Etching Examination for the Scratched Wafer of Silicon Single Crystal

引っかき傷づけしたシリコン単結晶板の腐食法による研究

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Etching examination is made for the scratched wafer of silicon single crystal with Dash etchant. With the scratch without microcrack (load of 100 g), rapid eching is observed in the surface layer of the scratch, of the order of 0.3 μm in thickness, and this suggests that some defects are induced in the layer. With the scratches accompanied with microcracks (200 g and 300 g loads), the remarkable distortion in the surrounding of the scratch is removed by light etching and especially deep etching is observed in the microcrack. This give a evidence for the poor heading of the microcrack. Etch pits are observed in the etched furrow in front of the microcrack but it is difficult to relate the pit with dislocation. The elastic character of the distortion is due to the poor healing of microcracks.

§1 Introduction

Although it is known that no dislocation glides in the silicon crystal at room temperature, surface damage is produced by indenting and scratching. It arises a question how the damage is produced without dislocation glide. Some investigators concluded that the damages consisted of microcracks,$^{1,2)}$ while the others concluded that dislocations were formed in abrasion process in association with microcracks$^{3)}$ or without connection with microcracks.$^{4,5)}$

In the previous reports,$^{6,7)}$ the author investigated the distortion of the silicon crystal induced by scratching with various loads by means of microscopic and the divergent X-ray techniques, and made clear that the distortion was basically of elastic character. The aim of this work by etching technique is to examine whether the distortion due to scratching is related with dislocation generation or not.

§2 Experimental

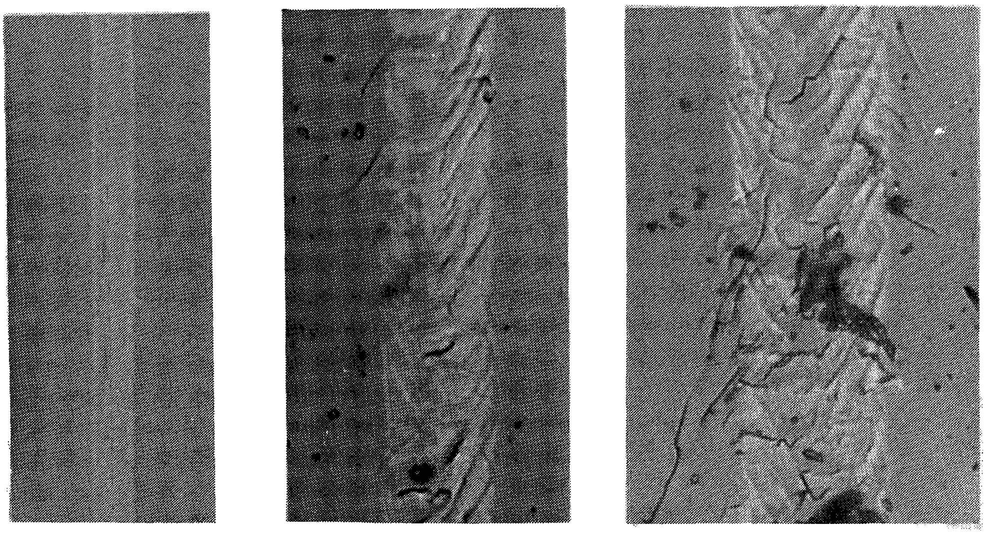
Specimen used in this experiment, are the silicon single crystal wafers of 0.3 mm in thickness with low dislocation density. The surfaces are parallel to (111) and free from defect. Scratching were made along the $[\bar{2}11]$ direction with the load ranging 75 g to 400 g. After microscopic and X-ray observations as in previous paper, the scratched wafers were etched for several hours with Dash etchant for revealing dislocation etch pits. At subsequent stages of etching progress, the specimen were subjected to microscopic and X-ray examinations. Furthermore, the micrographs of Cr-shadowed replica were taken for the etched wafer and the depth of etching was determined locally.

§3 Results and discussions

3-1. Distortion induced by scratching

In the previous paper,$^{6,7)}$ the distortion induced by scratching was investigated by means of microscopic and divergent X-ray techniques, and the some results relating to this experiment are described again below.

