

Research Article

TEM Observation of the Dislocations Nucleated from Cracks inside Lightly or Heavily Doped Czochralski Silicon Wafers

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The crack propagation from the indent introduced with a Vickers hardness tester at room temperature and the dislocation nucleation from the cracks at 900°C inside lightly boron (B), heavily B, or heavily arsenic (As) doped Czochralski (CZ) Si wafers were investigated with transmission electron microscopy (TEM) observations. It was found that the dopant concentration and the dopant type did not significantly affect the crack propagation and the dislocation nucleation. The slip dislocations with a density of about $(0.8\sim 2.8) \times 10^{13}/\text{cm}^3$ were nucleated from the cracks propagated about 10 μm in depth. Furthermore, small dislocations that nucleated with very high density and without cracks were found around the indent introduced at 1000°C.

1. Introduction

As the Si wafer increases in diameter to and larger than 300 mm, thermal and gravitational stresses greatly increase [1, 2]. In some cases, these stresses cause Si wafers to deform [3] and/or get destroyed due to the damages introduced by cracks and dislocations. Thus, significant efforts are currently underway to eliminate cracks and slip dislocations that damage Si wafers. In parallel to this, the character of the cracks [4] and slip dislocations [5, 6] are being studied.

The Vickers hardness test is a useful tool that introduces an indent at the Si wafer surface with a controlled load and time [5, 6]. Cracks are introduced from an indent along the $\langle 110 \rangle$ directions in the (001) Si surface at room temperature. Furthermore, punched-out dislocations that had moved several tens of micrometers from an indent during heat treatments have been observed at the surface with an etching method and after heat treatments [6].

In recent times, primarily heavily boron (B) or arsenic (As) doped p/p+ or n/n+ Czochralski (CZ) Si epitaxial wafers are used as substrates for LSI or power devices, respectively. Maintaining the strength of large-diameter heavily doped Si wafers is also a major problem. It was determined with an etching method that the movement of punched-out dislocations was drastically suppressed by the pinning effect

of heavily doped B [6] or As [7] atoms. However, the effect of the heavy B or As doping on the propagation of cracks and the nucleation of dislocations inside Si wafers has not been fully investigated. Therefore, in this TEM study we have investigated the propagation of cracks introduced from the indent with a Vickers hardness tester at room temperature and the nucleation of dislocations from cracks at 900°C inside lightly B, heavily B, or heavily As doped CZ Si wafers. Furthermore, we have investigated the dependence of the characters of cracks and dislocations on the temperature at which an indent is introduced. To our knowledge, the cracks and dislocations temperature dependence has not yet been reported in spite of results on the performance of heat treatments on Si wafers during some manufacturing processes, such as epitaxy and LSI processes.

2. TEM Observations of Cracks Introduced at Room Temperature

2.1. Before the Heat Treatments. Lightly B doped (5×10^{15} atoms/cm³), heavily B doped (1×10^{19} atoms/cm³), and heavily As doped (4×10^{19} atoms/cm³) CZ Si (001) wafers were prepared as samples. The oxygen concentration of each wafer is about 1.0×10^{18} atoms/cm³ (old ASTM). The

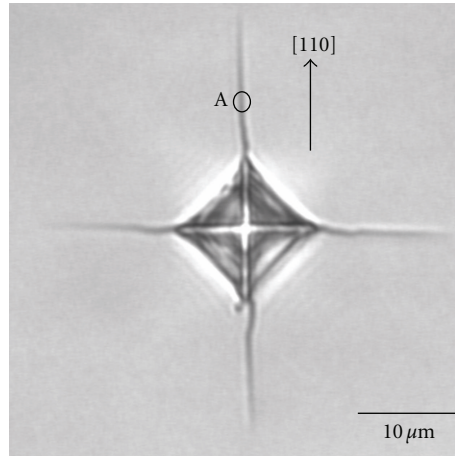


FIGURE 1: Typical image of optical microscopy for indent introduced with Vickers hardness tester at room temperature with load of 100 gf for 10 sec in lightly B doped Si.

