Microwear of Si Single Crystal

シリコン単結晶のマイクロ摩耗

Makoto Takagi†, Norikazu Arima††, Hiroyuki Iwata†††, Toru Imura†,
Katsuhiro Sasaki†††† and Hiroyasu Saka††††

高木　誠，有馬　則和，岩田　博之，井村　徹
佐々木　勝寛，坂　公恭

Abstract Microtribology of Silicon single crystals is one of the important factors for the practical use of MEMS. In this study, the effect of crystal orientation on microwear of Silicon single crystal and the wear structure were mainly investigated. Microfriction experiments using atomic force / friction force microscope (AFM / FFM) were carried out to investigate the effect of crystal orientation on the microwear depth of Silicon single crystals. In these experiments, the scanning-scratching directions of a tip of AFM / FFM were <100> and <110> on Si(100) surface and <112> on Si(111) surface. As a result, it was found that the depth of the wear marks generated on Silicon surfaces increased in the following order: <112>, <100>, <100>. Cross-sectional TEM observations of the microwear marks were carried out. As a result, it was found that the small dislocation loops were generated in the surface region at the first stage of the microwear, and the size and the number of dislocations increased with the progress of the microwear.

1. INTRODUCTION

Many researches and developments on MEMS (Microelectromechanical systems) have been carried out in recent years. Microtribology is one of the key technologies for the practical use of MEMS.

The developments of scanning-probe microscope (SPM), especially, atomic force microscope (AFM), provided the new approaches for the studies on microtribology. A lot of investigations on microtribological properties of various materials including hard thin films using these microscopes have been conducted since around 1990 [1-4]. Among these investigations, a few reports on the effects of crystal orientation and grain boundary on the microfrictional properties have been published [5]. However, no many reports on the effect of crystal orientation to microwear properties and on the microwear structures have been available so far.

In the present study, the effect of crystal orientation on microwear properties of Silicon (Si) single crystal and the microwear structures were mainly investigated. In order to study the microwear structures which were expected to result in the clarification of mechanism of microwear, cross-sectional TEM observations of the microwear marks generated by microwear tests using AFM were carried out.

†　愛知工業大学　工学部　機械工学科（豊田市）
††　愛知工業大学　大学院工学研究科（豊田市）
†††　愛知工業大学　工学部　電子工学科（豊田市）
††††　名古屋大学　大学院工学研究科（名古屋市）

2. EXPERIMENTAL DETAILS

Microwear properties of Si single crystals were studied by the scanning-scratch tests using AFM. In these microwear tests, two kinds of tips made of diamond and Si_3N_4 were used to generate the normal force of 100-200 and 1-5μN, respectively. The testing area, 5μm × 5μm in size, was scanned by 512 line-scratching of the tip and this operation was repeated to the range of 1-40 cycles. The scanning speeds of diamond and Si_3N_4 tips were 10μm/s and 5μm/s, respectively. After the scanning-scratch tests, surface morphology around the testing area of Si single crystal was measured using AFM with a normal force of 5nN. Two types of Si single crystals, that is, Si(100) and Si(111) wafers, were used to study the effect of crystal orientation on microwear properties. In these experiments, the scanning-scratching directions of the diamond and Si_3N_4 tips were <100> and <110> on Si(100) surface, and <112> on Si(111) surface.

After the above-mentioned microwear tests, cross-sectional TEM observations of microwear marks generated by the scanning-scratch tests were carried out in order to study the structural changes of Si surface. Because the area and the depth of microwear 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 [6,7].

3. RESULTS AND DISCUSSION

3.1 Microwear properties

Figure 1 shows effects of the scanning-scratching cycle and the normal force on wear depth. The wear depth of wear marks generated after microwear tests increased with increasing the scanning-scratching cycle and the normal force. At a large number of scanning-scratching cycles, the influence of normal force on wear depth appeared remarkably.

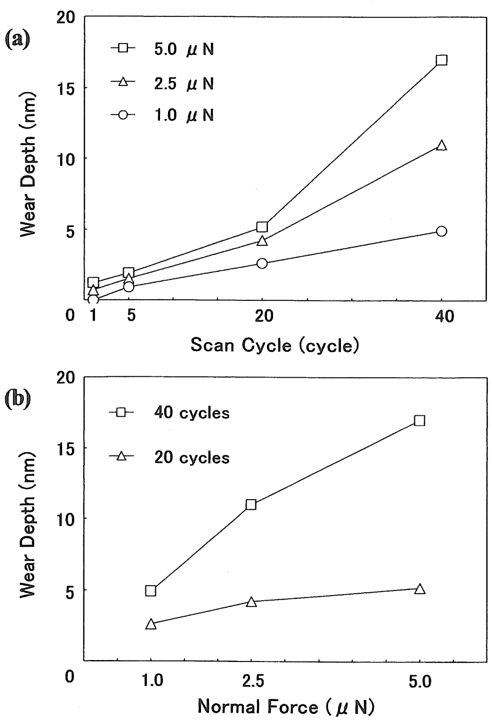

Figure.1 Effect of (a) scanning-scratching cycle and (b) normal force on wear depth.

