rates around them. Thus, it is difficult to make an accurate separation between TSDs and TEDs if only based on EBIC images. Short lines of BPDs in epilayer have shown the darkest contrast. Their length in EBIC image is about 6~8 μm, which is consistent to the 8° off angle and thickness of the electric filed free region in the epilayer (about 1 μm). All the BPDs found in epilayer are along $\langle 11\bar{2}0 \rangle$, probably a result of step-flow growth mode in CVD. Long lines of BPDs in the substrate have less dark contrast compared to other types of dislocations due to their deeper position from sample surface. The orientations of these BPD lines are random, indicating a very different mechanism of BPD formation in sublimation growth mode.

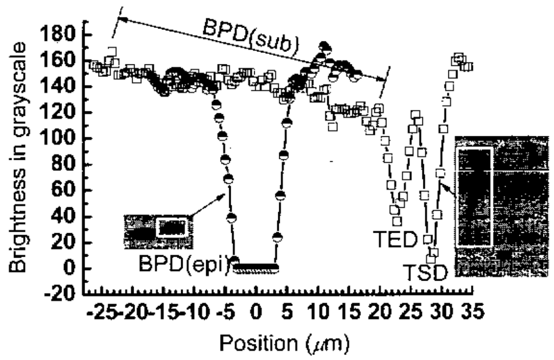

Fig. 4 Line profiles of BPD(epi), TSD, TED and BPD(sub). Brightness in grayscale is extracted from EBIC image shown in Fig. 2.

In the literatures, it is commonly believed that it is difficult to collect EBIC signals from n^+-samples due to the greatly reduced diffusion length of minority carriers. However, our calculation has shown that the diffusion length of holes in n^+-$4H$-SiC ($n=10^{19}$ cm^{-3}) is around 1.7μm. Therefore the e^--h^+ pair generated in the topmost 1.7 μm in the n^+ substrate should be able to diffuse into the epilayer and then be collected through the Ni electrode. This is in good agreement with our observation that V_{acc}=20 kV (R_e=3.6 μm) gives the highest S/N ratio. The above examples have demonstrated that by forming two internal electric filed regions (Ni/n-SiC and n/n^+-SiC) dislocations in the epilayer and substrate can be revealed simultaneously. Further, by changing V_{acc}, one can observe the depth dependent presence of the dislocations and where and how the dislocation conversion happens if any, e.g., a BPD in substrate to a TED in epilayer.

Summary and conclusions

We propose a Ni/n-SiC/n^+-SiC/Al structure for a simultaneous observation of dislocations in n-SiC epilayer and n^+-SiC substrate by EBIC. The classification of dislocation type according to the EBIC results has been examined by wet etching. TSDs and TEDs have been revealed as dark dots slightly differing in contrast. Although TSDs are a little darker, EBIC is not able to provide an accurate separation between TSDs and TEDs due to the similar carrier recombination rate around them. BPDs in the epilayer are observed as short dark lines along $\langle 11\bar{2}0\rangle$, while BPDs in the substrate are less dark and they are randomly oriented. By this technique, we are able to analyze the dislocation conversion behavior from the substrate to the epilayer.

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