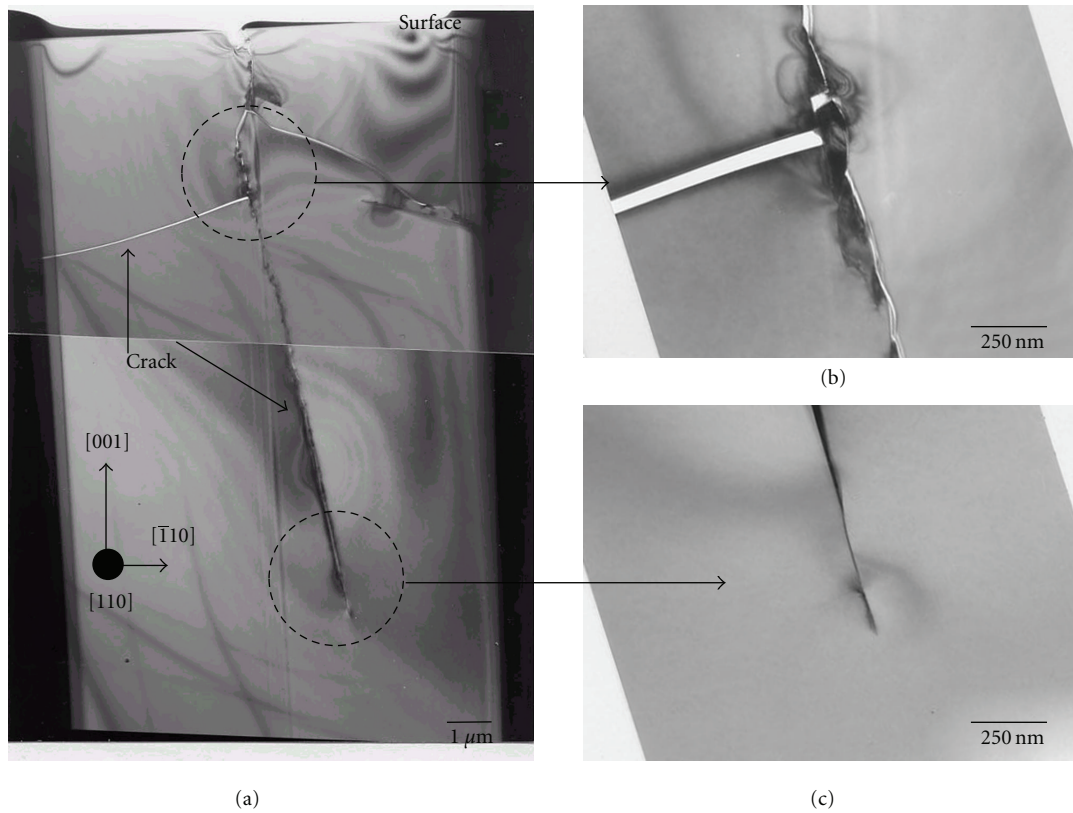


FIGURE 2: Cross-sectional TEM images of lightly B doped Si including crack generated from indent (point A in Figure 1) introduced with Vickers hardness tester with load of 100 gf for 10 sec.

diameter was 200 mm for B doped wafers and 150 mm for As doped wafers. Each wafer was cut into quarter samples.

Twenty indents were introduced at the surface of lightly B doped Si with a load of 100 gf for 10 sec using Vickers hardness tester (Akashi, HM) at room temperature. Figure 1 shows the typical image of optical microscopy for four cracks generated from an indent at the surface of lightly B doped Si. It was found that the cracks were introduced about $10\ \mu\text{m}$

in length along the Si $\langle 110 \rangle$ directions. The average Vickers hardness was 780.2 HV0.1 at room temperature. A cross-sectional TEM sample of one crack at about $3\ \mu\text{m}$ from the edge of an indent (point A in Figure 1) was prepared with a focused ion beam process (Hitachi, FB-2000A). TEM (JEOL, JEM-3010) observations were carried out at 300 kV from the $[110]$ direction. Figure 2 shows bright-field TEM images of lightly B doped Si including a crack introduced with the

