ELSEVIER

Surface Science 383 (1997) 78–87

surface science

Nature of multilayer steps on the {100} cleavage planes of MgO single crystals

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Received 9 July 1996; accepted for publication 24 February 1997

Abstract

The results of an AFM study of the nature of multilayer steps on the {100} cleavage faces of MgO crystals is described and discussed. The results show that (i) multilayer cleavage steps of a width between 55 and 590 nm and of a slope between about 1 and 8° are stable, (ii) the terminal planes of multilayer steps are composed of high-index vicinal faces with indices (7 0 1), (42 0 1) and (77 0 1) for the ⟨010⟩ steps with respect to the (100) cleavage, which do not correspond to the low-index planes of the equilibrium form of MgO crystals, and (iii) that the curved parts of the profile of a multilayer cleavage step in its upper and lower regions extend to a distance equal to the width of the step. © 1997 Elsevier Science B.V.

Keywords: Atomic force microscopy; Faceting; Magnesium oxide; Single crystal surfaces; Surface structure

1. Introduction

Knowledge of the morphology of surfaces of crystalline solids is of practical and theoretical interest because many processes like the growth of epitaxial films, catalytic reactions and nanomanipulations are intimately connected with surfaces. In many cases cleavage faces are preferred to surfaces prepared by cutting, abrasion and polishing, because these preparation steps introduce defects and impurities in the surface layer. Cleavage varies from crystal to crystal, and optical microscopy and interferometry reveal large, thick cleavage steps of heights varying from less than 5 to 200 nm [1], isolated and relatively thin V-shaped cleavage steps [1,2], and river patterns terminating in the surface [1,2]. The height of thick isolated steps usually varies along their length [1].

Since the first study of the topography of cleaved sodium chloride crystals by electron microscopy using decoration replicas [3], the decoration technique has also been extensively employed to characterize the surface structures of cleaved MgO [4] and NaCl crystals [5]. With the advent of atomic force microscopy (AFM) and scanning tunneling microscopy (STM) in recent years there has been a renewed interest in the investigation of the surface morphology of cleavages of a variety of ionic single crystals [6–10]. The majority of these studies was devoted to the observation of mono- and bilayer steps [7–10], and step–terrace structures on the as-cleaved crystal faces [9].

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PII S0039-6028(97)00132-5

MgO single crystals are widely used as a substrate material for the growth of epitaxial thin films and superlattices for a variety of applications [11–13]. AFM studies of the cleavages of MgO crystals reveal monolayer steps [10], bilayer steps [9,11], trilayer steps [11], multilayer steps of a height of up to 2 nm (i.e. up to ten monolayers height [10]), and thick multilayer steps of heights of 20–50 nm [11].

During our studies of nanoindentation hardness on the cleaved faces of MgO single crystals, a variety of surface structures at the atomic level, including the emergence of isolated steps on the surface, disc-shaped hillocks and depressions, local pinning of monolayers, and thick multilayer steps of heights up to 30 nm, were observed. Since until now no systematic study dealing with the cleavage morphology of ionic crystals has been carried out, it was thought worthwhile to undertake this type of investigation on MgO single crystals. Moreover, the simple halite-type structure of MgO, and the existing literature on the deformation behavior and cleavage morphology of halite-type crystals including MgO, are additional advantages in the understanding of the various step patterns produced during its cleavage.

The present paper is devoted to the study of multilayer steps on the {100} cleavage faces of MgO crystals, where some general features of their formation during the process of cleavage are described and discussed.

2. Experimental

The MgO single crystals for the study were obtained by cleavage along the {100} planes from a batch of crystals (AERE, Harwell, UK). Both freshly cleaved specimens and specimens cleaved several hours prior to the observations were used. The atomic force microscopy (AFM) was carried out employing a commercial Nanoscope III manufactured by Digital Instruments, Santa Barbara, CA. The AFM images were recorded in ambient atmosphere at room temperature, in both contact and tapping modes. For the AFM images, the cleaved samples were mounted in the nanoscope in such a manner that their ⟨010⟩ edges were used as reference for the determination of the direction of steps. Initially, 15 μm × 15 μm areas with cleavage multilayer steps oriented in various directions were scanned. However, for the precise measurement of the angle of inclination θ and the slope m of multilayer steps (see below), 1 μm × 1 μm and 1.5 μm × 1.5 μm areas of cleavages with multilayers oriented in the [010] directions were scanned.

