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Dislocation revelation in highly doped n-type 4*H*-SiC by molten KOH etching with Na_2O_2 additive

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Abstract. We have proposed a new wet etching recipe using molten KOH and Na_2O_2 as the etchant ("KN etching") for dislocation revelation in highly doped n-type 4*H*-SiC (n^+-4*H*-SiC). Threading screw dislocations (TSDs) and threading edge dislocations (TEDs) have been clearly revealed as hexagonal etch pits differing in pit sizes, and basal plane dislocations (BPDs) as seashell-shaped pits. This new etching recipe has provided a solution to the problem that conventional KOH etching is not effective for dislocation identification in 4*H*-SiC if the electron concentration is high (>mid-10^{18} cm^{-3}). We have investigated the effect of SiC off-cut angle on KN etching and it has been shown that the "KN etching" is applicable for the n^+-SiC substrate with off-angle from 0^o to 8^o.

Introduction

Revealing and identifying dislocations in a crystal is of great importance for quality control and device reliability analysis. Among a variety of dislocation revealing techniques, wet etching is extensively used for SiC due to its low cost, simple operation and nearly zero-requirement for sample geometry. In moderately doped n-type 4*H*-SiC (n-4*H*-SiC, $n<10^{18}$ cm^{-3}) or p-type 4*H*-SiC (p-4*H*-SiC), dislocations like TSDs, TEDs and BPDs can be easily revealed and identified according to the size and shape of the etch pits formed during molten KOH etching around 500 °C [1]. However, in the case of n^--4*H*-SiC ($n=10^{18}$~10^{19} cm^{-3}), conventional KOH etching reveals TSDs and TEDs as round pits with continuous size distribution instead of hexagonal ones, making it difficult to classify the dislocations, especially to separate TSDs from TEDs [2]. Besides the issue of identifying dislocations types, it is also debatable whether KOH etching can correctly show the density of threading dislocations, because the etching of n^+-SiC in pure KOH is driven by electrochemical force, therefore it is more affected by the hole concentration on the surface inversion layer than by the strain around a dislocation. To solve this problem, we have proposed to use molten KOH+Na_2O_2 as the etchant for wet etching of SiC to enhance the defect-selective oxidation process. In this paper, we report an *in-situ* comparison between KOH and KOH+Na_2O_2 etching ("KN etching"). The signature of each type of dislocations after KN etching are presented.

Experimental procedure

Commercially available n^+-4*H*-SiC (0001) substrates with different off-axis angles of 0^o, 4^o and 8^o towards $\langle 11\bar{2}0\rangle$ were etched in molten KOH and Na_2O_2 at 510 °C. Typical etching time was 2~6 min. After etching, the SiC substrates were cleaned with ultrasonic bath in DI water and the Si-face was observed by laser microscope that has a high depth sensitivity (vertical resolution=10 nm).

Results and discussion

Etch pits revealed by KOH+Na_2O_2. Figures 1(a)-(d) show the Si-faces of the n^+-4H-SiC (4° off) etched in KOH and in KOH+Na_2O_2 for 2, 4 and 6 min, respectively. For KOH etching (Fig. 1(a)), most of the etch pits are of round shape and these pits have shown a continuous distribution in their size. Although on some of the round pits, hexagonal inner lines can be seen, these lines are very blurred and six-fold facets are not clear on the bottom of etch pits. This phenomenon is commonly observed when conventional KOH etching is carried out for 4*H*-SiC with high electron concentration. According to Gao *et al.* [2], TSDs and TEDs are revealed as round pits instead of hexagonal ones because KOH etching is isotropic on n^+-samples, while it is more preferential on moderate n-, p- and p^+- samples. They suggest that this isotropic etching behavior is caused by a p-type inversion layer on n^+-SiC surface due to serious upwards band bending. The round pits with continuous size distribution makes it impossible to separate TEDs from TSDs. In Fig. 1(a), seashell-shaped pits have also been found, presumably attributed to BPDs.

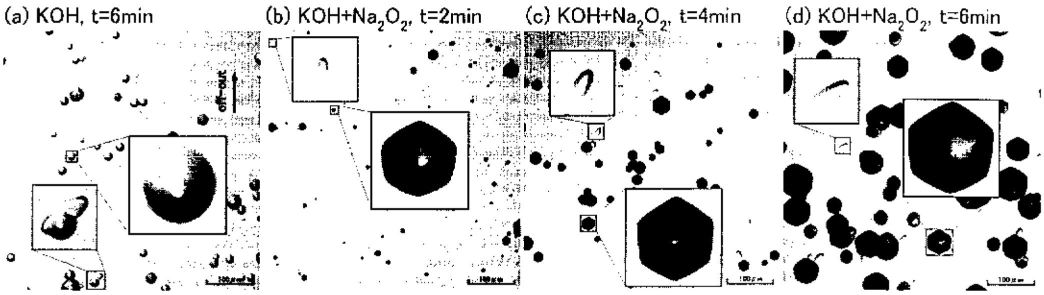

Fig. 1 Microscope photographs of n^+-4*H*-SiC (Si-face, 4° off) etched in (a) KOH for 6 min; (b) KOH+Na_2O_2 for 2 min; (c) KOH+Na_2O_2 for 4min; and (d) KOH+Na_2O_2 for 6 min. Representative pits are enlarged.

