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# Observation of Defects in the Alpha-Alumina Single Crystal by TEM and REM Techniques

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# 투과전자현미경(TEM)과 반사전자현미경법(REM)을 이용한 알루미나 단결정내의 결함들에 관한 관찰

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## ABSTRACT

Transmission electron microscopy(TEM) technique which is useful for investigating the crystalline structure and defects, and reflection electron microscopy(REM) of which technique has very high sensitivity of detecting the defects on the crystal surfaces were applied to study the behaviour of dislocations and twins in or on the $\alpha$-$Al_2O_3$ single crystals.

## 요 약

결정 구조와 결정 내부의 결함들을 연구하는데 매우 유용한 투과전자현미경법(TEM)과 결정 표면에 존재하는 결함들을 발견하는데 있어 매우 민감한 인지력이 있는 반사현미경법(REM)을 이용하여 알루미나($\alpha$-$Al_2O_3$) 단결정내 또는 표면에 존재하는 전위(dislocations)와 쌍정(twins)들의 거동에 대하여 연구하였다.

## 1. INTRODUCTION

The excellent properties of $\alpha$-$Al_2O_3$ such as mechanical hardness, chemical stability, a high melting point, a low electrical conductivity lead to the many industrial applications. The studies of $\alpha$-$Al_2O_3$ have been well established by many researchers and the amount is also plenty as much as its history[1-3]. The crystallographic and electron diffraction data were also well collected[4].

In recent years, the studies on $\alpha$-$Al_2O_3$ have been substantially reduced because most of the studies were only focused on the new and advanced materials. Furthermore, defect studies on or in the $\alpha$-$Al_2O_3$ by using electron microscopy are only a few[5]. If any, most of the studies were done in the past. Especially, few electron microscopic scale observations have been made since rhombohedral twinning had been observed in a macroscopic observation with the compression test specimens at the elevated temperatures[6,7]. Therefore, the studies on the defect structure need to be re-elucidated and to be investigated continuously.

This paper presents the observation of dislocations and twins of $\alpha$-$Al_2O_3$ with the transmission electron microscopy( TEM) and the reflection electron microscopy( REM) techniques.

## 2. EXPERIMENTAL PROCEDURES

Pellet type $\alpha$-$Al_2O_3$ single crystals grown by Verneuil Method were used as samples. The dimensions of specimens were approximately 3mm in diameter and 10mm in length. Both ends of specimen rods were polished until optically flat. The compressive test specimens were oriented with the c-axis coinciding with compression axis to within $\pm 2°$. Compression tests were done on a universal testing machine in air up to 1373K with a 500kg capacity load cell. Crosshead speeds from 0.005 to 0.5mm/min. were used. Test were performed until load drops and failure occurs.

After the compression test, specimens for TEM observation were cut by 1mm thick perpendicular to the c-axis, then mechanically polished and dimpled to obtain a thin area of about 20$\mu$m in thickness at the center of 3mm diameter. The final specimen are obtained by ion thinning at liquid nitrogen temperature. All of the TEM specimens were coated with carbon to prevent charging effect. Specimens for REM observation were polished until no scratch was found under the optical microscope and then cut into pieces approximately $1 \times 1 \times 0.5mm^3$ size with a diamond saw or razor blade along the (0001), $\{1\bar{1}02\}$, and $\{11\bar{2}0\}$ planes. Annealing was performed at 1400℃ for 24 hrs in an electric furnace in air for recovery of surfaces from the damage caused by polishing. Both the TEM and the REM observations were carried out on JEOL-2000EX electron microscope at 200kV and 100kV, respectively. Particulars relevant to REM observation of $\alpha$-$Al_2O_3$ have been published elsewhere[8].