Typical aspects of scratches are shown in Figs. 1(a), 1(b) and 1(c), where (a), (b), and



(a) (b) (c)

Fig. 1. Typical aspects of the scratches under various loads. (x 500)

(c) correspond to loads of 100 g, 200 g and 300 g, respectively. Microcracks occured with load beyond a critical value (150 g), and the distortion in the surroundings of the scratch increased remarkably when microcracks occurred. With load less than a critical value (see Fig. 1(a)), no sink of wafer surface in the scratched trace was detected even by interference microscope but only minute strain was induced to the immediate surrounding of the scratch. With loads beyond the critical value, (see Figs. 1(b) and 1(c)), on the other hand, surface deformation was as sketched in Fig. 2.

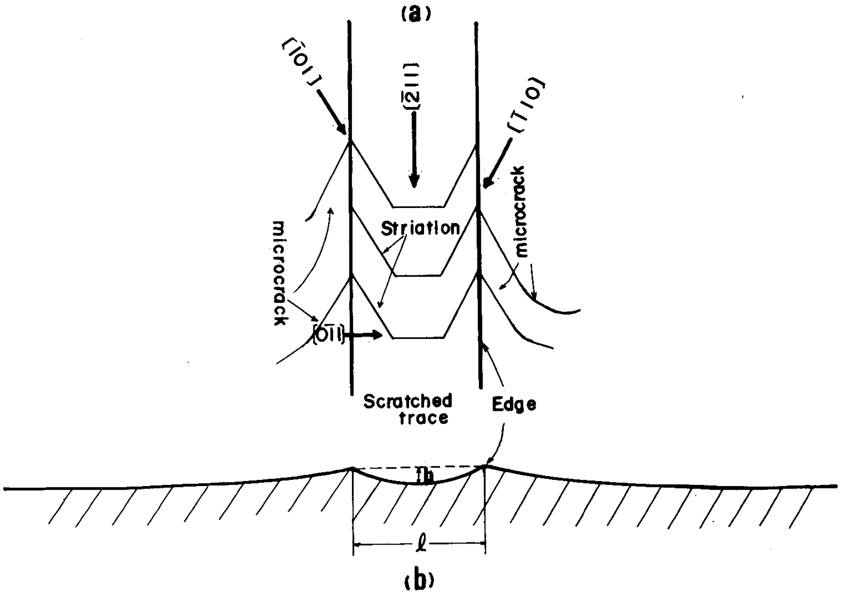


Fig. 2. Schematic representation of the surface distortion at and around the scratch accompanied with microcrack. (a) top view, (b) cross sectional view.

More detail aspects of the scratches are shown in the electron micrograph, Fig. 3, which was taken with the Cr-shadowed replica of scratched wafer. It was seen that the striation in the scratch was the shade of step in fact. Furthermore, misorientation of lattice planes was found to be several tens of minute of arc in the region near the scratch by the divergent X-ray observation.

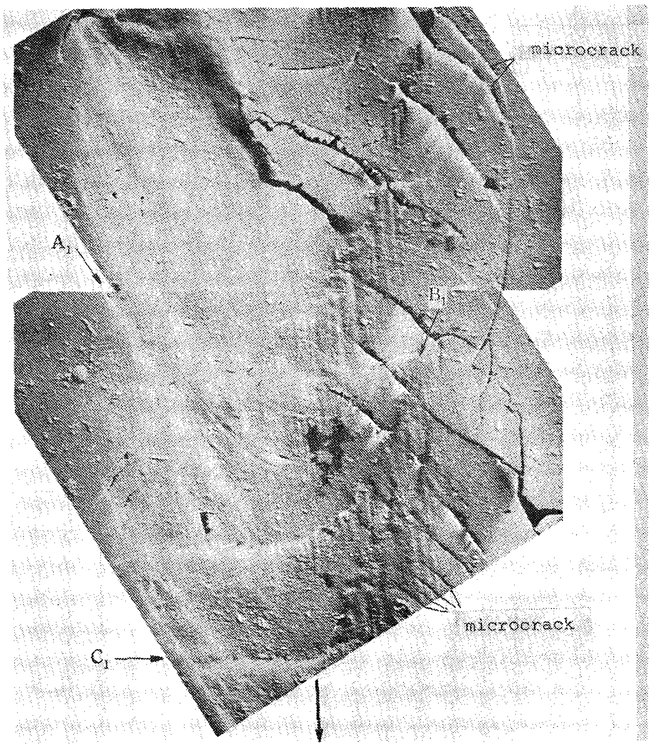


Fig. 3. Electron micrograph of replica corresponding to Fig. 1(b). (x 1500)