In all 37 images from about 50 images recorded on five cleavage faces were randomly selected for the measurement of the height h and width d of cleavage multilayer steps. Usually one measurement was made on one multilayer step but, in the case of multilayer steps covering large areas of 15 μm × 15 μm images, measurements were made at two places, i.e. close to their upper and lower extremes in the images. A total of 104 measurements was made on 88 multilayer steps. The height h and the width d of the cleavage multilayer steps were determined from the recorded images in such manner that only heights in the interval of constant slopes were considered for the analysis.

The errors in the measurements of constant values of the slope of terminal planes of multilayer steps are associated with the digitalization procedure involved in the measurement of their horizontal distance (i.e. width) d and vertical distance (i.e. height) h from the digital images of forces, and depend on the dimensions of the images. For example, for the 15 μm × 15 μm images, the maximum error involved in the measurement of d was ± 50, ± 25 and $\pm 17\%$ for values of d of 58.6, 117.2 and 175.8 nm, respectively, while the error in the measurement of h was ± 5, ± 2.0 and $\pm 0.5\%$ for values of h of 1.43, 3.58 and 14.35 nm, respectively. However, in the case of 1.5 μm × 1.5 μm images, for the above values of d and h, the corresponding errors were reduced by a factor of 10. Therefore, for the purpose of establishing relationships between the slopes and the widths d of multilayer steps, the slopes were measured from images of small scan areas.

3. Results

3.1. General features of multilayer steps and their profiles

During the successive scanning of a particular region of the {100} cleaved faces of MgO in the contact mode, it was found that with an inappropriate force set-point, the configuration of monolayer steps (i.e. layers of height equal to the lattice parameter $a = 0.21$ nm) can undergo modification, but the configuration of multilayer steps remains practically unaltered. Similarly, although the tapping mode exerts forces up to 10^{-6} N on hard samples [14], no influence of this imaging mode was observed on monolayer or as multilayer steps during the repeated scanning of a particular region of cleavage. However, during our study of the cleavages of MgO, the selected region of a particular cleavage was scanned only once. Therefore, it can be concluded that the configurations of the multilayer steps recorded in our images represent an equilibrium state which is not affected by the type of imaging mode.

It is well known [15] that humidity strongly affects the configuration of monolayer steps on the {100} cleavage faces of NaCl and leads to their motion. However, in the case of MgO, such an effect was not observed in the case of monolayer or multilayer steps. In contrast to the fairly soluble NaCl, MgO is practically insoluble in water. Thus, it may be argued that the difference in the influence of humidity on the structure and motion of steps on the {100} cleavage faces of NaCl and MgO crystals is associated with the difference in their solubility in water.

Fig. 1 shows an example of the optical photograph of the morphology of the {100} cleavage faces of MgO crystals. As observed in the case of other rock-salt type ionic crystals [2], the cleavage consists of clearly visible sharp multilayer steps parallel to the ⟨010⟩ direction close to the left edge, which become parallel to the ⟨011⟩ directions in the interior regions of the cleavage, diffuse (ill-defined) steps, V-shaped patterns, and river patterns composed of multilayer steps of different heights. For the AFM observations, flat regions between steps oriented along the ⟨010⟩ directions and between inclined multilayer steps were scanned. An example of the scanned image of the morphology of the cleavage surface is illustrated in Fig. 2. Fig. 2 shows that thin, rounded monolayer steps originating from disk-like elevations cover the relatively flat areas between thick parallel multilayer steps, and that many isolated monolayer steps leave one multilayer step and join another. The isolated monolayer steps have a height of 0.21 nm, which is equal to the lattice parameter of MgO crystals. The details of the distribution of monolayer steps are clearly seen in Fig. 2. However, since this paper is confined only to the nature of multilayer steps, the results of the distribution and structure of monolayer steps will not be described here.

Fig. 3 illustrates the top view of the image of Fig. 2 and the profile of its multilayer steps. The acquisition data, representing the corresponding horizontal distances (width) d and vertical distance (height) h for an angle of inclination θ of two steep multilayer steps (hereafter referred to as left and right steps, respectively, with reference to the left edge) are also given in Fig. 3. As can be seen from Fig. 3, the value of the angle θ of a multilayer step is not constant in the entire region of the step (compare the angles of the left step profile, indicated by the light-grey and dark-grey markers), and the values of the angles of two multilayer steps can differ enormously (compare the angles of the left and right steps).