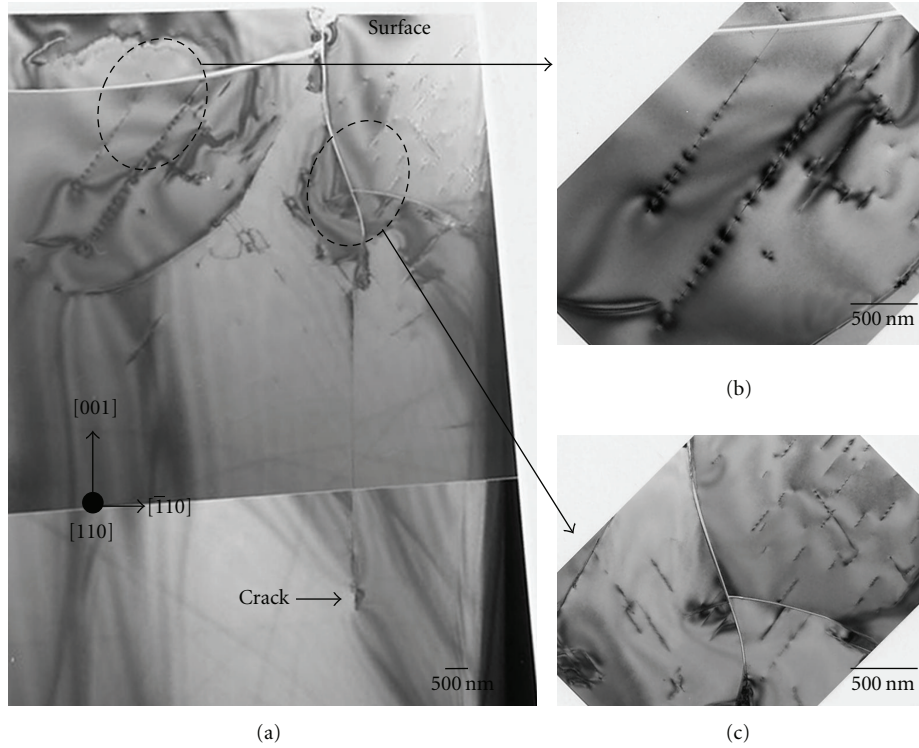


FIGURE 3: Cross-sectional TEM images of lightly B doped Si including crack after heat-treatment at 900°C for 30 min. Indent is introduced at room temperature with Vickers hardness tester with load of 50 gf for 10 sec.

