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Nanoscale experimental study of the morphology of a microcrack in silicon by transmission electron microscopy

D S LIU[1,2], C W ZHAO[2,*] and X H HOU[3]

[1]Communications and Electronics Engineering Institute, Qiqihar University, Qiqihar 161006, China
[2]College of Science; [3]Test Center, Inner Mongolia University of Technology, Hohhot 010051, China
*Corresponding author. E-mail: zhaocw@imut.edu.cn

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Abstract. A microcrack in a silicon single crystal was experimentally investigated using high-resolution transmission electron microscopy (HRTEM). In particular, the numerical Moiré (NM) method was used to visualize the deformations and defects. The lattice structure of the microcrack was carefully observed at the nanoscale. HRTEM images of the microcrack demonstrated that the lattice structure of most of the microcrack regions is regular with good periodicity. In addition, the microcrack cleavage expands alternately along different crystal planes, where the principal cleavage plane is the (1 1 1) crystal plane. The NM maps showed no sharp plastic deformation around the microcrack, but discrete edge dislocations can be found only near the crack tip.

Keywords. Microcrack; high-resolution transmission electron microscopy; numerical Moiré; cleavage plane.

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1. Introduction

Crack plays a very important role in the fracture of structural and functional materials. The research on crack is a significant topic in fracture mechanics and has attracted considerable scientific interest. Rice [1] predicted a type of patchy plastic deformation around crack tips in metallic single crystals, a behaviour that was confirmed experimentally [2] and investigated numerically [3]. Gan *et al* proposed that it was possible to observe a regular lattice array of periodic structure in front of a crack tip [4], which led us to the question whether the lattice structure of crack in single crystals is regular with good periodicity. Although numerous experimental, analytical, and numerical investigations of the behaviour of crack in single crystals are available in the literature, few authors could predict certain mechanical behaviours at the nanoscale.

Recently, high-resolution transmission electron microscopy (HRTEM) has become a powerful characterization and metrology tool for solid materials at the nanoscale [5]. Many experimental methods for measuring deformation have also been developed and applied at the nanometre scale. The nano-Moiré method is used to measure deformation up to 0.1 nm [6]. The scanning Moiré method [7] is developed and applied for measuring the deformation at the nanometre scale. Geometric phase analysis (GPA) [8] and numerical Moiré (NM) [9,10], two recently developed techniques that are high-accuracy nanoscale deformation field measurement methods, are sensitive to small displacements of lattice fringes in HRTEM images. HRTEM and GPA have been applied to a wide variety of systems such as dislocation [11], nanoparticles [12] and low-angle grain boundaries [13]. In the present paper, we present a nanoscale experimental study of a microcrack in silicon using TEM and the NM derived from the GPA.

2. Experimental method

The experimental sample was the silicon single crystal. The TEM sample was prepared for cross-sectional imaging along the [1 1 0] direction using standard techniques involving mechanical grinding followed by ion milling [14]. For brittle materials such as silicon single crystal, many microcracks could be formed in this process. However, there were very few cracks that were suitable for HRTEM observation. A crack whose tip is in the thin area of the TEM sample may be ideal for HRTEM observation. The HRTEM experiment was performed using a JEM-2010 TEM at an acceleration voltage of 200 kV. Images were recorded on a Gatan 1024 × 1024 slow-scan CCD camera and processed using the software GPA Phase, developed in the Gatan Digital Micrograph environment.

3. Results and discussion

A bright-field TEM image of the examined microcrack is depicted in figure 1. The observed orientation is [11 0]. The HRTEM images of regions A–C in figure 1 are shown in figures 2a, 3a and 3b, respectively. Figure 2a depicts the lattice structure of the crack tip (region A from figure 1). The area of the observed crack tip is 17.8 nm × 17.8 nm. The figure shows that the lattice structure of the area is regular and has good periodicity. Through careful observation, however, a few deformation and defects can be found.