## 3. EXPERIMENTAL OBSERVATIONS AND DISCUSSION

### 3.1. Dislocation

Only a few dislocation have been found in all specimen. However, if the dislocations are present, they are mostly in-line conformation. These in-line dislocations had often been observed in the low or high angle boundaries[5]. In our observation, conformation and distribution of dislocations are unique and differ from that of low angle boundary. There is no boundary around dislocations and they are all isolated by a few hundred nanometers distance as shown in Fig. 1a. Barber et al.[9] reported that in no case were dislocations found to be associated with the tips of cracks, nor could they be introduced by beam stresses. In our observation, dislocations introduced by the electron beam were also not found; however, crack tips were, thought to be initiated from the region where in-line dislocations piled up in some

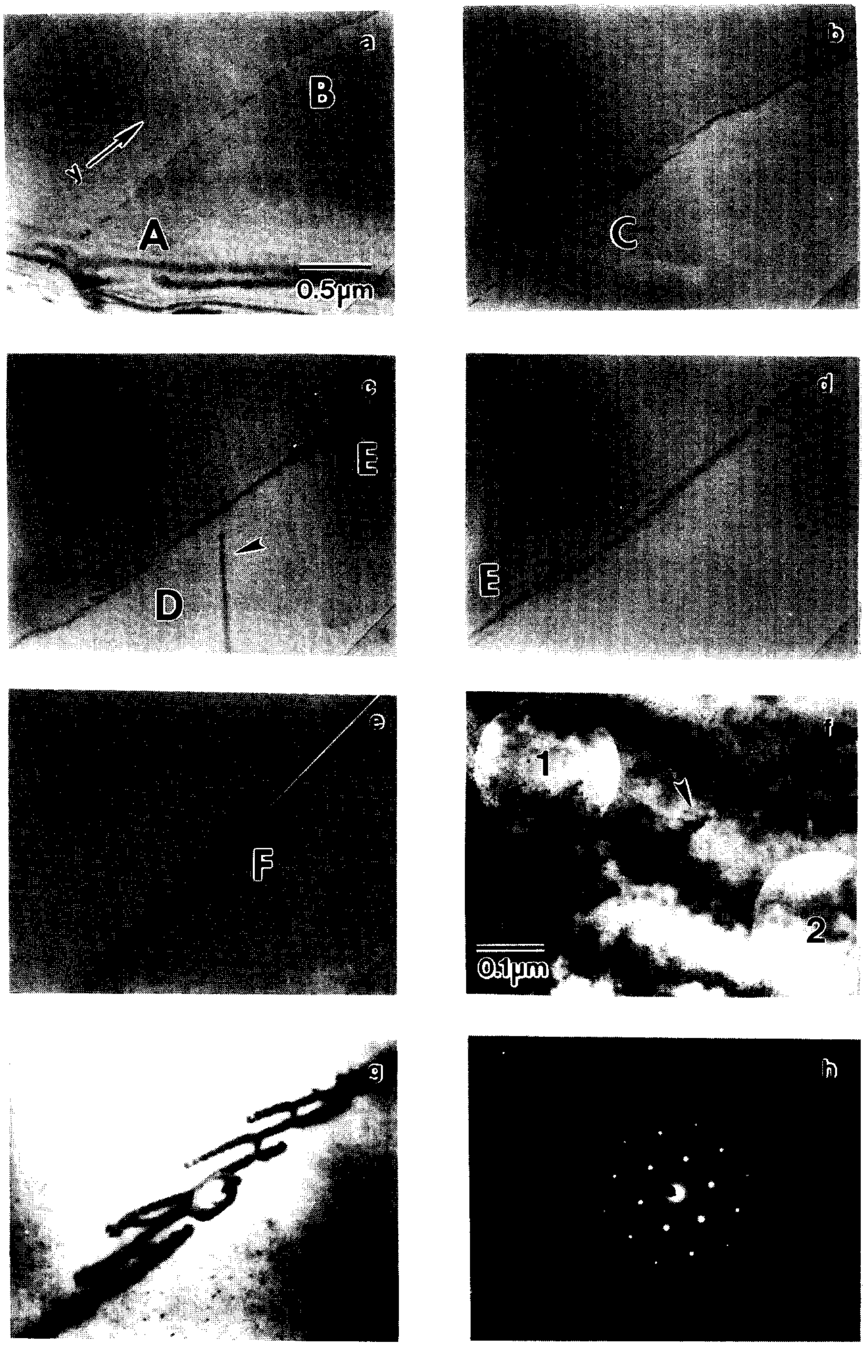



**Fig. 1.** The conformation of in-line dislocations near the crack front.
(a) Isolated dislocations, (b) overlapped dislocations, (c) overlapped and tangled dislocations, (d) tangled dislocations, (e) a crack front, (f) an enlarged image of region B in Fig. la, (g) an enlarged image of region E in Fig. 1c and 1d, (h) SADP from the region in Fig. 1f.