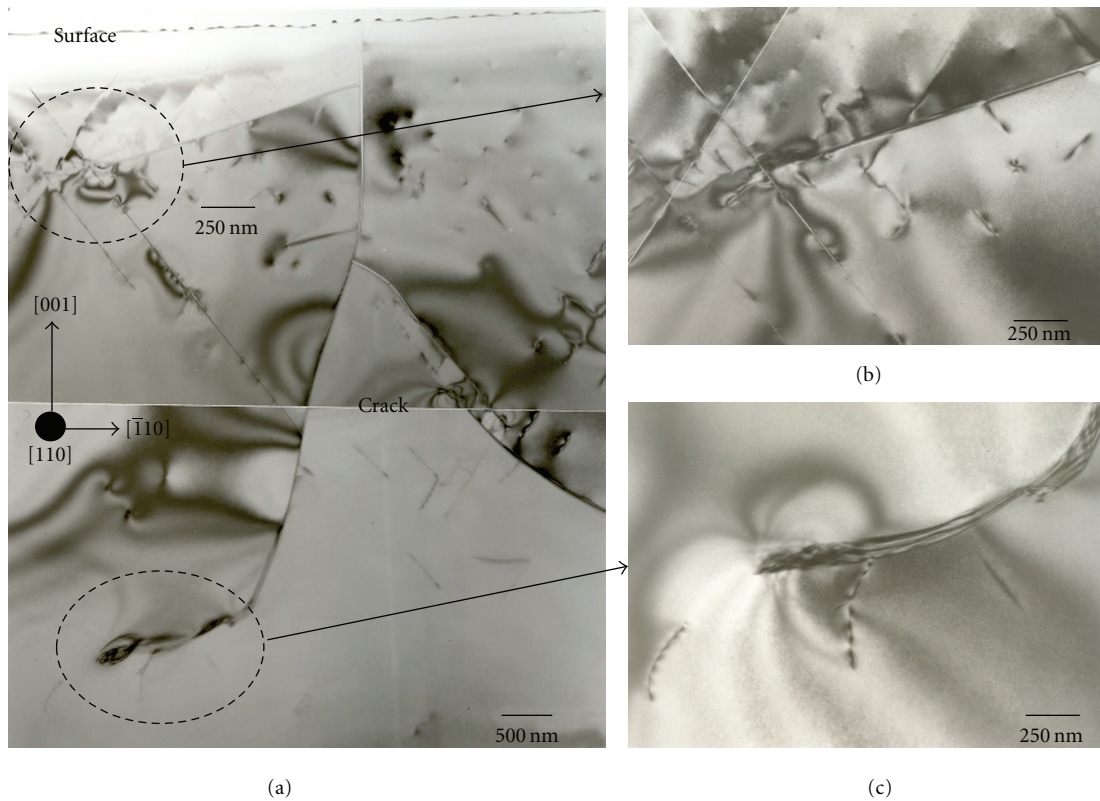


FIGURE 4: Cross-sectional TEM images of heavily B doped Si including crack after heat-treatment at 900°C for 30 min. Indent is introduced at room temperature with Vickers hardness tester with load of 50 gf for 10 sec.

