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SPMによる引掻き試験でシリコン単結晶に生じた微構造変化のTEM観察
TEM Observation of Microstructural Change of Silicon Single Crystal Caused by Scratching Tests Using SPM

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Abstract　In this study, the microstructural change of the surface of Si single crystal (Si(100)) after the scratching tests under very small loading forces was investigated. At first, line-scratching tests and scanning-scratching tests were carried out using an atomic force/friction force microscope (AFM/FFM). Next, cross-sectional TEM observations of the wear marks which were generated by the scratching tests were carried out. As a result of the TEM observations after the line-scratching tests, it was found that dislocations were observed in the area of less than 100nm thickness from the surface of the wear marks which were formed under the loading forces of more than 5 μN. In the case of the loading forces of more than 20 μN, an amorphous region was also observed just under the wear marks. As a result of the TEM observations after the scanning-scratching tests, it was found that the introduction of dislocations took place and no amorphous region appeared. It was also found that the several atomic layers at the top surface of the wear marks shifted in parallel to (100).

1. INTRODUCTION

Nanotribology is one of the key technologies for the practical use of NEMS or another engineering applications. The developments of a scanning-probe microscope (SPM), especially, an atomic force microscope (AFM), provided the new approaches for the studies on micro/nanotribology. A lot of investigations on the micro/nanotribological properties of various materials including hard thin films using these microscopes have been conducted since around 1990 [1-4]. On the other hand, the investigations on TEM observation of structural change caused by indentation or machining have been reported [5-7]. However, few experimental reports on micro/nanotribological behavior, i.e., the microstructural change of materials caused by micro/nanofriction have been available so far.

The present authors have been studying on the microstructural change of the surface of Si single crystal after micro/nanofriction by using TEM, and the preliminary results obtained have been reported so far [8]. In the present study, the microstructural change of Si surface after scratching tests under very small loading forces was investigated by cross-sectional TEM observations in order to clarify the micro/nanotribological behavior. The effects of the loading forces on the microstructural change were also studied.

2. EXPERIMENTAL DETAILS

Line-scratching tests and scanning-scratching tests using an atomic force/friction force microscope (AFM/FFM) were carried out to investigate the micro/nanotribological behavior of Si single crystal. In both of the scratching tests, the loading forces of a diamond tip of the AFM / FFM were changed over a range from 1 μN to 200 μN. The scratching direction of the tip was <110> on Si(100) surface, and the scratching velocity was 5 μm/s. In the line-scratching tests, the scratching length was 10 μm. In the scanning-scratching tests, the area of 5 μm × 5 μm was worn by the scanning of 512 lines per cycle, and the number of the cycles was varied from 1 to 20.

After the above-mentioned scratching tests, cross-sectional TEM observations of wear marks generated by the scratching tests were carried out in order to study the microstructural changes of Si surface. The cross sections observed in this study were perpendicular to the scratching direction of <110>. Because the area and the depth of wear marks were very small, a new technique utilizing a focused-ion beam (FIB) system, which was made possible to prepare TEM specimens from a pre-selected region with a pin-pointing accuracy, was used to the preparation of TEM specimens in the present study [9,10].

3. RESULTS AND DISCUSSION

Line-scratching test

Figure 1 shows TEM micrographs of wear marks generated by line-scratching tests under loading forces of 100 μN and 200 μN. The size of the wear mark generated by a loading force of 200 μN was larger than that generated by a loading force of 100 μN, but both of the wear marks have similar morphology.

Figure 2 shows a TEM micrograph and diffraction patterns of the cross section of a wear mark generated by line-scratching test under a loading force of 50 μN. Some dislocations were observed in the crystalline area near the surface of the wear mark. An amorphous structure was also observed in the large deformation areas just under the wear mark. As a result of EDS analyses, it was found that Si single crystal

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was partly transformed to the amorphous structure.

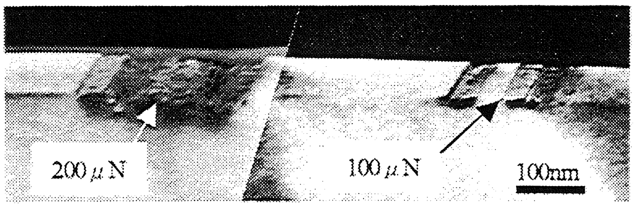

Fig. 1. TEM micrographs of wear marks generated by line-scratching tests under loading forces of 100 μN and 200 μN. (The wear marks are observed obliquely by TEM.)