Figure 2b shows a Fourier transform of the HRTEM image (depicted in figure 2a). Geometric phase images were calculated for the two sets of {111} lattice fringes using Gaussian masks. These images are shown in figures 2c and 2d, respectively. To visualize this arrangement, two NM images have been calculated using a magnification factor of three. The results are shown in figures 2e and 2f. The Moiré pattern acts as a lens which magnifies both lattice spacing and deformation. We can observe that the distance between $(1\bar{1}1)$ crystal planes is larger at the crack tip, which shows that the crack extension probability along the $(1\bar{1}1)$ crystal planes is the biggest. No sharp plastic deformation can be found at the crack tip area, though discrete edge dislocations can be found near the crack tip.

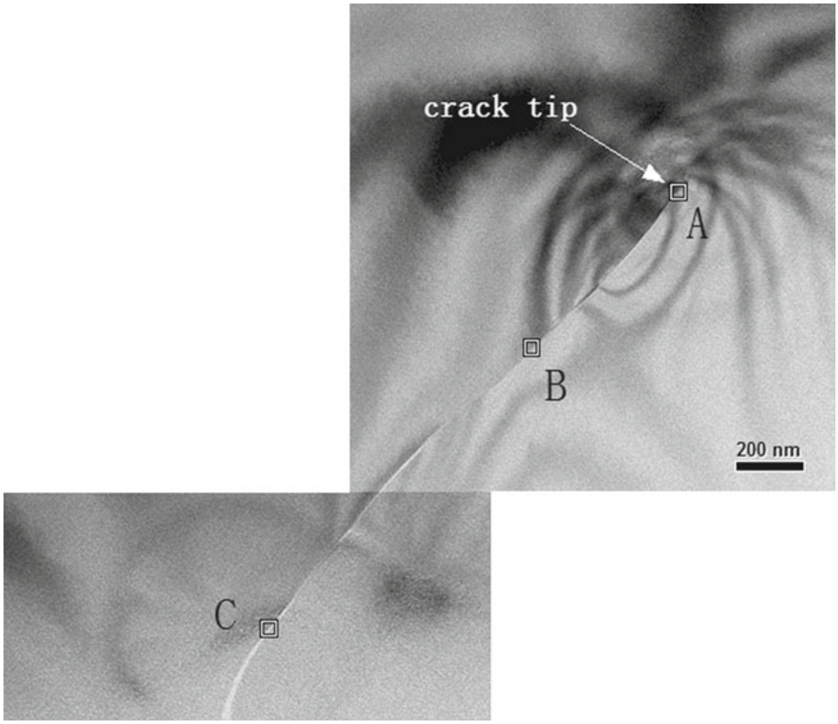

Figure 1. Bright-field TEM image of a microcrack in silicon in the [1 1 0] orientation.

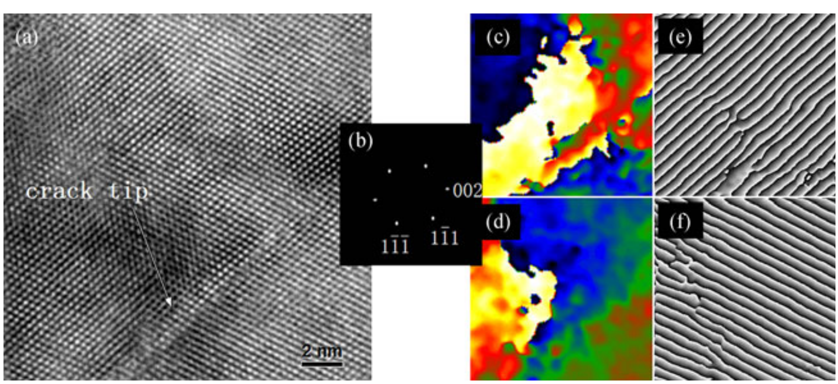

Figure 2. NM analysis of the crack tip area: (**a**) HRTEM image of the crack tip area (region A from figure 1); (**b**) Fourier transform image of the image (**a**); (**c**) phase image of $(1\bar{1}1)$ lattice fringes; (**d**) phase image of $(1\,\bar{1}\,\bar{1})$ lattice fringes. Colour range $-\pi$ to π rad; (**e**) NM image calculated from $(1\,\bar{1}\,1)$ phase image; (**f**) NM image calculated from $(1\,\bar{1}\,\bar{1})$ phase image.