cases. In Fig. 1a, isolated dislocations were separated by a few hundred nanometers in the region A; the length of dislocations increased as y(distance from the first dislocation in the direction of arrow shown in the figure) increased and the distance between the dislocations decreased as y increased in the region B; the length of dislocations continuously increased as y increased and dislocations were no more in-line but overlapped in the region C of Fig. 1b; several dislocations were overlapped and tangled in the region D in Fig. 1c and E in the Fig. 1d, respectively; tangled dislocations initiated a micro-crack in the region F of Fig. 1e. Fig. 1f and 1g show the enlarged images of region B and E, respectively.

As shown in these figures, most of the dislocations were directed into nearly one direction (electron diffraction analyses obtained for these dislocations have shown them to have $1/3[11\bar{2}0]$ Burgers vectors); however, some of the dislocations indicated by arrows in Fig. 1c and Fig. 1f are not only out of line but also isolated. No diffraction pattern has been changed across the in-line dislocations. The white circles 1 and 2 in Fig. 1f indicate the area where selected area diffraction pattern (SADP) obtained as shown in Fig. 1h. From the series of pictures in Fig. 1, dislocations moved in the $[1\bar{1}00]$ direction and concentrated in the crack tip region. When critical stress was obtained in the dislocation concentrated region, dislocations interacted then initiated a micro-crack.

In REM observations, only few dislocations have also been found throughout the entire specimen surfaces. Unfortunately, in-line dislocations have not been found on the basal surface; however, isolated single edge-dislocation has been found as shown in Fig. 2. Strong black and white strain fields are shown in the right and the left, respectively[10]. The region between these strain fields is the edge-dislocation core because this dislocation does not accompany any atomic step[11].

Except some areas, most of the areas of (0001) basal plane show regularly spaced steps without particular defects as shown in Fig. 3a. The step edges looks running straight parallel to the horizontal line. However, the step edges are not really straight but fluctuated. The reason these step edges look straight in the REM image is that the foreshortening effect of REM image in the vertical direction substantially reduced the appearance of the deviation of step edges [12]. Fig. 3b shows a plane view of (0001) plane which has regularly spaced steps but not in straight manner.

The terrace width in Fig. 3a is about 0.3$\mu$m and step height is unknown. It is believed that the height is only a few atoms high from the image contrast and the our previous experiences[13].

Micro-cracks without a dislocation have also been found. A typical micro-crack in the basal plane is shown in Fig. 4a. Discontinuous bend contour fringes around the micro-crack could be seen[9].

In some areas(Fig. 4b), dislocation networks were formed, but no crack around them was found. Wiederhors et al.[14] reported the networks of dislocations may be formed by spontaneously healed crack.

### 3.2. Twinning

Krongberg[1] has concluded that the deformation in his observation was not slip, but twinning. Barber et al.[9] also reported the only mechanism allowing rapid deformation at nor-