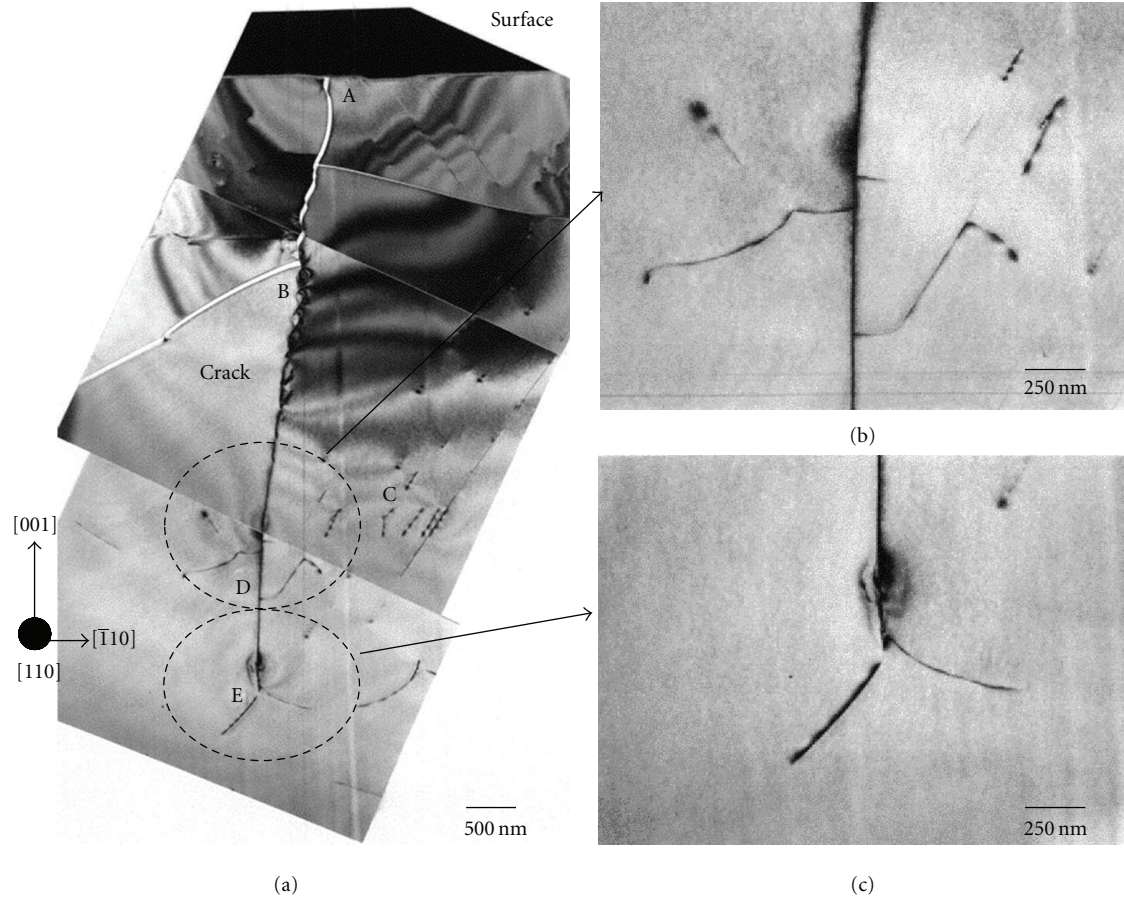


FIGURE 5: Cross-sectional TEM images of heavily As doped Si including crack after heat-treatment at 900°C for 30 min. Indent is introduced at room temperature with Vickers hardness tester with a load of 50 gf for 10 sec.

Vickers hardness tester. It was found that cracks about 13 μm deep propagated along the $\{111\}$ and $\{110\}$ Si planes. Lattice strains are observed around the cracks as shown in Figures 2(b) and 2(c). However, no dislocations were detected with TEM observations.

2.2. After the Heat-Treatments at 900°C. Twenty indents were introduced at the surface of lightly B, heavily B, or heavily As doped Si with a load of 50 gf for 10 sec. These samples were set on a quartz boat and heat treated in a horizontal furnace at 900°C for 30 min. Figure 3 shows TEM images of the dislocations nucleated from the cracks in lightly B doped Si. It was found that the cracks about 10 μm deep propagated along the $\{111\}$ and $\{110\}$ Si planes. Most dislocations are screw or 60° slip dislocations, which lie along the $\langle 110 \rangle$ directions on $\{111\}$ Si planes. The density of the dislocations in mostly nucleated area was roughly evaluated at about $2.0 \times 10^{13}/\text{cm}^3$. Because no dislocations were observed before the heat-treatment, the observed slip dislocations were expected to nucleate during heat-treatment at 900°C. It is worthwhile noting that, in previous etching works, dislocations observed several tens of micrometers apart from

an indent were not reported as slip dislocations but as punched-out dislocations [6]. From our TEM observations it is concluded that (i) cracks are propagated inside the Si crystal at room temperature and (ii) that these cracks are the sources of slip dislocations at 900°C. Figures 4 and 5 show the cross-sectional TEM images for heavily B and As doped Si, respectively. It was confirmed that the slip dislocations were nucleated from the propagated cracks in both samples. The propagated depths of the cracks along the $\{111\}$ and $\{110\}$ Si planes in both samples were almost equal at about 7 μm . The depth of the propagated cracks was close to that in lightly B doped Si. Furthermore, the density of dislocations in the highly nucleated area around the cracks was roughly evaluated at about $2.8 \times 10^{13}/\text{cm}^3$ or $0.8 \times 10^{13}/\text{cm}^3$ in heavily B or As doped Si, respectively. These densities do not differ significantly from those of lightly B doped Si. Therefore, it is concluded that the dopant concentration and type did not affect significantly the propagation of cracks and the nucleation of dislocations. It is valuable to note again that dislocation movement was drastically suppressed by the pinning effect of heavy doped B [6] or As [7] atoms, although the dislocation nucleation was not suppressed, as determined in the present study.