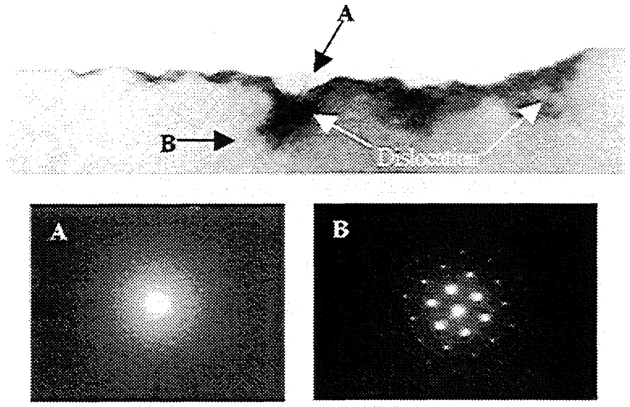

Fig. 2. TEM micrograph and diffraction patterns of the cross section of a wear mark generated by a line-scratching test under a loading force of 50 μN.

In order to study the nature of the dislocations generated by the scratching tests, cross-sectional TEM observations of a wear mark were conducted with different reflection vectors **g**. Some micrographs obtained by the TEM observations are shown in figure 3. Most of the dislocations observed were in the form of loops, and they lay on {111} planes, that is, easy slip planes of Si crystal. Burgers vectors **b** of the dislocations were determined to be $[\bar{1}10]$, [110], [101] and $[10\bar{1}]$.

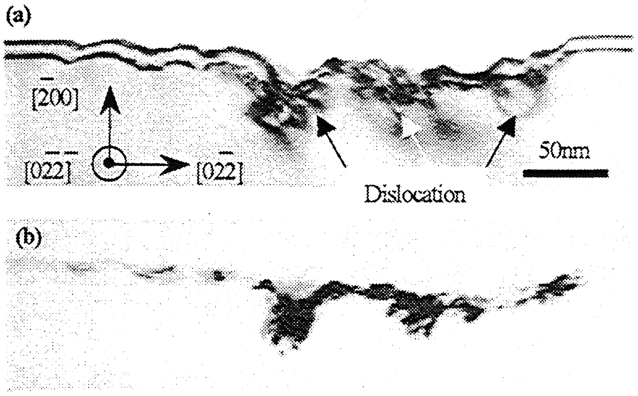

Fig. 3. TEM micrographs of the cross section of a wear mark generated by a line-scratching test under a loading force of 50 μN. (a) **g** = [111],. (b) **g**= [111]

Cross-sectional TEM observations of the wear marks generated by line-scratching tests under various loading forces were carried out to study the effect of loading force on microstructural change of Si single crystal. Figure 4 shows TEM micrographs obtained by these experiments. The depth and the width of the wear marks increased with increasing the loading force, and the shape of the wear marks was replicated that of the diamond tip of AFM (shown in figure 5). In the case of the loading forces of 5 μN and 10 μN, a few dislocations with small sizes were observed in the area of less than 20nm thickness from the surface of the wear marks. In the case of the loading forces of more than 20 μN, many dislocations in the form of loops were observed, and amorphous regions were also observed just under the wear marks. The size of the dislocations increased with increasing the loading force, and no dislocation was observed under a loading force of 1 μN.