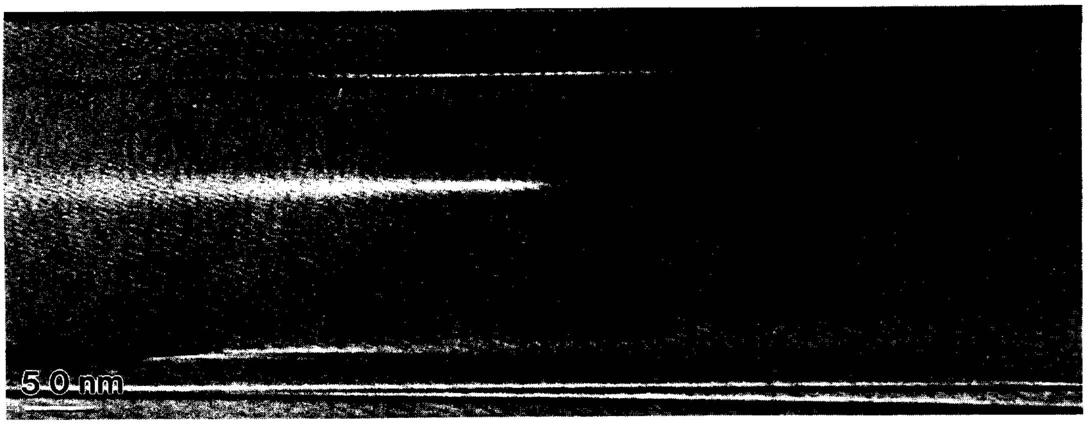
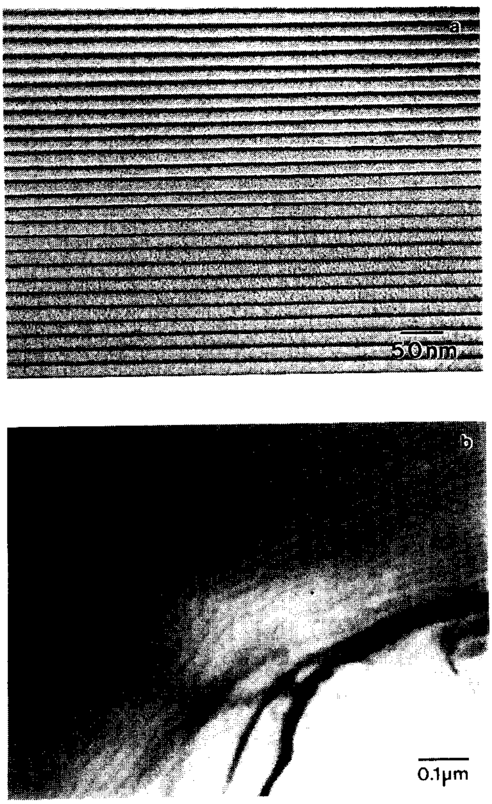



Fig. 2. An REM image of the pure edge dislocation on the $\alpha$-$Al_2O_3$ surface.



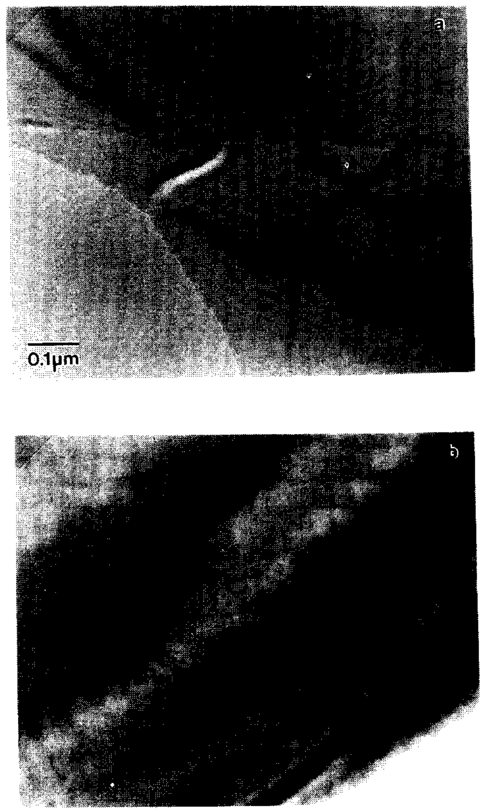




Fig. 3. Regularly spaced steps on the $\alpha$-$Al_2O_3$ surfaces could be seen from (a) the REM and (b) the TEM images.

Fig. 4. TEM images from different area. (a) A typical micro-crack in the basal plane. Discontinuous bend contour fringes could be seen. (b) Dislocation networks.