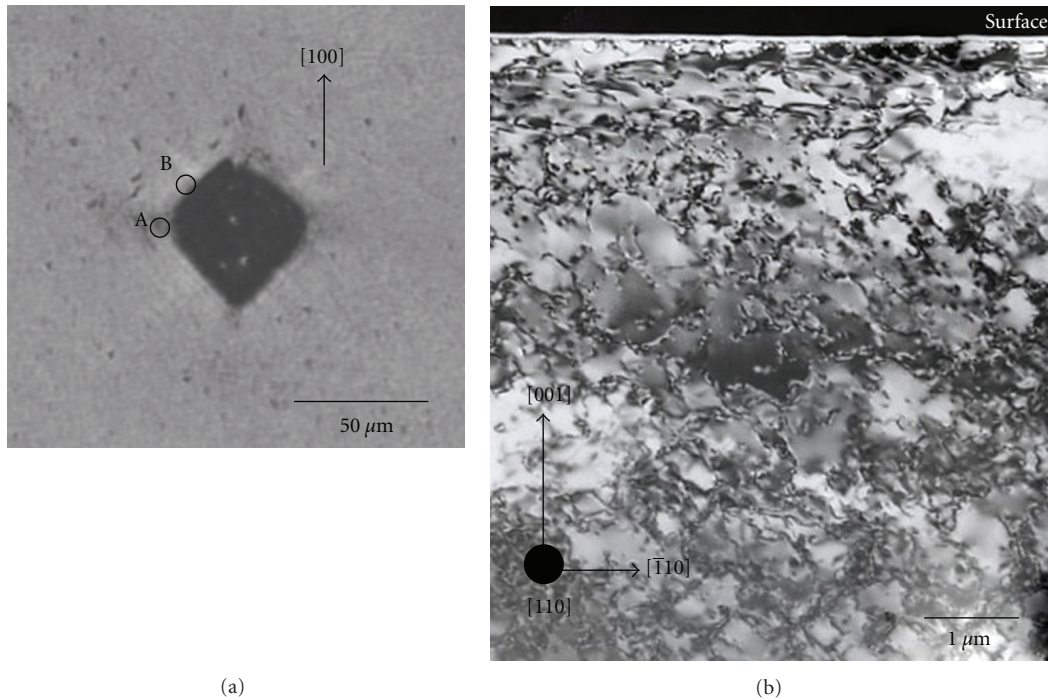


FIGURE 6: (a) Typical image of optical microscopy for indent introduced with high-temperature Vickers hardness at 1000°C with a load of 100 gf for 30 sec in lightly B doped Si and (b) cross-sectional TEM image of lightly B doped Si at edge of indent (point A in (a)).

3. TEM Observations of Cracks Introduced at 1000°C

We investigated the dependence of the characters of cracks and dislocations on the temperature at which an indent is introduced. Lightly B doped sample was indented at 5 points with a load of 100 gf for 30 sec using high-temperature Vickers hardness tester (Nikon, QM) at 1000°C . The samples are held at 1000°C for 5 min after the indentation. Figure 6(a) shows the typical image of optical microscopy for an indent. It was found that the indent is about five times larger than the indent introduced at room temperature (point A in Figure 1). Furthermore, no cracks are observed at the wafer surface. The average Vickers hardness was 55.3 HV0.1 at 1000°C , which was about one order less than the hardness at room temperature. Cross-sectional TEM samples at points A and B shown in Figure 6(a) were prepared. Figure 6(b) shows a cross-sectional TEM image at point A. Very high density of small dislocations is observed. No cracks were observed in the TEM sample. Similar TEM observation results are obtained at point B.

TEM observations in this work showed that only cracks are introduced at room temperature, while only dislocations are introduced at 1000°C . These results can be explained by the evolution of the Si crystal from ductile to brittle with increased temperature. The optimal temperature was estimated to be about 740°C , below which the punched-out dislocations were not nucleated while above which the punched-out dislocations were nucleated from oxide precipitates [8].

4. Conclusions

We have used TEM observations to investigate the propagation of cracks from the indent introduced at room temperature with a Vickers hardness tester and the nucleation of dislocations from the cracks at 900°C inside lightly B, heavily B, or heavily As doped Si. It was found that the dopant concentration and the dopant type did not significantly affect the propagation of cracks and the nucleation of dislocations. In every sample, cracks about $10\mu\text{m}$ deep propagated along the $\{111\}$ and $\{110\}$ Si planes. Furthermore, slip dislocations with density about $(0.8 \sim 2.8) \times 10^{13}/\text{cm}^3$ were nucleated from these cracks at an elevated temperature. We also found that small dislocations with very high density and without cracks were nucleated around the indent introduced at 1000°C .

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