It should be mentioned that, as in Fig. 1, the edges of the images of Figs. 2 and 3 are parallel to the [010] and [001] directions, and the parallel multilayer steps are oriented in the [017] direction. However, multilayer steps oriented in the [010], [001] and other [0kl] directions are also frequently observed.

Fig. 4 illustrates typical examples of the profiles of multilayer steps of different angles obtained from images of 1 μm × 1 μm and 1.5 μm × 1.5 μm areas. Examination of these profiles of multilayer steps reveals that they may be grouped broadly into three categories. In category (a), the bunches of steps have a slope θ up to 1°, and individual steps could be resolved. In category (b), the slope of multilayer steps was between 1 and 6°, but individual steps were not resolved. The multilayer steps of category (c) had a slope exceeding 6° and differed from those of category (b) in having clearly distinguishable rounded upper and lower parts of the profiles.

3.2. Dependence of the slope and height of multilayer steps on their width

Fig. 5 shows the experimental data on the dependence of the slope m of multilayer cleavage steps

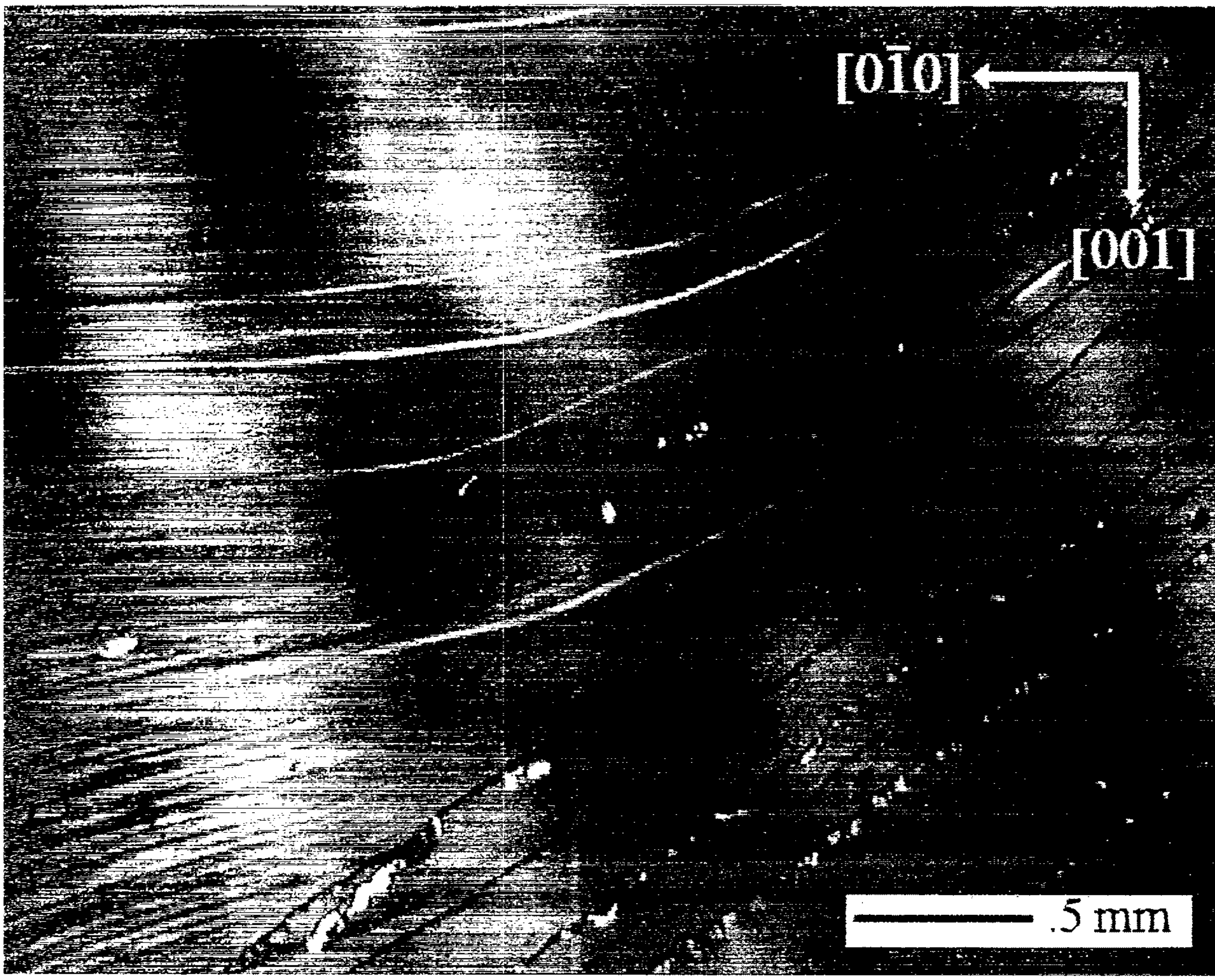


Fig. 1. Optical photograph of the morphology of the {100} cleavage faces of MgO crystals. Note that the cleavage consists of clearly visible sharp multilayer steps parallel to the [010] direction close to the edge and inclined by an angle up to 45° in other regions, diffuse (ill-defined) steps parallel to the [011] direction, V-shaped patterns and river patterns composed of multilayer steps of different heights. The river patterns can be seen in the region of the bottom left corner.

on their width d for the {100} cleavage face of MgO crystals

$$m = \tan\theta = h/d. \tag{1}$$

Fig. 5 includes all the experimental data on the slope m for ⟨010⟩ (open and closed squares) as well as inclined multilayer steps (open triangles), measured from AFM images of 15 μm × 15 μm as well as smaller areas. It may be noted from Fig. 5 that there are values of the slope m of steps, which are piled up, especially for step widths up to 240 nm, at some particular values of the width d (i.e. 120, 180 and 240 nm). This pile-up of points corresponds to the measurement of the slope of multilayer steps from images of 15 μm × 15 μm areas, and is associated with the digitalization procedure involved in the measurement of d.

It is clear from Fig. 5 that the slope of both ⟨010⟩ and inclined multilayer steps is not constant, but depending on the value of the step width d, lies in a particular range of the highest and the lowest values of the slopes. The dependence of the highest and the lowest values of the slopes m of multilayer steps on their width d was analyzed