AFM images of the wear marks produced on Si surface after different scanning-scratching cycles at normal force of 5μN are shown in figure 2 (cf. figure 1). In this figure, arrows indicate the scanning-scratching direction of <110> on Si(100). Wear marks appeared after 1 cycle of scanning-scratching, and the depth increased

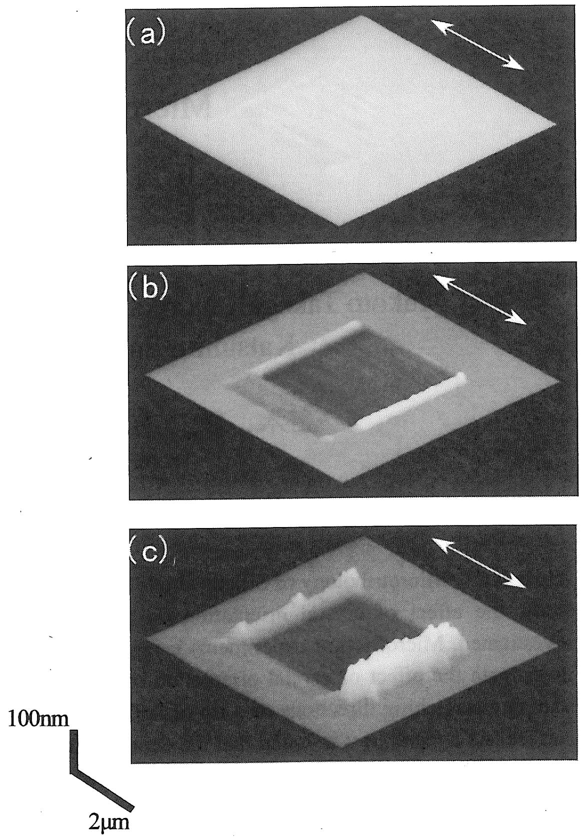

Figure.2 AFM images of the wear marks produced on Si surface at normal force of 5μN after various scanning-scratching cycles of (a) 1 scan cycle, (b) 20 scan cycles and (c) 40 scan cycles.

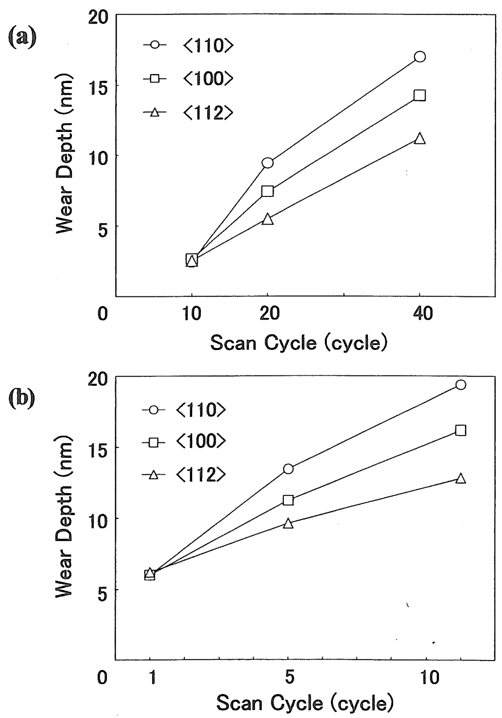

Figure.3 Wear depth vs. scanning-scratching cycle curves obtained by scanning-scratching of various crystal orientations at normal force of (a) 5μN and (b) 200μN.

apparently with increasing the scanning-scratching cycles. At the edges of wear marks, the risen areas generated perpendicular to the scanning-scratching direction, and the height of them increased with increasing scanning-scratching cycles, that is, with the progress of microwear. Because the morphology of the risen areas did not change by ultrasonic cleaning, it is considered that the risen areas were caused by plastic deformation or oxidation of Si single crystals, not by aggregation of wear powders. The dug volume of a wear mark was apparently larger than the risen volume at the edges of a wear mark. Therefore, it is thought that most of the dug volume of Si became wear powders and departed from the surface during the progress of microwear.