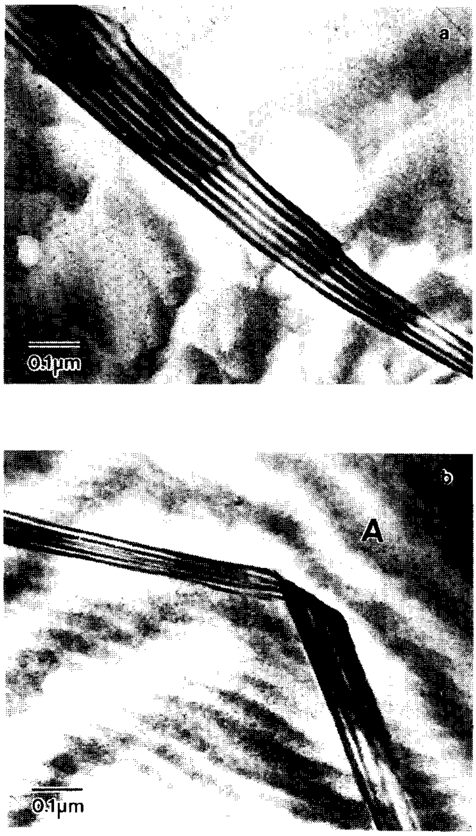



**Fig. 5.** Brigt-field TEM images. (a) Inclined twin boundary, (b) Two twin boundaries meet and overlapped.

mal pressures and temperatures is that of mechanical twinning.

The observation of twinning was therefore expected in our specimens from the above considerations. In practice, some of the twin band and boundaries have been found in the (0001) plane view.

Fig. 5a shows a typical (bright field) image of twin which is obtained with dynamical two-beam conditions in the matrix or the twin. Under this condition, the image of an inclined boundary simply consists of conventional thickness fringes and is similar to that of a grain boundary[15]. The fringes in the twin boundary is easily distinguishable with thickness fringes as shown at the specimen edge(region A) in Fig. 5b. Details of theoretical considerations on the formation of contrast at twin boundaries are presented in elsewhere[16].

Fig. 5b is taken from the near region of Fig. 5a. This figure shows two twin boundaries meet and overlapped. Although twin planes could not be positively identified with this figure, it is believed that these twins must be rhombohedral according to the theory and observation of Scott and Orr[17, 18].

Fig. 6a and 6b show bright field images of twin band (edge-on-view) having different features. Fig. 6b shows three boundaries indicated by arrows which suggest a double twin possibility. The corresponding diffraction pattern taken from an area including both matrix and twin in Fig. 6a is shown in Fig. 6c. Extra spots are caused by a 38° rotation from matrix about the [0001] axis. The forbidden reflection spots are caused by the width of twin is so small that only one directional spots are thought to be reflected.

Faceted basal twin interfaces which had been observed by Morrissey and Carter[5] have not been found in our experiment. Neither was only dislocation found around twin boundary nor was any twin-dislocation presents in the twin band. However, a few dislocations within the twin boundaries have been found as seen in Fig. 5a.

Fig. 6b which is the enlarged image of one of the two twin boundaries in Fig. 6a shows fine lattice fringes having 2.4Å spacing. Only one side (matrix part) of the fringes are obtained. This suggest that there may be a very small ro-