3-2. Etching effect for the distortion

Microscopic aspects of etching progress are shown in Figs. 4, 5 and 6 for the respective scratches of the loads of 100 g, 200 g and 300 g, where (a), (b) and (c) correspond to the etching, 1 min., 5 min. and 5 hours, respectively. Since the used etchant has low etching rate for the defect free surface and the dislocation etch pits become to be revealed by etching of several hours, we can guess the nature of defect from the aspects of etching progress at and around the scratches as shown in Figs. 4, 5 and 6.

With the load of 100 g (see Fig. 4), the interference microscope observation shows that the furrow of the trace of the order of 0.3 μm in depth is produced at the early stage of etching and no change in depth is found even at the final stage of etching. A few additional etch pit was found at the final stage of etching. However, the divergent X-ray observation showed that the reflection resulting from the distortion due to the scratching disappeared entirely at the early stage of etching. From these facts, it is seen that some disturbances of lattice planes are produced in the surface layer of the scratch, of the order of 0.3 μm in thickness, and are removed entirely at the early stage of etching. It is difficult that the origin of the disturbance is attributed to dislocation generation because the etching for the scratch progresses too rapid than the etching for revealing dislocation etch pits.

With the load of 200 g, etching progresses rapidly at and around the scratch and especially for the striation and microcrack as shown in Figs. 5(a), 5(b), and 5(c). Etching depth was determined by using the micrographs of the replica as shown in Figs. 7(a) and 7(b), where (a) and (b) correspond to the etching, 1 min. and 5 min., respectively.

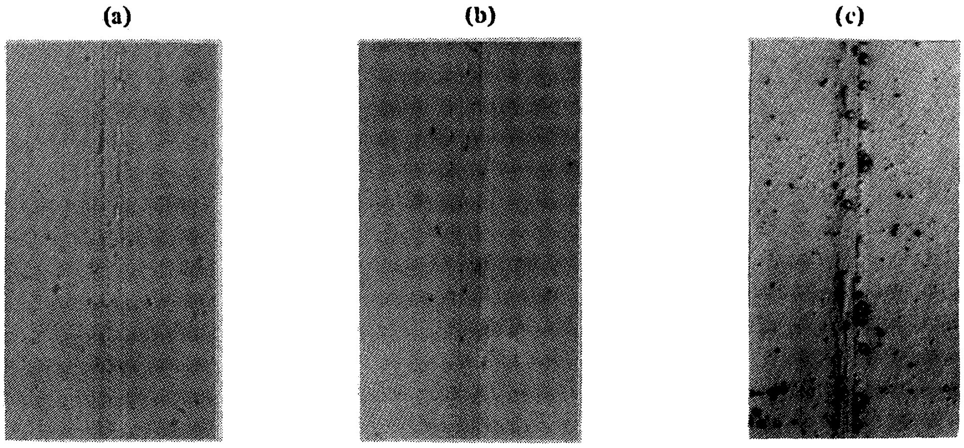


Fig. 4. The load of 100 g.

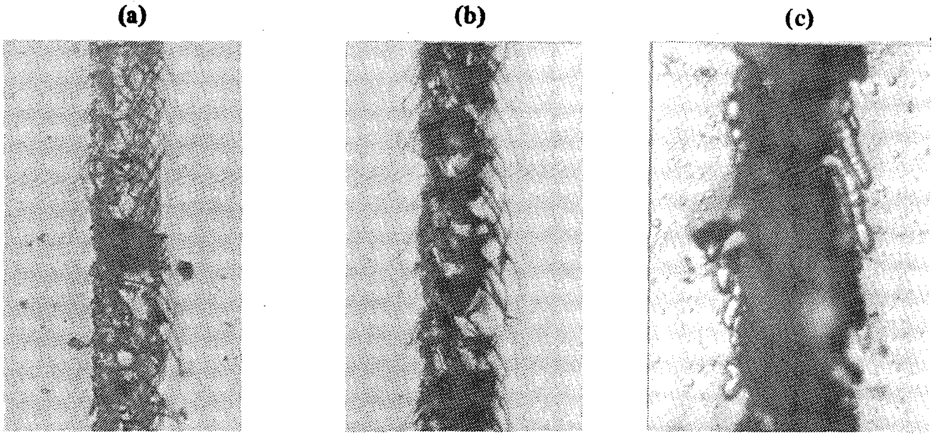


Fig. 5. The load of 200 g.

