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TEM and HRTEM Observations of Microstructural Change of Silicon Single Crystal Scratched under Very Small Loading Forces by AFM

Makoto Takagi[1], Kenji Onodera[2], Akihito Matsumuro[1],
Hiroyuki Iwata[3], Katsuhiro Sasaki[4] and Hiroyasu Saka[4]

[1]*Department of Mechanical Engineering, Aichi Institute of Technology, Toyota 470-0392, Japan*
[2]*Graduate School, Aichi Institute of Technology, Toyota 470-0392, Japan*
[3]*Department of Electrical and Electronics Engineering, Aichi Institute of Technology, Toyota 470-0392, Japan*
[4]*Department of Quantum Engineering, Nagoya University, Nagoya 464-8603, Japan*

The microstructural change of the surface and the subsurface regions of a Si single crystal (Si(100)) after scratching tests under very small loading forces was investigated. First, the scratching tests were carried out using an atomic force microscope (AFM). Then, the profiles of those wear traces which were generated by the scratching tests were observed using a transmission electron microscope (TEM). TEM observations revealed that dislocations were activated in the sub-surface within less than 100 nm depth from the surface of the wear traces when the loading force was higher than 5 µN. When the loading force was higher than 20 µN, patches of amorphous Si was observed occasionally at the surface of the wear traces. High-resolution TEM (HRTEM) observations revealed that a dislocation introduced by the scratching test was a total dislocation with Burgers vector of $1/2\langle 110\rangle$. [doi:10.2320/matertrans.MRA2008017]

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1. Introduction

Micro/nanotribology is one of the key technologies for the practical use of Micro/Nanoelectromechanical systems (MEMS/NEMS) or other engineering matters. The materials used for the parts of MEMS/NEMS are worn by the friction under very small loading forces, which makes the long run of the systems difficult. Hence, it is of academic as well as practical importance to study the microstructural change of the materials caused by the friction under very small loading forces. From the academic point of view, it contributes to clarifying the origin of the conventional friction and wear. From practical point of view, the microstructural change caused by the micro/nanofriction is to be correlated to the polishing process of silicon single crystals in the field of the semiconductors.[1)]

Recent developments of a scanning-probe microscope (SPM), especially an atomic force microscope (AFM), have provided new approaches for the studies on micro/nanotribology. Many investigations on the micro/nanotribological properties of various materials including hard thin films have been carried out by using these microscopes over the past two decades.[2–6)] In most of these investigations emphasis was placed on the change of surface morphology or the measurements of mechanical properties, but not on the microstructural changes of the subsurface materials underneath.

On the other hand, the development of a focused-ion beam (FIB) system provided a new technique for preparing specimens for the cross-sectional TEM observations of various materials. By using this preparation technique, the investigations on the cross-sectional TEM observations of the structural change of the materials caused by indentation or machining have been reported.[7,8)] However, only few experimental reports are available on the micro/nanotribological behavior, i.e., the microstructural change of the materials caused by micro/nanofriction.[9)]

The present authors have been studying the microstructural change of a Si single crystal caused by the micro/nanofriction; the preliminary results obtained by TEM observations have been reported.[10,11)] In the present study, the microstructural change of the Si surface after scratching tests under very small loading forces was investigated in detail by cross-sectional TEM and HRTEM observations in order to clarify the micro/nanotribological behavior. The effects of the loading forces on the microstructural change were studied systematically.

2. Experimental Procedure

At first, p-type Si single crystal (100) wafer (purity: 11 N) was carefully degreased in acetone and ultrasonically cleaned. Figure 1 shows the experimental method used to prepare a specimen for the cross-sectional TEM observation

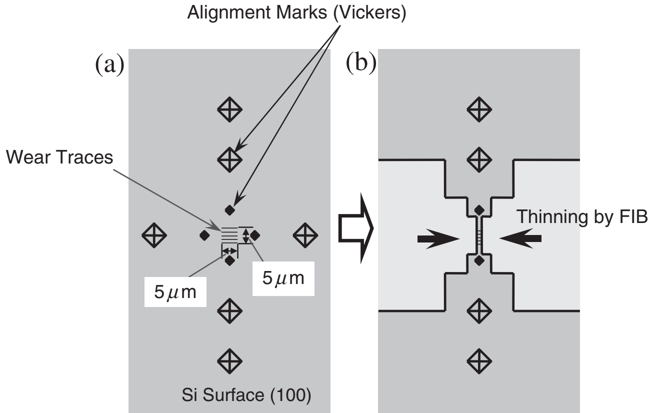

Fig. 1 Schematic illustration of the preparation method of a specimen for the cross-sectional TEM observation of the wear traces generated by the scratching tests under very small loading forces.

