Measurement of Vertical Section Geometry of Surface by Electron Microscope

Tadaaki MIYASAKA*

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I. Introduction

In the electron microscopic study on many kinds of surfaces, it is necessary to make not only the two-dimensional measurement but also the three-dimensional measurement.

For the three-dimensional measurement by electron microscope, two methods have been used for examining the geometry of the specimen surface. One is the stereo photogrammetric method developed by Haidenreich and Matheson[1]. The other is the oblique shadowing method developed by Seelinger[2]. The former has the advantage of simultaneously measuring both the two and the three-dimension in spite of the complicated calculations. Although the latter has the same advantage as the former, the influence of fragility of the shadowed boundary cannot be neglected from the view point of measurement accuracy.

It is necessary for us to measure the depth of the scratches of the stylus[3] on the examination of the metal surface. For this purpose, this paper describes a new method that is used to measure the vertical section geometry of the surface. And the vertical section geometry of the diffraction grating is measured.

II. Method

1. Procedures of making the two-stage replica of the original surface.

Using an acetylcellulose film, one-stage replicas of the original surface are obtained (**Fig. 1** (**b**)). Ethylcellulose films about 3 mm in thickness are beforehand made from a mixture of ethylcellulose and trichloroethylene. A few drops of trichloroethylene are dropped on these films, the acetylcellulose one-stage replica already obtained is mounted on them. These two jointed films are clipped by paper clip. Several minutes after the acetylcellulose film is stripped off, the ethylcellulose two-stage replica keeping the shape of the original surface is obtained (**Fig. 1** (**c**)).

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* Research Associate, Department of Precision Engineering, Faculty of Engineering, Shinshu University, Nagano City.

2. Procedures of embedding the two-stage replica and making its cross section.

The two-stage replica above mentioned is evaporated by Al so that the Al layer about 2,000 Å in thickness can be formed (**Fig. 1** (**d**)). The same evaporated replicas are pasted by epoxy resin, facing each other (**Fig. 1** (e)). Two jointed replicas are clipped by a paper clip for 24 hr at room temperature, and for 24 hr at a temperature of 40°C.

The specimen obtained above is polished perpendicularly to the embedded surface by a grinder (♯300) and emery papers (♯100, 240, 600) so as to reach the objective point of the embedded surface, and lastly is lapped for 5 min, with a 20 g/cm^2 of lapping load, using a lapping powder of CrO, by means of a 600 r. p. m rotating disk (**Fig. 1** (f)). Through the observation of the pasted layer coming in sight on the lapped surface by a 100 times optical microscope, the specimens clearly having gaps in the pasted boundary are thrown away. On the other hand, the specimens having the embedded Al layer and the lapped surface of good state are adopted for next procedures.

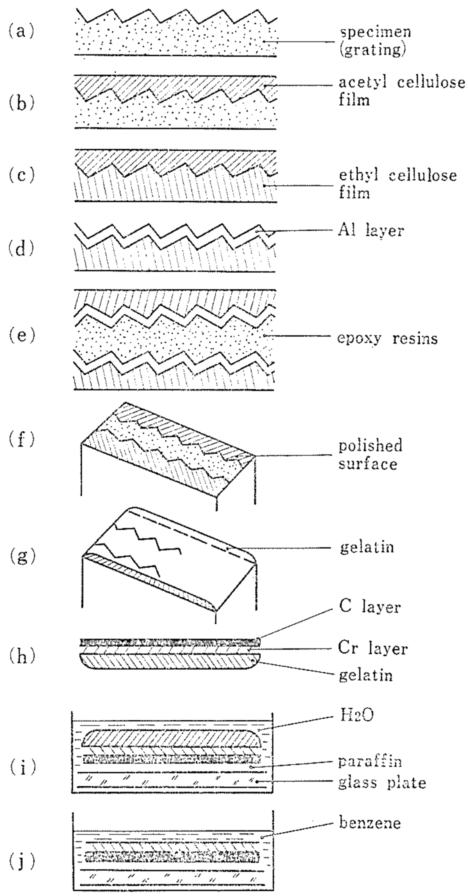


Fig. 1. The sequence of the technique to observe the vertical section geometry of the surface.

3. Procedures of etching the lapped surface and making two-stage replicas of this etched surface.

The lapped surface is steeped into a solution of 50 g hydrochroric acid in the 50 g water for about 3 min, and so the micro-slit will appear in place of the Al layer between the lapped epoxy resin and the ethylcellulose. The ethylcellulose side of this slit represents the vertical section geometry of the original surface.

In order to replicate the slit now appearing, we have coated the lapped surface with 20 % solution of pure gelatin warmed to about 30°C (**Fig. 1** (**g**)).

In this procedure, when it is desired to observe the special portion of the slit, the two-stage replica technique of the objective field[4] can be applied.

The obtained gelatin replica is shadowed by Cr at a shadow angle of 30°, and is coated vertically with C (**Fig. 1 (h)**). A paraffin having a melting point of 45°C is melted on a glass plate, and the solid paraffin of a 1.0–0.5 mm layer is made. The coated replica surface is placed on the solid paraffin, and is warmed up gradually until the coated replica surface touches the melted paraffin. After the paraffin has been hardened, the glass plate which fixes the replica is steeped into the distilled water and is warmed up gradually to about 35°C for about 30 min (**Fig. 1** (i)). The gelatin layer in sight is dissolved completely in the water, but a portion of the gelatin between the mesh and the Cr-C layer is not dissolved yet.