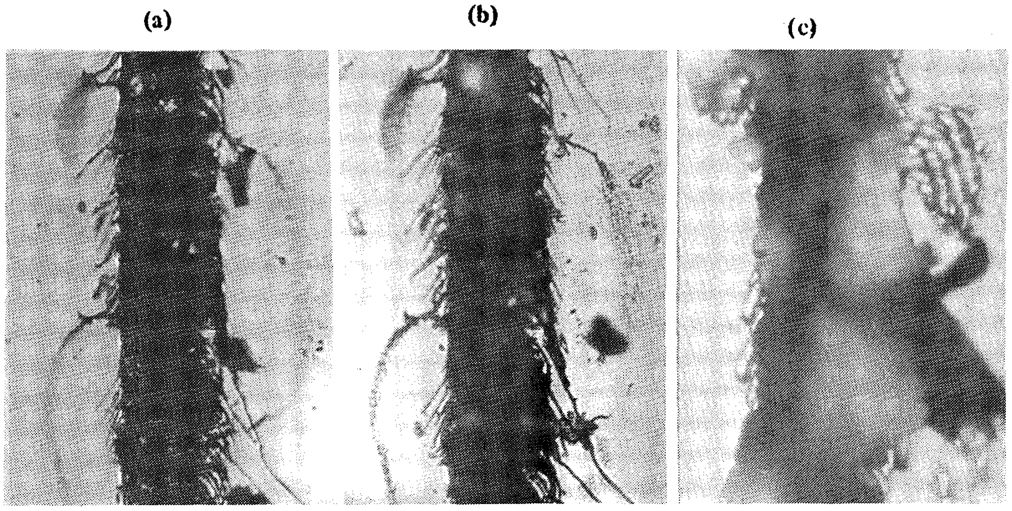


Fig. 6. The load of 300 g.

In Figs. 4, 5 and 6, (a), (b) and (c) correspond to the etching, 1 min., 5 min. and 5 hours, respectively. (x 300)

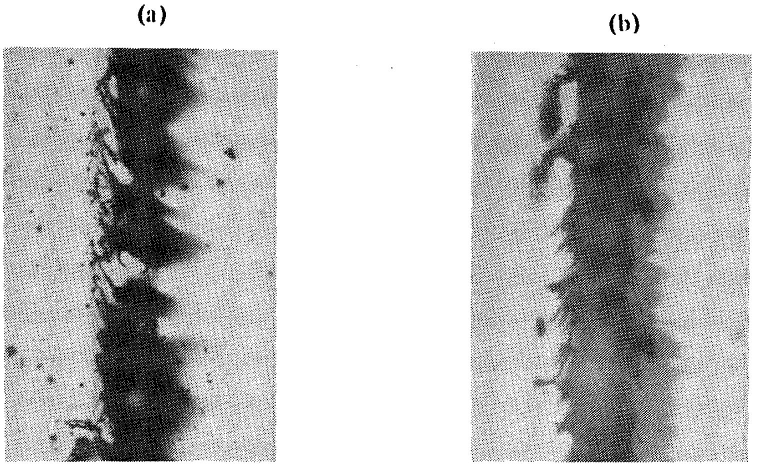


Fig. 7. Micrographs (a) and (b) of replicas correspond to (a) and (b) in Fig. 5, respectively. Cr. shadowing was made with the angle of 45°.

The etching for the striation in the scratch forms deep furrows at the early stage of etching as well as for the microcrack, and the depth reached 10–20 μm. This shows that the striation corresponds to the microcrack. The correspondence of the striation and the microcrack is supported by the fact that the striation are produced along the intersections of (111) wafer surface and $\{\bar{1}11\}$ primary cleavage planes as well as the microcracks. Furthermore, it is known that the internal faces of the microcrack are prevented from the complete healing by some obstacles and the etchant can easily penetrates between the interfaces.