Effect of crystal orientation on microwear properties of Si single crystals was studied. Figures 3 (a) and (b) show wear depth vs. scanning-scratching cycle curves obtained by scanning-scratching process of various crystal orientations at normal force of 5 and 200μN, respectively.

The wear depth of wear marks generated by microwear tests at normal forces i.e. 5μN and 200μN depended on the crystal orientations, and increased in the following order: <112>, <100>, <110>.

3.2 Microwear structure

Cross-sectional TEM observations of the wear marks generated by the scanning-scratch tests were carried out in order to study the structural change of Si surface with the progress of microwear. Figure 4 shows TEM image of the cross section of a wear mark generated by 1 cycle of scanning-scratching at normal force of 100 μN. A carbon film covered with Si surface, shown in this micrograph, was prepared in order to protect the surface structure of the wear mark from Ga ion radiation of FIB. A lot of small dislocation loops, under 40nm in size, were observed at the surface of the wear mark. Figure 5 shows TEM image of the cross section of a wear mark generated by 20 cycles of scanning-scratching at normal force of 200 μN. Some dislocations reaching to the depth of 50-140nm from surface were observed clearly.

It is considered that the introduction of these dislocations was caused by the growth of small dislocation loops, as shown in figure 4, with the progress of microwear. A black-colored thin layer, about 40nm in thickness, was also observed at the top surface of the wear mark. Figure 6 shows TEM diffraction patterns of the black surface layer, the area near dislocations and the internal area of Si single crystal (cf. figure 5). These results indicate that the structure of Si single crystal was preserved, and that the black surface layer was Si single crystal containing a large number of lattice defects such as small dislocation loops. Based on the above-mentioned results of TEM observations, it can be said that small dislocation loops generated in the surface region at the first stage of microwear, and the size and the number of dislocations increased with the progress of microwear.

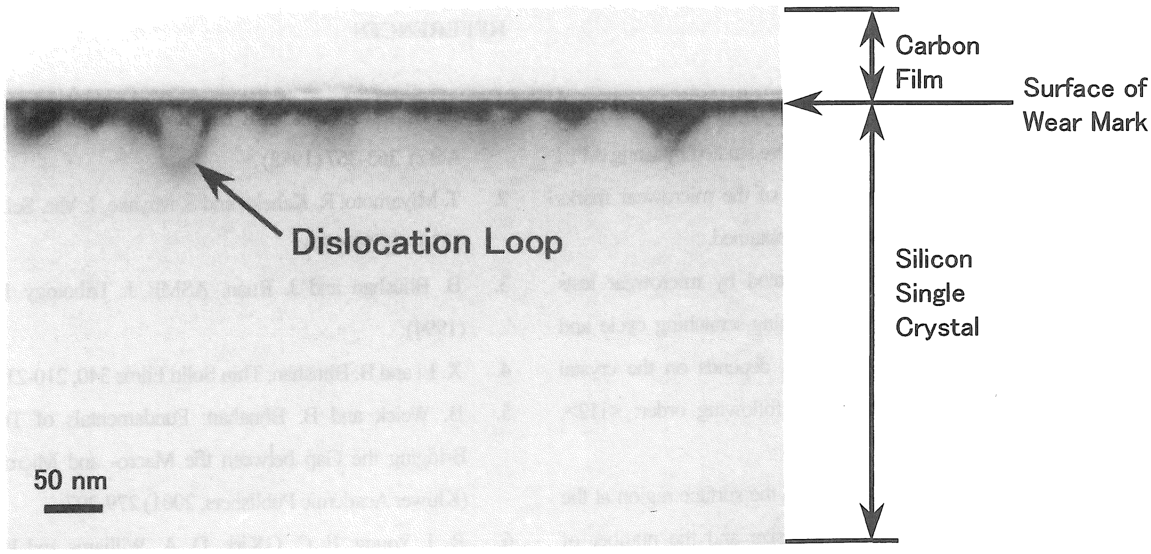

Figure.4 TEM image of the cross section of a wear mark generated by 1 cycle of scanning-scratching at normal force of 100 μN (scanning-scratching direction: <110> on Si(100)).