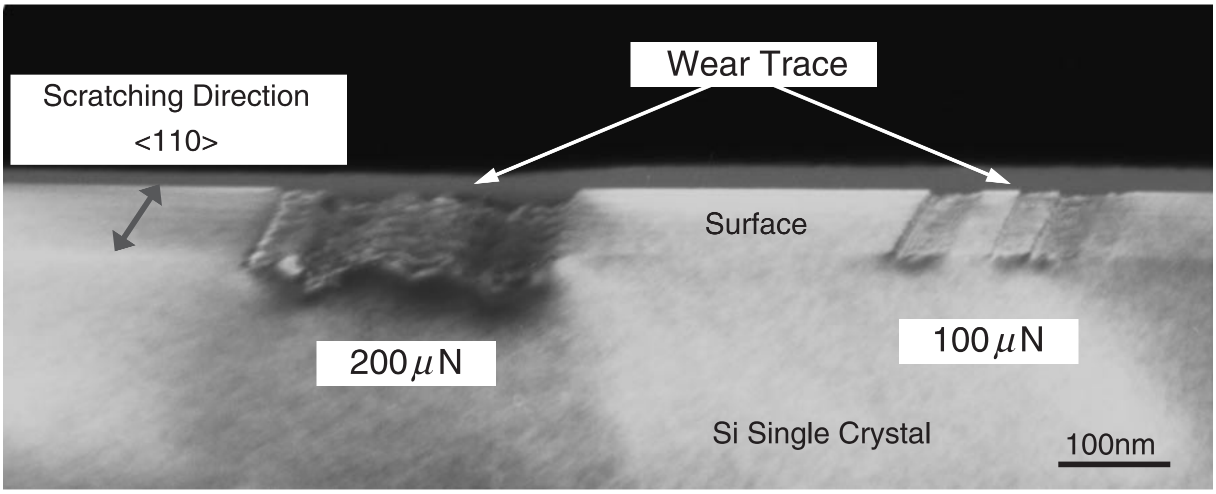

Fig. 2 TEM micrograph of wear traces generated by the scratching tests under loading forces of 100 μN and 200 μN. The wear traces are obliquely observed.

of the wear traces caused by scratching tests under very small loading forces. Before the scratching tests, a series of alignment markers with different sizes were printed on the Si surface by using a Vickers hardness tester to locate the area on which the scratching tests using AFM were carried out, as shown in Fig. 1(a). In the scratching test, a diamond tip was used as the counter material. The loading forces between the diamond tip and Si surface were changed over a range from 1 μN to 200 μN. The scratching direction of the tip was ⟨110⟩ on a Si(100) surface with a scratching velocity of 5 μm/s. The scratching length was 5 μm, and the wear traces were formed in the selected area (5 μm × 5 μm).

After the aforementioned scratching tests, the scratched Si surface was coated with carbon film in order to protect the surface structure from Ga ion radiation of FIB. The specimens for the cross-sectional TEM observations of the wear traces were prepared by using FIB as shown in Fig. 1(b).[12,13] The cross sections observed in this study were perpendicular to the scratching direction of ⟨110⟩. A Hitachi HU-1000D microscope (accelerating voltage; 1000 kV) and a JEOL2010 (accelerating voltage; 200 kV) were used in this study. HRTEM observations were carried out to identify the nature of a dislocation generated by the scratching test.

3. Results and Discussion

Figure 2 shows a TEM micrograph of the wear traces generated by the scratching tests under loading forces of 100 μN and 200 μN. In this figure, the wear traces are obliquely observed. The size of the wear trace generated by a loading force of 200 μN was larger than that generated by a loading force of 100 μN, but both of them have similar profiles.

Figure 3 shows scanning electron microscope (SEM) micrographs of the diamond tip of AFM used in the above-mentioned scratching tests. The top of the tip was not sharp, and it is therefore considered that the profiles of the wear traces shown in Fig. 2 were the replicas of the top of the diamond tip.