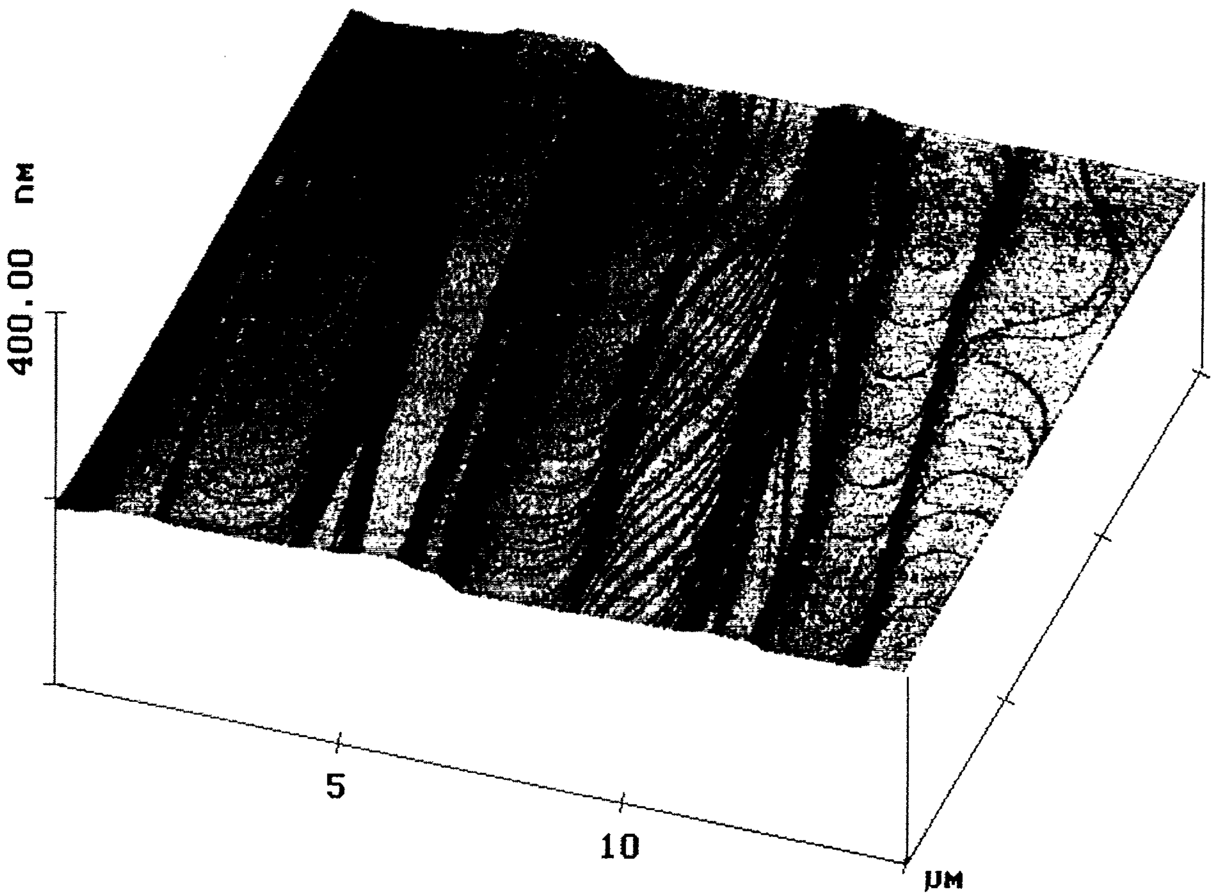


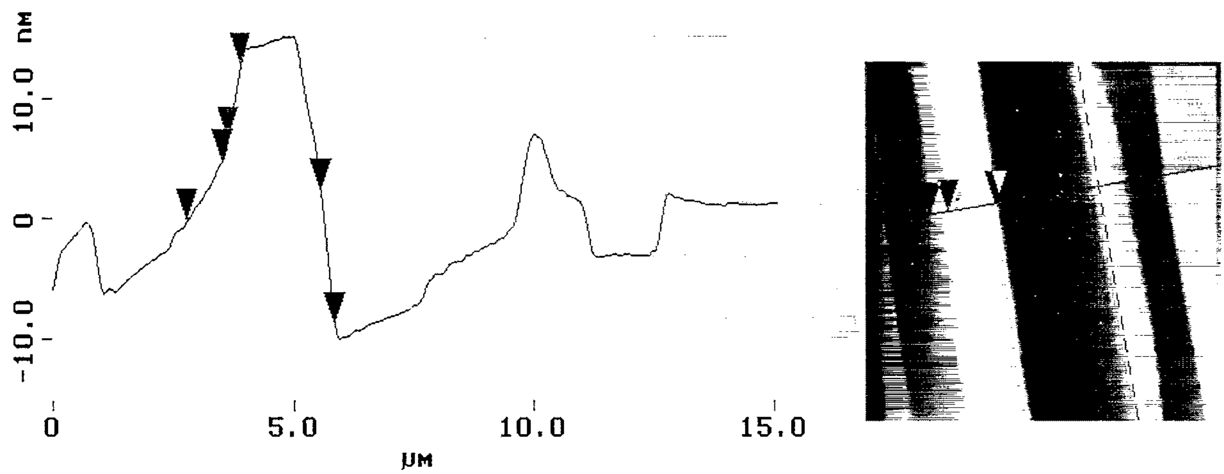
Fig. 2. 3D AFM image of the cleavage surface containing multilayer cleavage steps oriented in the [017] direction. As in Fig. 1, the edges of the image correspond to the [010] and [001] directions. Note that monolayer steps originating from disk-like elevations cover relatively flat areas between thick multilayer steps, and that many isolated monolayer steps leave one multilayer step and join another.

from the experimental data, shown by the peripheral filled squares in Fig. 5, obtained from 1 µm × 1 µm and 1.5 µm × 1.5 µm images for the ⟨010⟩ steps. For the analysis, the data of the peripheral filled-square points corresponding to the ascending and descending parts of the highest slopes and to the lowest slopes were considered in such a way that end points at the vertices of the quasi-triangle were treated as common points to obtain best-fit correlations for two plots around a vertex on either side. The number of points for analysis is then 7, 6 and 11 for the ascending, descending and lowest slopes, respectively. It was found that the step slope m and the step width d are related by

$$m = kd^n, \qquad (2)$$

where the constants k and n in the ascending and descending parts of the highest values (curves 1 and 2) and in the lowest values of slopes (curve 3) are listed in Table 1.

