Establishment of 30mm diameter milling and curtaining effect reduction by large area planar surface milling

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Background incl. aims

The planar surface milling method is effective for sputtering a specimen surface by irradiating an Ar ion beam at an adjusted irradiation angle while the specimen is continuously rotated. This method is used for final finishing of mirror polished specimens by mechanical polishing for SEM observation and EBSD analysis. And the sputtering area is about 5 mm in diameter using the conventional planer surface milling method. However, there is an increasing demand for final finishing by planer surface milling over a larger area, such as a diameter of over 10 mm, where mechanical polishing is generally used. The followings are some of the issues with conventional planar surface milling.

Problem 1: Ion irradiation is performed from one direction at the periphery of the ion irradiation area. This causes a curtaining effect, making it difficult to obtain a flat surface over a large area.
Problem 2: Differences in etching rates are caused by differences in specimen material and crystal orientation, resulting in unevenness on the ion-irradiated surface1).

In this study, we examined methods to resolve these issues.

Methods

Specimen for this test is rolled aluminum foil (100 µm thickness) without mirror polishing or other pretreatments. This is because this specimen has uniform scratches in one direction generated during rolling process, and these scratches were evaluated as polishing scratches.

The Ar ion beam processing system IB-19530CP/IB-10500HMS (JEOL Ltd.), and IB-11550LSRH (JEOL Ltd.) as holders for planer surface milling were used for this study. By combining these units, the stage rotating and stage swinging during planer surface milling can be executed simultaneously. This enables milling over a large area. We named this method to large area planar surface milling. In this method, the center of rotation and the ion beam center are eccentrically aligned during the swing of the specimen stage, allowing ion beam irradiation of the outer periphery from various directions. Therefore, it can reduce the curtaining effect at the outer area of the ion irradiation area. In addition, the tilt angle of the planar surface milling holder is adjusted to make the ion beam irradiation angle lower to 10° or less, which can suppress the unevenness of the ion irradiated surface2).

Experiment 1: Verification of reducing the curtaining effect
Specimens were prepared by the planar milling method using a specimen rotation motion and by the large area planar milling method which combines the specimen rotation with the stage swing motion. Subsequently, the quality of surface was compared. Specimens were processed under the following conditions: an acceleration voltage of 10 kV, a processing time of 1 hour, and an ion beam irradiation angle of 5°. In the large area plane milling method, the stage swing angle was set to ±30°.

Experiment 2: Verification of unevenness reduction
Specimens were prepared using the large area planar surface milling method at ion irradiation angles of 5° and 2°, and the quality of surface was compared. Specimens were processed under the following conditions: an acceleration voltage of 10 kV, a stage swing angle of ±30°, and a processing time depended on irradiation angle of ion beam : 12 hours at an ion beam irradiation angle of 5° and 24 hours at an ion beam irradiation angle of 2°.
An FE-SEM (JSM-IT800 (JEOL Ltd.)) and an AFM (JSPM-5200 (JEOL Ltd.)) were used to observe each processed surface.

Results
1. comparison of the conventional method and large area planar surface milling method
The conventional method produces a flat surface in the vicinity of the milling center. However, striped structures can be seen on the surface about 5 mm away from the milling center due to the curtaining effect caused by the limited direction of ion beam irradiation. On the other hand, the large area planar surface milling method reduced the curtaining effect even at a distance of about 5 mm away from the milling center. (Fig. 1 A).

2. effect of irradiation angle on large area planar surface milling:
Unevenness due to differences in etching rate, such as crystal orientation, could be reduced by changing the ion beam incidence angle from 5° to 2°. In addition, the average surface roughness of the center of the processed area was measured using AFM. The average surface roughness was Ra: 86.7 nm at an ion beam irradiation angle of 5°, and Ra: 24.1 nm at an ion beam irradiation angle of 2°. This indicates that unevenness reduction was achieved even in the center of the processed area. The processing at an ion beam incidence angle of 2° extends the ion irradiation range, and thus reduces the curtaining effect over a wide area of 30 mm diameter (Fig. 1 B).

Conclusions

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We examined the reduction of unevenness and increase of the milling area by the planar surface milling. Using a new method that combines a large area planar surface milling method with an extremely low-irradiation angle ion beam, the unevenness caused by etching rate differences in materials or crystal orientation was reduced and a large range of specimen processing could be performed. This method is expected to be applied to specimens that are not easy to mechanically polish or as an alternative technique to buff polishing, because it can produce a fine surface with reduced unevenness over a wide area exceeding 20 mm in diameter.
In this presentation, application examples will be presented in addition to the above results.

Graphic:

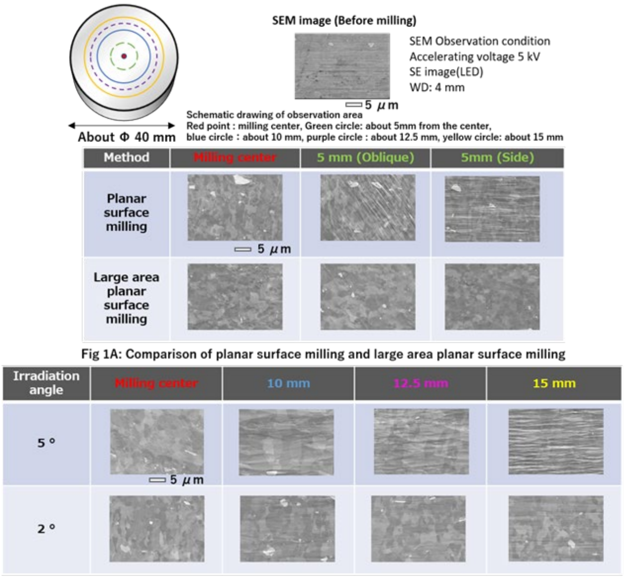

Fig 1A: Comparison of planar surface milling and large area planar surface milling

Fig 1B: Comparison of irradiation angle 5° and 2° by large area planar surface milling

Keywords:

Ion etching, Specimen preparation method

Reference:

1 R. Gago, et al., Applied Physics Letter, 78, 3316-3318(2001).
2 A.W. Barnard, et al, Microscopy and microanalysis, 12, 1318-1319 (2006).