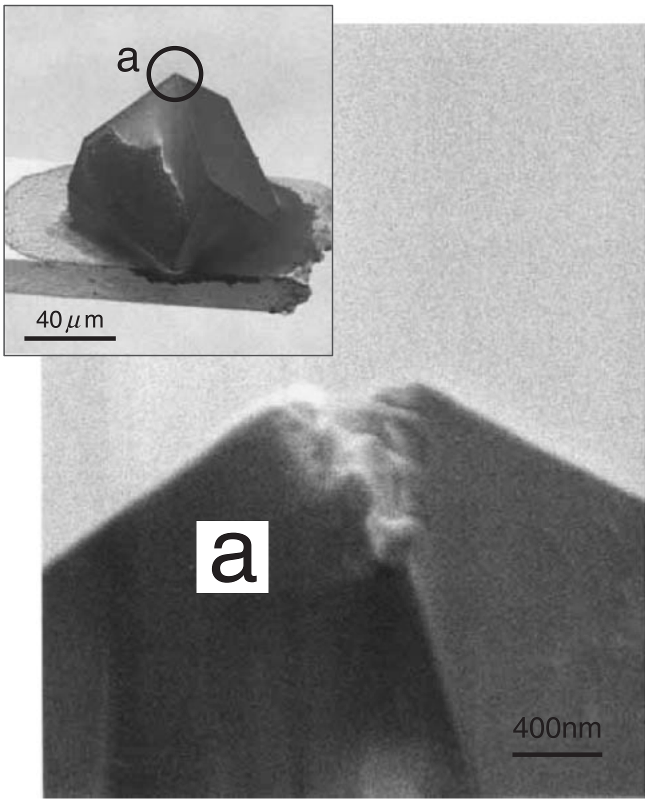

Fig. 3 SEM micrographs of a diamond tip of AFM used in this study.

Figure 4 shows a series of TEM cross sectional micrographs of the wear traces generated by scratching tests under loading forces ranging from 70 to 1 μN. When the loading force was higher than 5 μN, dislocations (indicated by arrows) were observed within the sub-surfaces of the wear traces. The dislocated region was not observed in the case of a loading force of 1 μN. When the loading force was higher than 20 μN, patches of area with uniform grey contrast were observed just underneath the wear traces. Again the size and number of such patches increased with the increasing loading force. A diffraction pattern from such a region is shown in Fig. 5(a): Obviously it shows halo rings characteristic of an