As can be seen from Table 1, the fit is reasonably good for the data in the ascending and descending parts of the highest slopes (curves 1 and 2), but it is very poor in the case of the lowest slopes (curve 3). The reasonably good and poor fits are associated with errors involved in the measurement of both the step height h and the step width d. As



Horiz distance	761.72 nm
Vert distance	5.052 nm
Angle	0.380 deg
Horiz distance	234.38 nm
Vert distance	5.566 nm
Angle	1.360 deg
Horiz distance	292.97 nm
Vert distance	11.102 nm
Angle	2.170 deg

Fig. 3. Top view of the cleavage of image of Fig. 2 and the profile of its multilayer steps. The acquisition data for the angle of inclination θ of two steep multilayer steps (referred to as left and right steps, respectively, with reference to the left edge) are also shown. Note that the value of the angle θ of a multilayer step is not constant in the entire region of the step (compare the angles of the left step profile, indicated by light-grey and dark-grey markers), and that the values of θ of two multilayer steps can differ enormously (compare the angles of the left and right steps).

mentioned above, the absolute values of the errors decrease with increasing values of these parameters.

Fig. 6 presents the experimental data of the dependence of the height h and the width d for the ⟨010⟩ multilayer steps corresponding to the lowest range of the slope in Fig. 5. Fig. 6 includes the experimental data for multilayer steps scanned in areas of various dimensions, and illustrates the data corresponding to both the open and filled squares of Fig. 5. It is interesting to note that the fit of the data is now very good (correlation coefficient 0.949) and may be expressed by Eq. (2) with $n=0.237$ and $k=5.3\times10^{-3}\ \text{nm}^{-0.237}$. These values of n and k are in good agreement with those given in Table 1. In comparison with a poor fit of the experimental data of m against d, a better fit of the data of h against d implies that the errors in their measurements are interrelated.

From Fig. 5 and Table 1 it may be noted that for the highest slopes of the ⟨010⟩ multilayer cleavage steps, with increasing step width d the slope m first increases practically linearly with d from an initial value of 0.013 (i.e. the angle of inclination $\theta=0.75°$) at $d=55$ nm up to a constant maximum slope m_{max} of about 0.138 (i.e. the angle of inclination $\theta=8°$) for $d=430$ nm and then rapidly decreases (m inversely proportional to $d^{5.5}$) to a limiting value m_{lim} of about 0.024 ($\theta=1.4°$) at $d=590$ nm, the latter being determined by

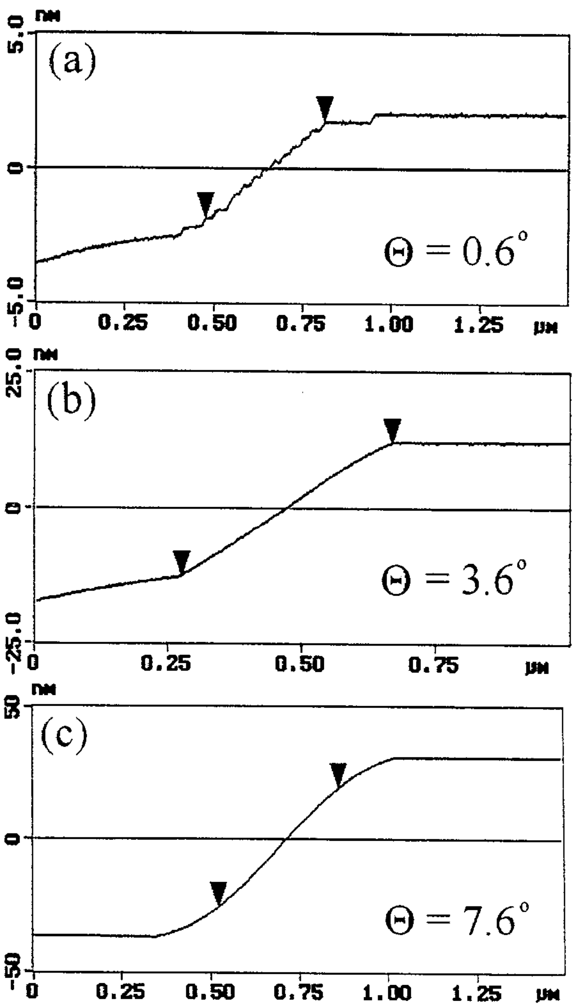


Fig. 4. Typical examples of the profiles of observed multilayer steps of different angles of inclination θ. Note that the ranges of observation of the steps with the above profiles are: (a) $\theta < 1'$, (b) $1° < \theta < 6°$, and (c) $\theta > 6°$.