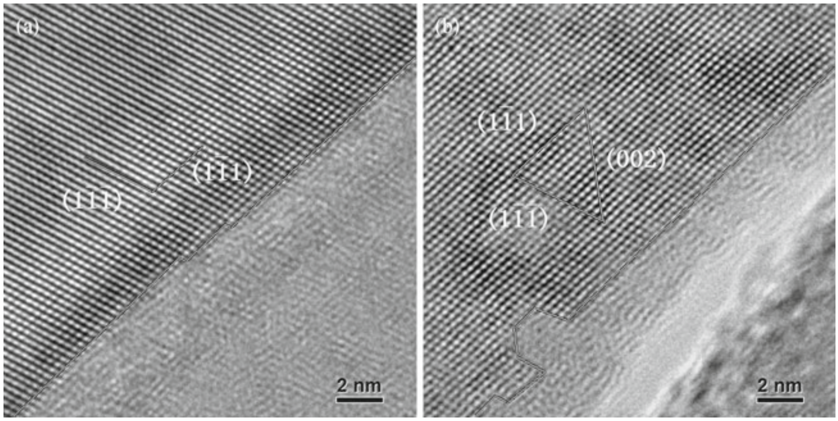

Figure 3. HRTEM images of the microcrack: (**a**) high-resolution image of the middle of the microcrack (region B from figure 1); (**b**) high-resolution image of the opening area of the microcrack (region C from figure 1).

Figure 3a depicts the lattice structure at the middle of the microcrack (region B of figure 1). Only a two-dimensional HRTEM image of one side of the microcrack was obtained because the two sides of the microcrack were not on the same plane. The size of the observed area is 17.8 nm × 17.8 nm. From this figure, it can be observed that the lattice structure contained therein is regular and has good periodicity. The crack cleavage expands alternately along the (1 $\bar{1}$ 1) and ($\bar{1}$ 1 1) crystal planes, while the principal cleavage plane is in the (1 $\bar{1}$ 1) crystal plane. We attempted to measure the deformation of the entire field by the NM method. However, the measurement result shows that there are no observable deformations or defects in the area.

Figure 3b depicts the lattice structure at the opening area of the microcrack (region C from figure 1). Only a two-dimensional HRTEM image of one side of the microcrack was obtained because the two sides of the microcrack were still not on the same plane. That is, the zone axis of the right side was not parallel to the [1 1 0] orientation of the left side. The size of the observed area is 17.8 nm × 17.8 nm. From this figure, we can also observe some amorphous phase content in the crack body, which may have been formed during ion milling of sample preparation [14]. Meanwhile, the lattice structure is still regular and has good periodicity. There are no observable deformations and defects in the area. The crack cleavage expands alternately along the (1 $\bar{1}$ 1), ($\bar{1}$ 1 1) and (0 0 2) crystal planes, while the principal cleavage plane is in the (1 1 1) crystal plane.

4. Conclusion

TEM and NM were employed to study the morphology of a microcrack in silicon. HRTEM images of the microcrack confirmed that the lattice structure of most of the microcrack regions was regular with good periodicity. The investigated microcrack was observed to have cleavage expanding alternately along different crystal planes, while the principal cleavage plane was the (1 1 1) crystal plane. The NM images of the microcrack proved that there was no sharp plastic deformation around the microcrack, and discrete edge dislocations could be found only near the crack tip.

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