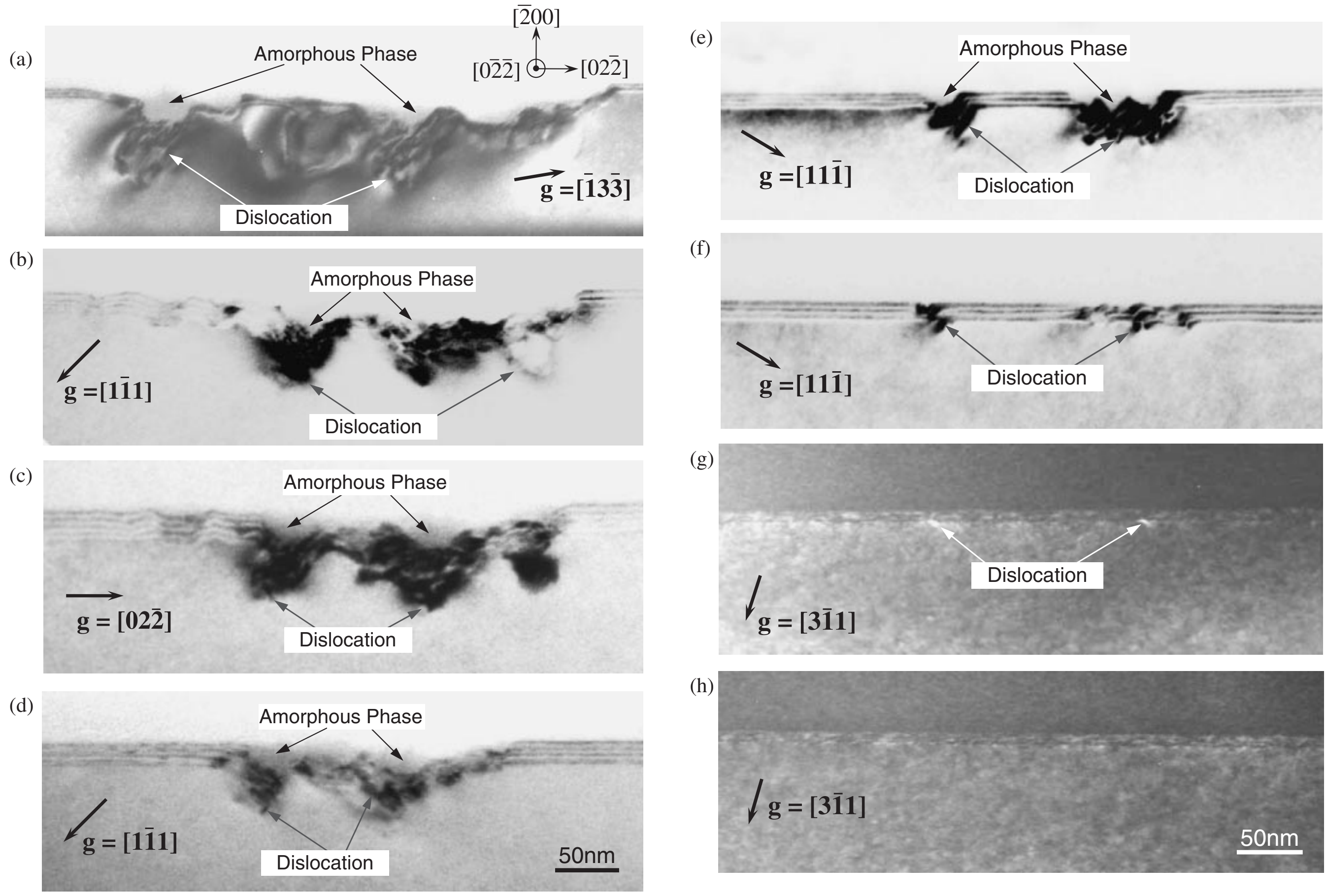

Fig. 4 TEM micrographs of the cross section of the wear traces generated by the scratching test under various loading forces of (a) 70 μN, (b) 50 μN, (c) 40 μN, (d) 30 μN, (e) 20 μN, (f) 10 μN, (g) 5 μN and (h) 1 μN. ((a)-(f): bright field images, (g),(h): dark field images).

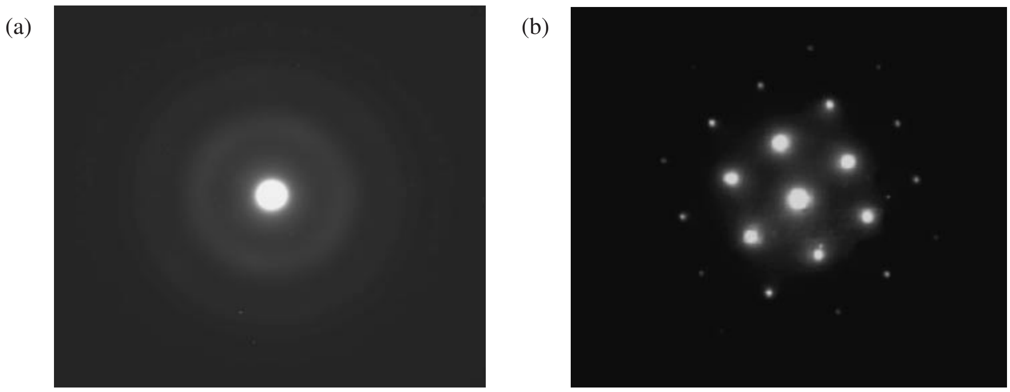

Fig. 5 Diffraction patterns of the cross section of wear traces in Fig. 4. (a) patches of area with uniform grey contrast (b) dislocated region.

amorphous phase. By contrast, the diffraction pattern from dislocated region shows a network of diffraction spots and it is evident that the single crystalline structure of Si was preserved (Fig. 5(b)).

One possibility is that the amorphous phase is oxides or other types of Si based compounds. Figure 6 shows the results of energy-dispersive X-ray spectroscopy (EDS) analyses in the dislocated region and the amorphous region. Silicon was detected in the both regions; this shows definitely that the amorphous phase under consideration resulted from amorphization of Si single crystal by the scratching test. Ga and C, which were slightly detected in the both regions, were from Ga ion of FIB process and carbon coat, respectively.

Figure 7 shows the effect of loading force on the cross-sectional area of amorphous region, which is estimated from the cross-sectional TEM images in Fig. 4. The cross-sectional area of amorphous region increased with increasing loading force, and the increasing rate was suppressed under the loading force higher than 50 μN. Figure 8 shows the effect of loading force on the depth of dislocated region. The depth of dislocated region increased monotonously with increasing loading force.