In comparison to Fig. 1(a), hexagonal pits are clearly observed for KN etching in Fig. 1(b)-(d). In our experiments, Na_2O_2 decomposes in molten KOH above 460 °C and release oxygen. By adding Na_2O_2, we intend to enhance the defect-selective anisotropic oxidation process and transform round pits to hexagonal ones.With increasing etching time from 2 min to 6 min, the hexagonal pits increase in size as a general trend. For a certain etching time, the pit sizes (diagonal along $\langle 11\bar{2}0\rangle$) have a distribution, which is divided into 20 levels with constant interval, as shown in Fig. 2. Here, the pit number (counted in a 2.15×1.56 mm^2 area) of each level is plotted as a function of pit size. These histograms clearly imply 3 groups of hexagonal pits, from small (S), middle (M) to large (L). Pit size in group "S" is relatively uniform and they are supposed to correspond to TEDs with $\boldsymbol{b}=\frac{a}{3}\langle 11\bar{2}0\rangle$.

Etch pits in group "M" are attributed to TSDs, as been examined later by using transmission electron microscopy (TEM). The TEM observation has also implied that the pit size fluctuation within the same type of dislocation might originate from the difference (several degrees) in the tilting angles of dislocation lines with respect to c-axis. In other words, the main component of the dislocations under "M" size pits is of screw type ($\boldsymbol{b}$=1c) with a possible mixing with small edge component. The etch pits with "L" size are much more complicated. There are several possible origins of the large pits: (1) mixed type dislocations with screw component ($\boldsymbol{b}$=1c) and relatively large edge component ($\boldsymbol{b}=\frac{na}{3}\langle 11\bar{2}0\rangle$, n>1); (2) dislocation complexes or multiple dislocations that are spatially close to each other; (3) TSDs with $\boldsymbol{b}$=2c or 3c, or MPs with $\boldsymbol{b}$>3c. Note that (3) is possible but rarely found in our samples, as indicated by our study on dislocation conversion from substrates to epitaxial layers.

BPDs are revealed as seashell-shaped pits with a density of ~2000 cm^{-2} independent of the etching time. The orientation of BPDs is not always along off-cut $\langle 11\bar{2}0\rangle$ direction.

It is worth noting that the observed hexagonal pit density depends on the etching time because of the overlapping effect. Therefore, etching time needs to be optimized according to the sample quality in order to correctly reveal the density as well as the type of dislocations. For our samples, the optimal etching time is around 4 min, and the representative etch pits are shown in Fig. 3.

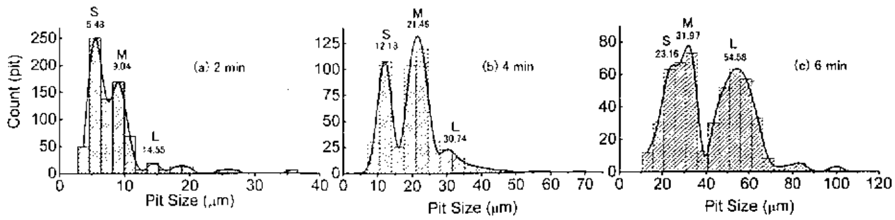

Fig. 2 Hexagonal pit size distribution for (a) 2 min; (b) 4min; and (c) 6 min etching in $KOH+Na_2O_2$. Calculation is based on a 2.15×1.56 mm^2 area. Solid lines are a guide for eyes.

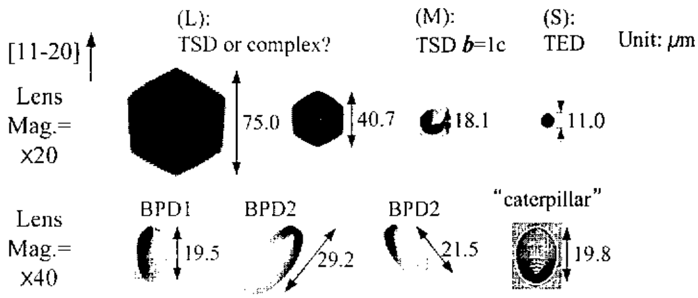

Fig. 3 Representative etch pits for 4 min KN etching. See Ref [3] for details about caterpillar pits.