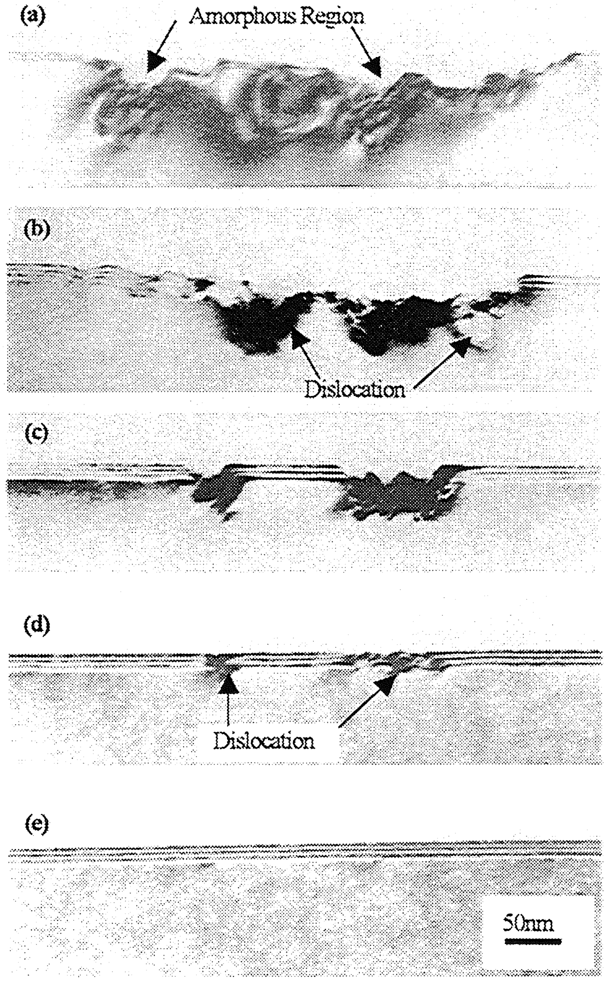

Fig. 4. TEM micrographs of the cross section of wear marks generated by line-scratching tests under various loading forces of (a)70 μN, (b)50 μN, (c)20 μN, (d)10 μN and (e)5 μN.

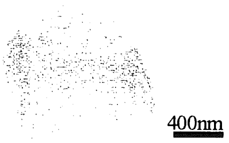

Fig. 5. SEM micrograph of a diamond tip of AFM.

Scanning-scratching test

Figure 6 (a) shows a TEM micrograph of the cross section of wear surface generated by scanning-scratching test under a loading force of 100 μN. This cross section observed is perpendicular to the scratching direction. Some dislocations reaching to the depth of about 50nm from the wear surface were observed clearly. These dislocations in the form of loops inclined at about 55° to the wear surface. Therefore, it is considered that the dislocations lie on {111} planes, that is, easy slip planes of Si crystal. In order to study the nanostructural change caused by micro/nanofriction, high resolutional TEM observation of the wear surface was carried out. Figure 6 (b) shows a HRTEM micrograph of the cross section of region **A** which has no well defined dislocation as shown in figure 6 (a). This micrograph exhibits that several atomic layers at the top surface of the scanning-scratching area shifted in parallel to surface (100). No amorphous region was observed in the case of the scanning-scratching tests (figure 6 (a)).

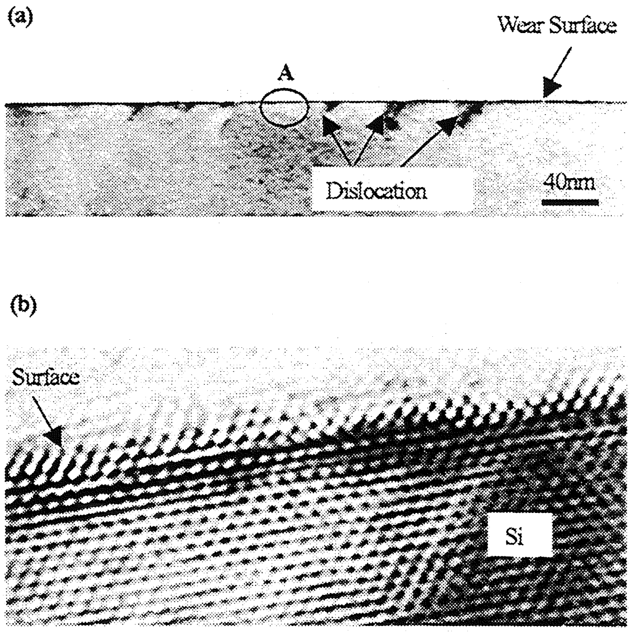

Fig. 6. (a) TEM micrograph of cross section of wear surface generated by scanning-scratching test under a loading force of 100 μN (3 cycles), (b) HRTEM micrograph of the cross section of region **A**. (Carbon film was formed for the protection of Si surface after the scratching test.)

4. CONCLUSIONS

(1) Dislocations are introduced by scratching tests on Si single crystal (Si(100)) under loading forces of more than 5 μN. The dislocations lie on {111} planes, that is, easy slip planes of Si crystal, and Burgers vectors **b** of the dislocations are [110], [110], [101] and [101].

(2) Si single crystal is partly transformed to the amorphous structure in the large deformation areas just under the wear marks which are generated by line-scratching tests.

(3) Several atomic layers at the top surface of scanning-scratching area shift in parallel to (100).

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