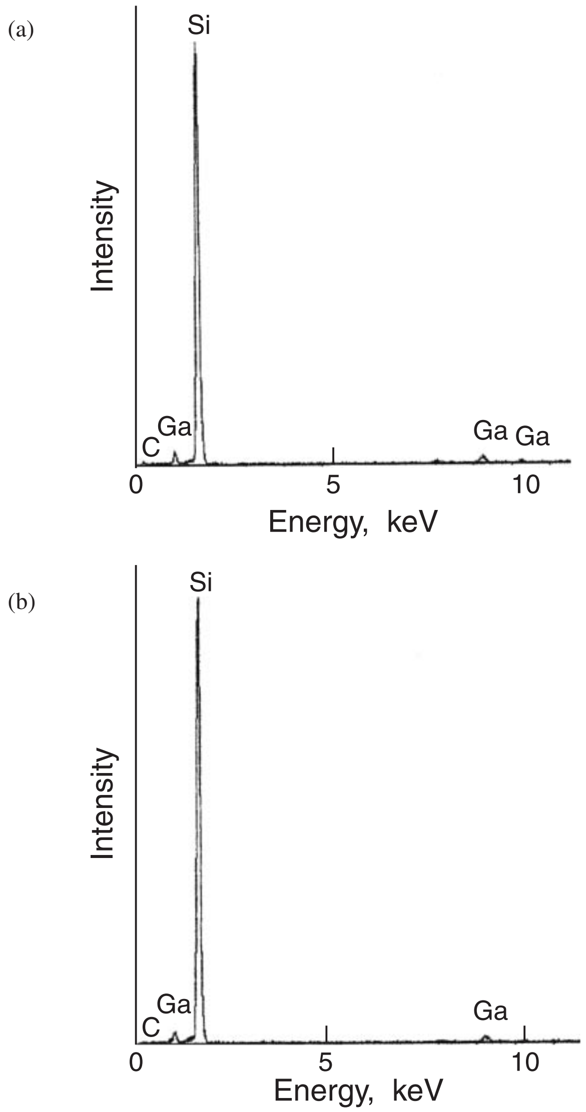

Fig. 6 Results of EDS analyses in amorphous region (a) and dislocated region (b).

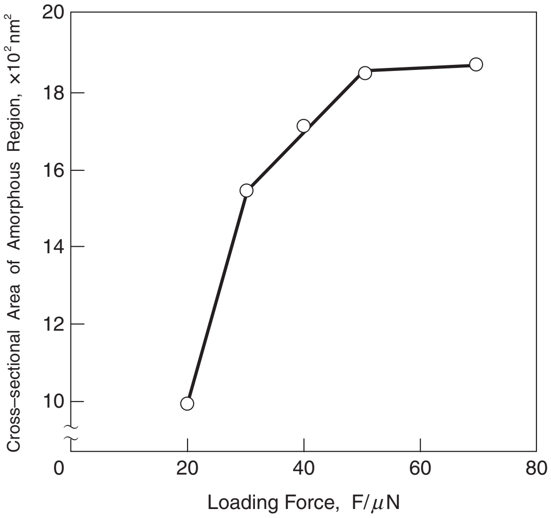

Fig. 7 Effect of loading force on the cross-sectional area of amorphous region. The cross-sectional area of amorphous region is estimated from the cross-sectional TEM images (in Fig. 4).

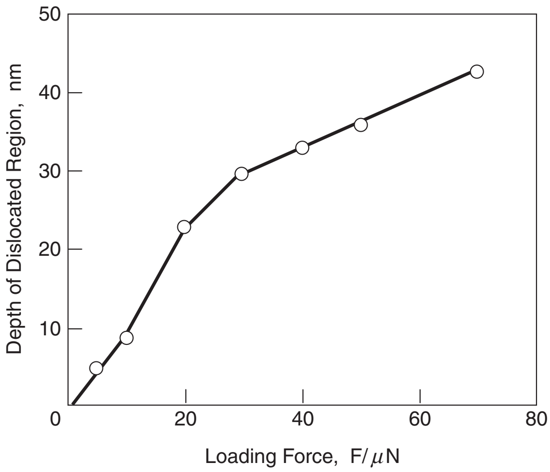

Fig. 8 Effect of loading force on the depth of dislocated region.

It was attempted to carry out contrast experiments to study the nature of the dislocations generated by the scratching test under very small loading forces. However, the dislocation segments introduced were so short and the internal stress in the dislocated region was so high that it was found very difficult, if not impossible, to determine the Burgers vectors of the dislocations by the conventional contrast experiments.