***In-situ* comparison between KOH and KN etching.** In order to understand the correlation between KOH and KN etching, we have carried out a two-time etching experiment. Figure 4 shows a side-by-side comparison of some representative areas between the first time KOH and the second time KN etching. We have found a one-to-one correlation of etch pits between the two times with extremely few exceptions. This indicates that *under the current etching condition* both KOH and $KOH+Na_2O_2$ can correctly reveal the overall density of dislocations although KOH etching can not separate TSDs from TEDs. In fact, in some other cases, KOH etching can not reveal all dislocations, resulting in an underestimated dislocation density. Furthermore, comparisons of the pit shapes between the two times has led us to several interesting findings: (1) BPDs are revealed as seashell-shaped in both cases (Fig. 4(a)); (2) KN etching can identify each type of dislocations existing as a complex or overlapped with each other (Fig. 4(b)); especially when TDs overlapped with BPDs, they can be easily separated due to very different pit shape; (3) Etch pits for TSDs and TEDs have changed from round to hexagonal after the second time KN etching (Fig. 4(c)-(f)). Double outlines composed of inner hexagon and circular periphery have been seen, suggesting the different etching mechanism and defect-selectivity between KOH and KN etching. Note that some KOH-etched round pits with similar size have transformed into different hexagonal size after KN etching (Fig. 4(f)), implying they actually correspond to different types of dislocations. Thus, it would be wrong to treat them as the same type, which is seemingly obvious if based only on KOH etching results.

Effect of off-axis angle. As shown above, the effects of KN etching has been examined using 4^o-off n^+-4*H*-SiC. We have also applied this etching method to other n^+-samples ($n \sim 10^{19}$ cm^{-3}) with different off-angles. Figure 5 shows typical etched surfaces for 0^o- (just-on-axis) and 8^o-off samples. For 0^o-off sample, the etch pits have shown perfect six-fold symmetric shapes and a sharp separation between two size levels. With this image, one can easily calculate the density for TSDs and TEDs. Note that BPDs do not appear for 0^o-off samples. For 8^o-off sample, the deformed hexagonal shape is well consistent with the off-angle. Similarly, TEDs, TSDs and BPDs can be easily separated. Therefore we concluded that the KN etching is applicable to any off-angles from 0^o to 8^o.

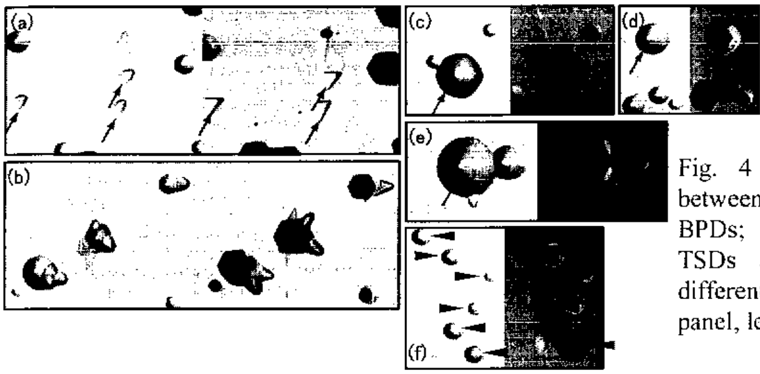

Fig. 4 An *in-situ* comparison between KOH and KN etching (a) BPDs; (b) TSDs+BPDs; (c)-(f) TSDs and TEDs. Scales are different from (a) to (f). In each panel, left: KOH and right: KN.

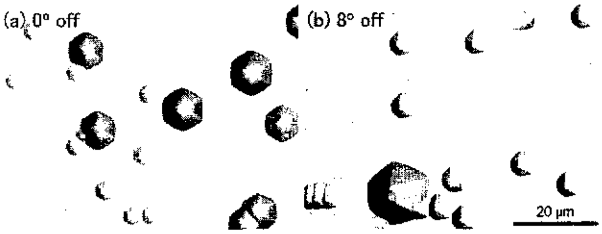

Fig. 5 n^+-4*H*-SiC with different off-axis angles (a) 0^o and (b) 8^o, etched by $KOH+Na_2O_2$.

Summary and conclusions

We have developed a new etching recipe, $KOH+Na_2O_2$, to reveal dislocations in n^+-4*H*-SiC. The "KN" method has effectively revealed TSDs and TEDs as hexagonal pits differing in size and BPDs as seashell-shaped pits. A one-to-one comparison between KOH and KN etching has proved an enhanced oxidation by Na_2O_2, which is the key to transforming round shape of the pits to hexagonal. Our results have also shown that the "KN etching" is applicable to the n^+-SiC substrate with off-angles from 0^o to 8^o.

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