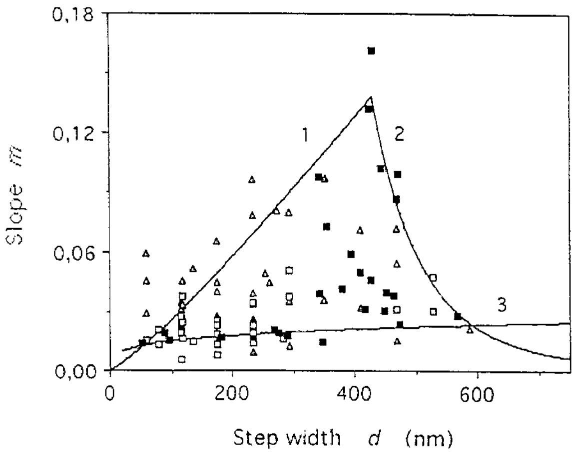


Fig. 5. Plots of the dependence of experimental slope m on the horizontal width d of multilayer steps of constant slope on the cleavage of MgO crystals. Open and filled squares represent data for ⟨010⟩ steps, while triangles represent inclined steps. Open squares correspond to data obtained mainly from 15 μm × 15 μm images, while filled squares correspond to data obtained from images of small areas. The peripheral filled squares in the ascending and descending parts and in the lowest part of the slope were considered for analysis of the relationship between m and d. The curves (1), (2) and (3) were drawn using constants given in Table 1. Note that there is a minimum value of the slope m_{min} beyond which multilayer steps are observed, and that inclined steps have a higher slope than ⟨100⟩ steps.

Table 1
Constants k and n of Eq. (2) in the ascending and descending parts of the highest range and in the lowest range of slopes

Curve	k (nm^{-n})	n	Correlation coefficient
Upper ascending (curve 1, Fig. 5)	1.27×10^{-4} (2.0×10^{-4})[a]	1.150 (1.15)[a]	0.957
Upper descending (curve 2, Fig. 5)	3.19×10^{13} (4.2×10^{13})	−5.452 (−5.50)	0.964
Lower (curve 3, Fig. 5)	4.52×10^{-3} (4.5×10^{-3})	0.258 (0.26)	0.396

[a]The values of k and n given in parentheses were used to plot the curves of Fig. 5.

the lower limit of the slope. Between the interval of step width 55 nm < d < 590 nm, the limit of the lowest slope depends weakly on d and the slope changes its indices from (77 0 1) at d = 55 nm to (42 0 1) at d = 590 nm. For the ⟨010⟩ cleavage steps, the steps corresponding to the maximum value of the slope have approximately the indices (701) at d = 430 nm.

It is interesting to note that the slope of the ⟨010⟩ and inclined steps essentially lies in the same range of d, but that in the interval of step width d < 590 nm the slope of the inclined steps is somewhat higher than that of the ⟨010⟩ steps. However, in general, the terminal planes of cleavage multilayer steps correspond to vicinal planes and not to low-index planes of the equilibrium form, and the steps in the range of slopes shown in Fig. 5 are stable.

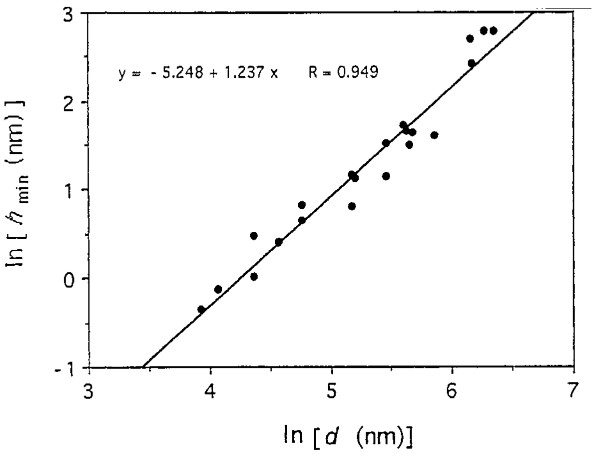


Fig. 6. Log–log plot of the dependence of the height h_{min} of steps of lowest slope m_{min} of ⟨010⟩ multilayer steps on the horizontal step width d. All experimental data obtained from large and small areas are included. In the case of the three clusters of points for $d=120$, 180 and 240 nm, the lowest three values were considered in the analysis. Note that h_{min} increases as $d^{1.24}$, i.e. m_{min} increases as $d^{0.24}$. The data essentially correspond to the multilayer step profile of category (a).