Therefore, HRTEM observation was carried out to study details of a dislocation generated by the scratching test. The results are shown in Fig. 9. Figure 9(a) shows a cross-sectional HRTEM micrograph of a dislocated area underneath the wear trace generated by the scratching test under a loading force of 40 μN. In the region A indicated by elongated circle, of which the depth from the top surface of the wear trace was about 25 nm, an atomic image of a dislocation was clearly observed as shown in Fig. 9(b). The dislocation lay on {111} plane, an easy slip plane of Si single crystal, and the Burgers vector of the dislocation was identified as 1/2[110] by the Burgers circuit construction. Furthermore, it is evident that the dislocation is not dissociated into partial dislocations.

4. Conclusions

Scratching tests under very small loading forces using AFM were carried out on the surface of Si single crystal (Si(100)), and the microstructural change of the cross section of the wear traces generated by the scratching tests were observed in detail using TEM and HRTEM. The following results were obtained.

(1) When the loading force was higher than 20 μN, patches of amorphous Si was observed occasionally at the surface of the wear traces. The cross-sectional area of the amorphous Si increased with increasing loading force.

(2) Dislocations were activated in the sub-surface within less than 100 nm depth from the surface of the wear traces when the loading force was higher than 5 μN. The depth of the dislocated region increased monotonously with increasing loading force. A dislocation introduced by the scratching test was a total dislocation laid on {111} plane with Burgers vector of $1/2\langle 110\rangle$.

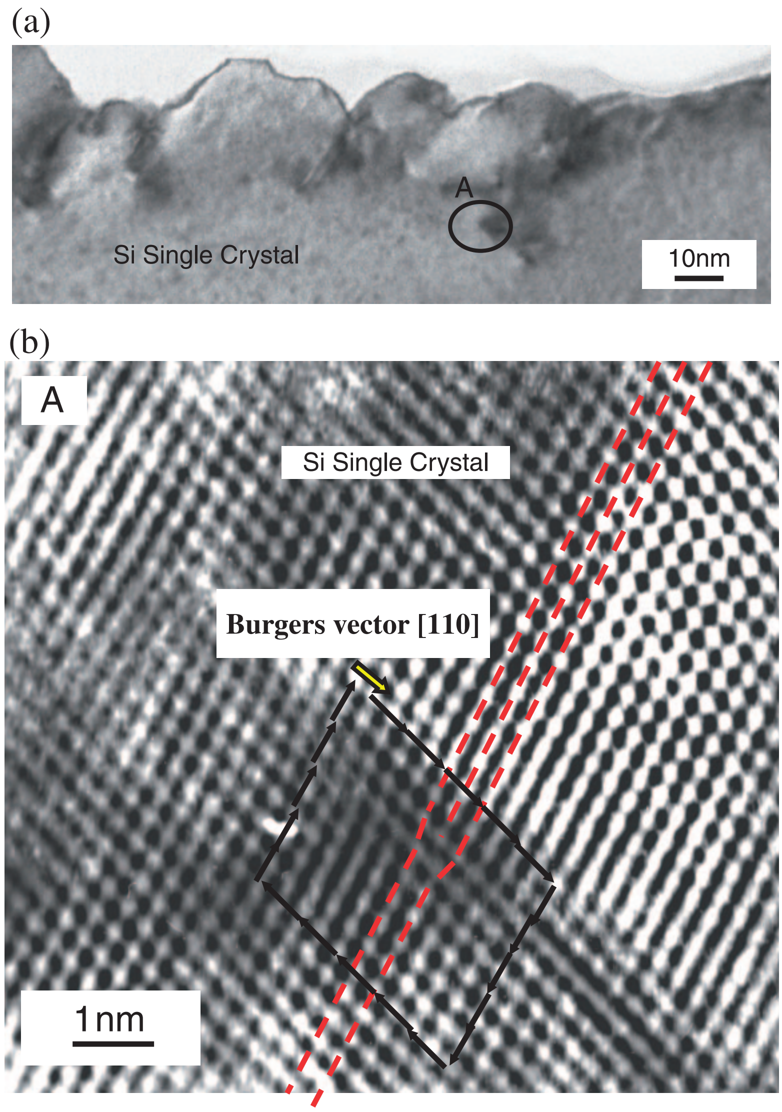

Fig. 9 HRTEM micrographs of the cross section of a part of the wear trace generated by the scratching test under a loading force of 40 μN. (a) the whole image (b) the image of region A.

(3) The depth and the width of the wear traces generated by the scratching tests under different small loading forces increased with increasing the loading force, and it is considered that the wear traces had a similar shape because of the replicas of AFM diamond tip.

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