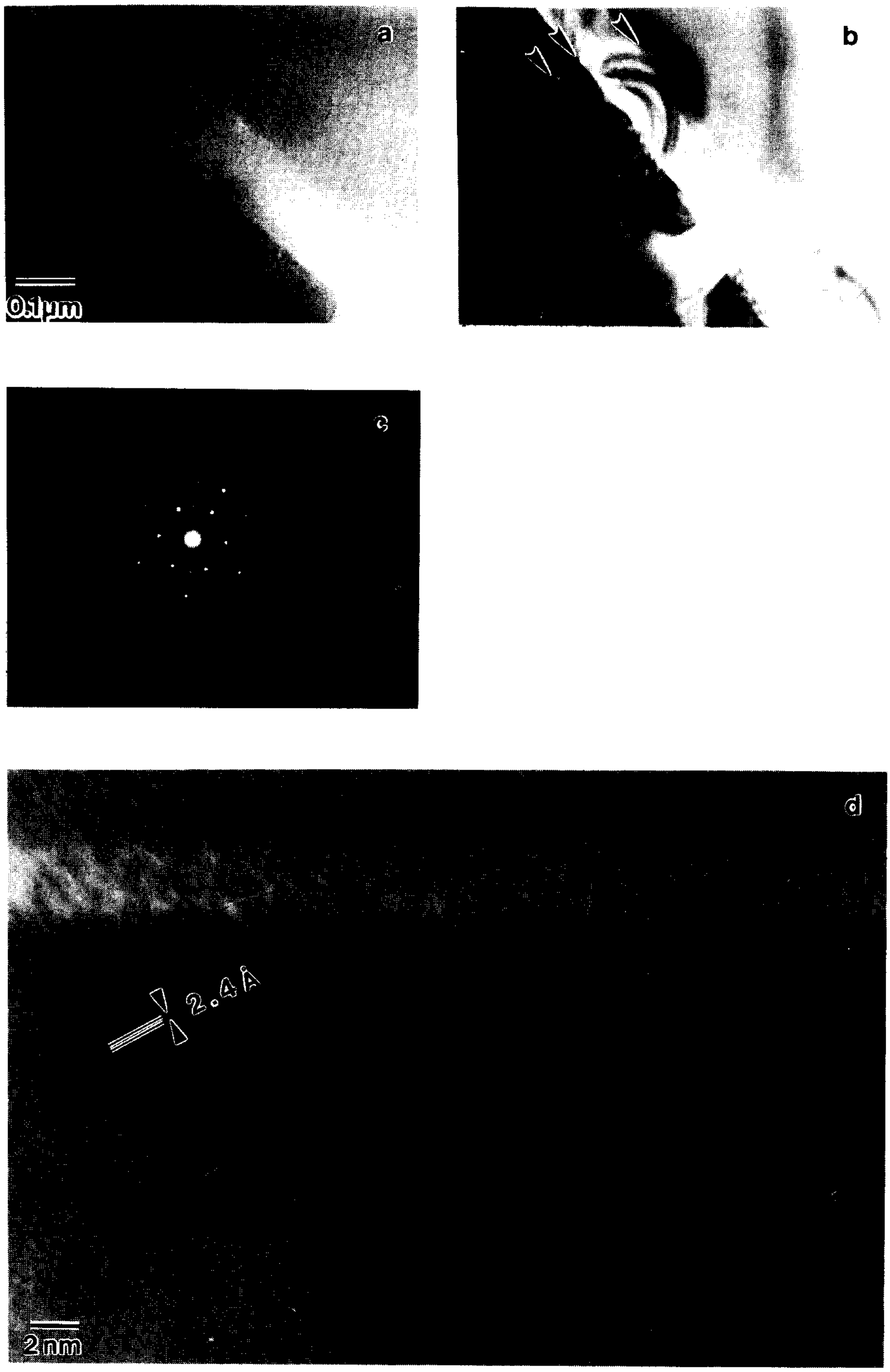



**Fig. 6.** Brigt-field TEM images of basal twins. (a) edge-on-view of basal twin, (b) a double twin, arrows indicate twin boundaries, (c) corresponding SADP from Fig. 6a, (d) high-resolution lattice image from the twin boundary.


Fig. 7. REM images from (a) (1120) surface, (b) (1102) surface. RHEED spot splitting occurred from the region of Fig 7a.

tation from the perfect basal twin orientation by a few degree about some axis except [0001], which can not be detected from the diffraction pattern.

In REM observation, no definite evidence of twin has been found; however, contrasts have been found in (11$\bar{2}$0) and (1$\bar{1}$02) surfaces as shown in Fig. 7a and 7b, respectively. There is no spot splitting in the diffraction pattern of Fig. 7b; hence, well defined split spots could be seen in the diffraction pattern of Fig. 7a. Only the curved lines of spots underlying Ewald sphere could be seen in the reflection high energy electron diffraction(RHEED) pattern, it is very difficult to say these split spots are caused by twin or by surface configuration (roof-top structure) [19]. These problem should be investigated by more sophisticated techniques or methods.

## 4. CONCLUSIONS

In-line dislocations were found at the front of cracks. It is believed that pile-up of these dislocations by dislocation movement causes crack initiation. This observation is controversy with the observation of Barber et al. [9].

Wiederhorn et al. [14] suggested that plastic flow involving dislocation or twin generation associated with fracture does appear possible. Our observation confirmed this possibility.

Basal and rhombohedral twins and their

boundaries are observed in both edge-on-view and inclined view. Lattice fringes near the basal twin boundary were obtained; however, only matrix side fringes are resolved. This suggests twin orientation is not the exact but slightly tilted. Therefore, the main observation of this paper is that the mechanism of stress-relief could be explained by formation of twins and cracks initiated by in-line dislocations pile-up.

Surface steps of which the heights are a few atoms high on the basal plane are also identified by both the REM and the TEM images.

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