To obtain a good electron micrograph, the cleaning of the replica must be done, as described below. The glass plate improving the replica is steeped in the other distilled water. Furthermore the replica surface is washed down by means of water jets of a squirt in the distilled water. The specimen treated above is dried at room temperature, and then, as soon as it is steeped into the benzene warmed about 35°C (**Fig. 1** (**j**)), the paraffin is dissolved in it, and the Cr-C layer is freed from the paraffin floating in the benzene. It is now moved to another benzene warmed to about 35°C, and when the benzene is warmed up to about 45°C, the procedure of cleaning is completed.

The floating Cr-C layer in the benzene is ladled up on a mesh so that the replicated portion of the dissolved Al layer is in the mesh hole. It is the two-stage replica that makes it possible to observe the vertical section geometry of the original surface.

In the two lines which indicate the Al layer thickness in the electron micrograph, the line of the ethylcellulose side shows the vertical section geometry of the original surface.

III. Measurement and Results

From the procedures described above, the vertical section geometry of the diffraction grating that has a grating constant of 240 lines/mm is illustrated in **Fig. 2** (**a**). The black band in the center of **Fig. 2** (**a**) is the portion of the dissolved Al layer. The upper side of the band is the epoxy layer. On the other hand, the lower side is the ethylcellulose layer. In the two-toothed lines which indicate the Al layer thickness in **Fig. 2** (**a**), that of the under side shows the vertical section geometry of these grating grooves.

In order to indicate both the relationship of the vertical section geometry

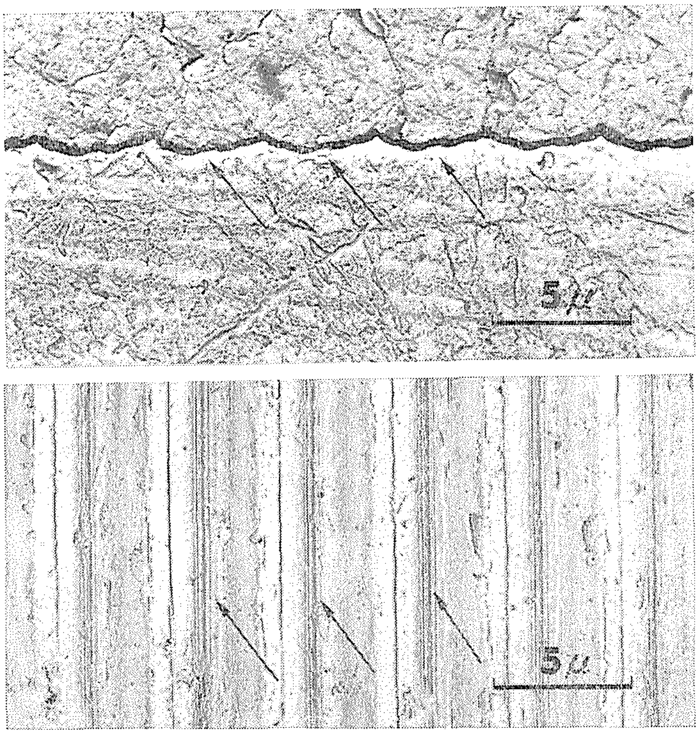


Fig. 2 (a). The vertical section geometry of diffraction grating grooves : The under edge of the black sawtooth line indicates it. X 5,500

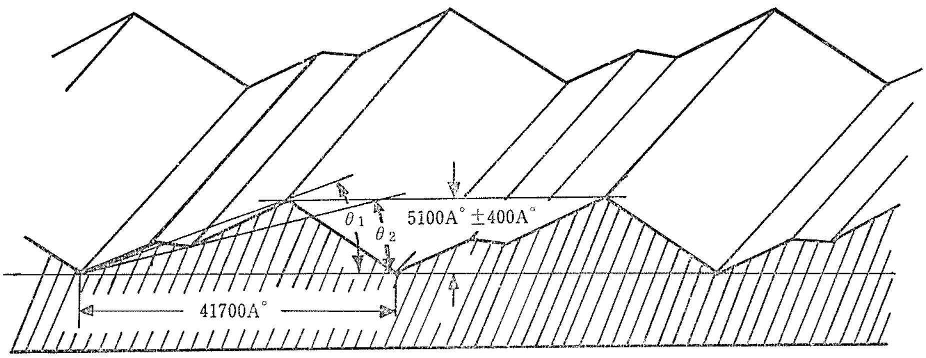
(b). The same grating surface view as Fig. 2 (a) : Both the three arrows indicate the same correspondences, which show the small mountains in the bottom of grooves. X 5,500

and the plane view, the palne view of the same grating as in Fig. 2 (a) is illustrated in Fig. 2 (b) with same magnification as in Fig. 2 (a). This is obtained by the filmy replica system[5] that is constructed on the acetylcellulose film and Cr-C layer. In this procedure, Cr is shadowed perpendicularly to the groove direction, and at the same time, at an angle of 45° with the grating surface.

The top and bottom of the grating grooves in Fig. 2 (a) have one to one correspondence to the grooves in Fig. 2 (b). There are small mountains, as indicated with three arrows, between the regular mountains in Fig. 2 (a), and these are recognized obviously as the characteristic streaks in Fig. 2 (b).

It is seen that the fine structure of the vertical section geometry of the grating grooves is kept in the procedure of this technique.

The measurements of the groove geometry are made directly from the



$\theta_1 = 11.0° \pm 0.3°, \theta_2 = 9.0° \pm 1.0°$

Fig. 3. The schematic geometry of grating grooves.

original film micrograph with help of a 50 times contour projector equipped with a protractor ring screen. The results are indicated schematically in **Fig. 3**. It is recognized that the results agree with those obtained from the shadowing method[6].

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