At the final stage of etching, etch pits are observed in the narrow etched furrow in the front of microcrack, suggesting the possibility of dislocation generation. However, the divergent X-ray observation for the etched specimen showed that the reflections resulting from the distortion due to scratching disappeared at early stage of etching and no evidence for dislocation nucleation was obtained. Accordingly, it is difficult to relate the etch pits in the furrow with dislocations.

Now, the distortion due to scratching was removed entirely at the early stage of etching as described above. Furthermore, the surface deformation described in §3–1 is removed in early stage of etching as seen in Figs. 8(a) and 8(b), where (a) and (b) are the interference micrographs of the scrach before and after etching, 1 min., respectively. Since no evidence

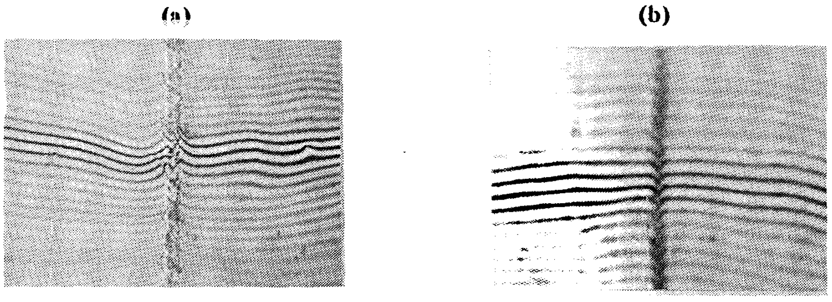


Fig. 8. Interference micrographs of the scratch before and after etching.

for dislocation generation was obtained, the distortion should be attributed to poor healing of microcracks, and this conclusion corresponds to the result obtained from the fracture experiment in the previous paper(7). The following is the reason why the distortion due to the scratching is removed at the early stage of the etching. The internal faces of microcrack are prevented from complete healing by some obstacles such as debris and cleavage steps, and the elastic residual distortion is induced in the surrounding of the microcrack. Thus, the remarkable distortion is formed at and around the scratch when microcracks occured. The etchant penetrates easily between the internal faces of the microcrack and the obstracles melt away, so that the distortion can be rapidly removed by light etching.

With the load of 300 g, the etching progresses rapidly than in the 200 g load scratching as shown in Figs. 6(a), 6(b) and 6(c). At the early stage of etching, the branchings from front of the microcrack are observed and the furrows of the branches are formed with etching progress. Etch pits array is observed in the furrow in Fig. 5(c) but this is no evidence for dislocation generation for the lack of the correlation of the etch pits in the furrow and dislocations as described above. The divergent X-ray observation showed that the distortion, with the load of 300 g, was removed incompletely even in the final stage of etching in contrast with the load of 200 g. The incomplete removal of the distortion by etching might be owing to the branching of microcracks.

In conclusion, the result from the etching experiment with Dash etchant are summarized as follows.

With the scratch without microcrack (100 g load scratch), rapid etching is observed in the surface layer of the scratch and the thickness was of the order of 0.3 μm. However, no evidence for dislocation generation was obtained by the X-ray observation. This suggests that some defects except dislocations are induced in the surface layer of the scratch.

With the scratches accompanied with microcrack, (200 g and 300 g load scratches), the remarkable distortion in the surrounding of the scratch was removed by lightly etching and especially deep etching was observed in microcrack. This means that the etchant can easily penetrate between the crack interfaces, and gives the evidence that the poor healing of the microcrack results from some obstacles such as debris and cleavage steps and elastic distortion is induced by microcracking. Since the obstacles are etched away soon by penetrating of etchant, rapid removal of the distortion in the surrounding of the scratch is appropriately explained if the distortion is atributed to the poor healing of microcracks. Etch pits were observed in the etched furrow in the front of microcrack. However, it is difficult to relate the etch pit in the furrow with dislocation by the divergent X-ray observation. Therefore, the elastic character of the distortion due to scratching by divergent X-ray observations(7) is understood from the character of microcracks as described above.

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