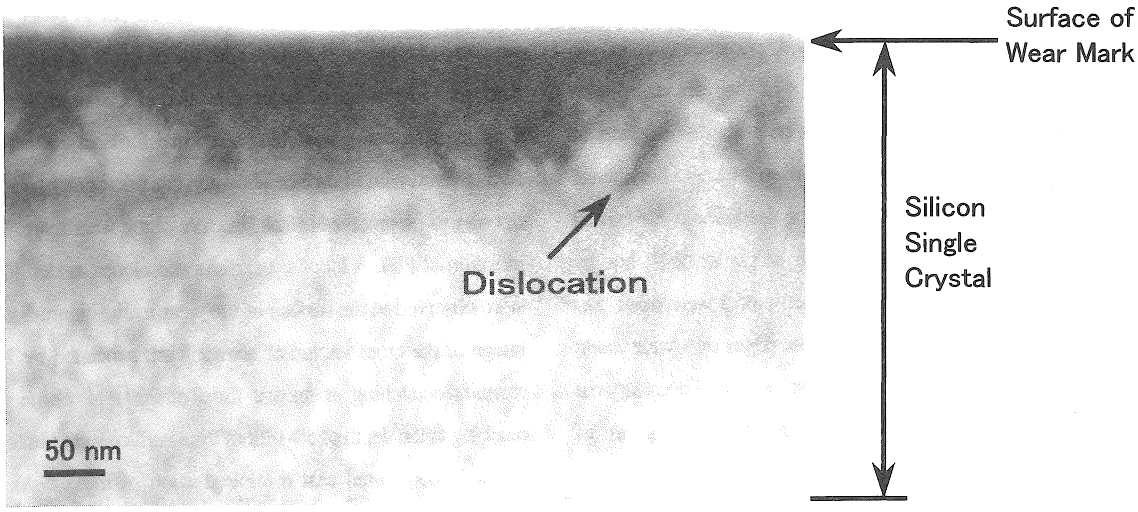

Figure.5 TEM image of the cross section of a wear mark generated by 20 cycles of scanning-scratching at normal force of 200 μ N (scanning-scratching direction: <110> on Si(100)).

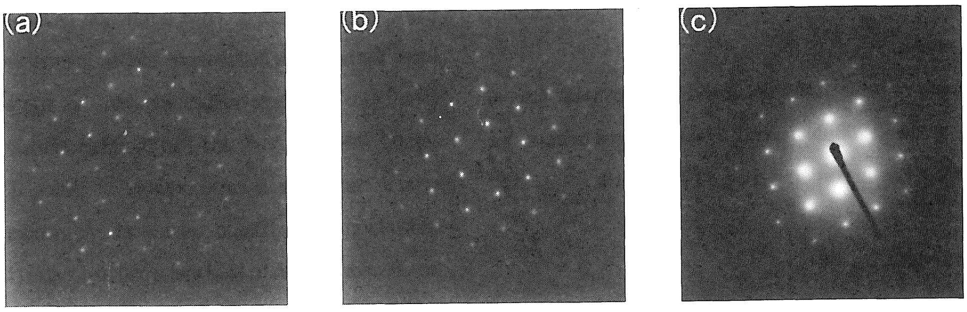

Figure.6 TEM diffraction patterns of (a) black surface layer, (b) area near dislocations and (c) internal area of Si single crystal (cf. figure 5).

4. CONCLUSIONS

Microwear properties of Silicon single crystal, especially, the effect of crystal orientation on microwear, were studied by using AFM and also cross-sectional TEM observations of the microwear marks were carried out. The following results were obtained.

(1) Wear depth of wear marks generated by microwear tests increases with increasing the scanning-scratching cycle and the normal force. The wear depth depends on the crystal orientations, and increases in the following order: <112>, <100>, <110>.

(2) Small dislocation loops generates in the surface region at the first stage of microwear, and the size and the number of dislocations increase with the progress of microwear.

REFERENCES

1. R. Kaneko, K. Nonaka and K. Yasuda, J. Vac. Sci. Technol. A6(2), 363-367 (1988).
2. T. Miyamoto, R. Kaneko and S. Miyake, J. Vac. Sci. Technol. B9, 1336 -1339(1991).
3. B. Bhushan and J. Ruan, ASME J. Tribology 116, 389-396 (1994).
4. X. Li and B. Bhushan, Thin Solid Films 340, 210-217 (1999).
5. B. Weick and B. Bhuahan: Fundamentals of Tribology and Bridging the Gap between the Macro- and Micro/Nanoscales. (Kluwer Academic Publishers, 2001) 279-297.
6. R. J. Young, E. C. G.Kirk, D. A. Williams and H. Ahmed in *Specimen Preparation for Transmission Electron Microscopy of Materials II*, edited by R.Anderson, (Mater. Res. Soc. Proc. 199, Pittsburgh, PA, 1990) .205-216.
7. T. Ishitani and T. Yaguchi, Microscopy Res. and Tech. 35, 320 -333(1996).

（受理　平成 15 年 4 月 30 日）