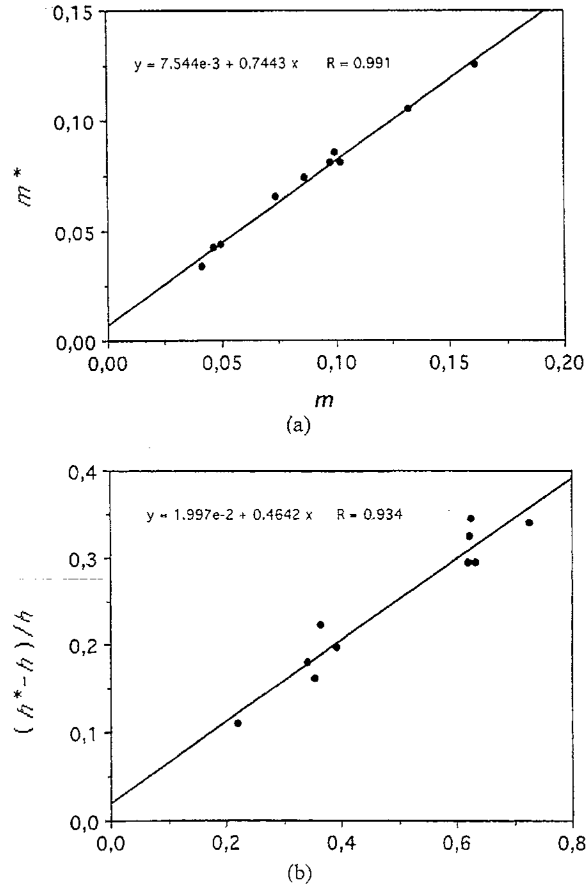


Fig. 7. Plots of dependencies of (a) h^*/d^* on h/d and (b) $(h^*-h)/h$ on $(d^*-d)/d$. Note that both these dependencies are linear and essentially pass through the origin. The data correspond to the multilayer step profiles of categories (b) and (c).

In order to establish a relationship between the curvature of multilayer steps, experimental data of horizontal and vertical distances obtained from their profiles in the range of slopes corresponding to categories (b) and (c) were analyzed. For this purpose, the data of the width d and the height h corresponding to constant slope m and the total width d^* and the total height h^* were presented as plots of h^*/d^* against h/d and $(h^*-h)/h$ against $(d^*-d)/d$. These plots are shown in Figs. 7a and b, respectively.

4. Discussion

From Fig. 5 and Table 1, the following features of cleavage steps on the {100} face of MgO crystals may be noted.

(1) The ⟨010⟩ multilayer cleavage steps with slopes enclosed in the triangular region of the graph of slope m against step width d are stable. In other words, the multilayer steps of a width d between 55 and 590 nm and of a slope m between 0.02 and 0.14 are stable.

(2) For the highest values of the slope of the ⟨010⟩ multilayer cleavage steps, with increasing step width, the slope m first increases practically linearly with d ($m \propto d^{1.15}$) from an initial value of $m=0.013$ up to a constant maximum slope m_{max} of about 0.138 and then rapidly decreases (m is inversely proportional to $d^{5.5}$) to a limiting value m_{lim} of about 0.024.

(3) Between 55 nm $< d <$ 590 nm the lower limit of the lowest slope increases weakly with d ($m \propto d^{0.26}$).

The above results of the dependence of the slope of ⟨010⟩ multilayer cleavage steps on the {100} faces of MgO crystals on the step width d may be explained in terms of their stability due to attractive and repulsive interactions between monolayer steps. When attractive interactions predominate over repulsive interactions, the formation of bunched steps is favored. However, when repulsive interactions predominate over attractive interactions, the bunched steps tend to dissociate. Moreover, there is a limiting value of slope when both these attractive and repulsive interactions between the steps take place. Thus the steps with slopes enclosed in the triangular region of the graph of slope m against step width d are stable.

Using the relation $N=md/a$, the number N of elementary steps of height 0.21 nm composing stable ⟨010⟩ cleavage multilayer steps on the {100} face of MgO crystals may be estimated to be about 3, 283 and 67 for multilayer steps in the initial stage at 55 nm, of maximum stability at 430 nm and in the final stage at 590 nm, respectively. Physically, this means that attractive interactions develop between monolayer steps when their number exceeds 3, i.e. their mutual distance λ is between 9 and 18 nm, while the multilayer steps attain maximum stability when $\lambda \approx 1.45$ nm.

To our knowledge, vicinal faces such as (701), (42 0 1) and (77 0 1) for the [010] steps have not yet been observed either on cleavage multilayer steps or on bunched steps due to growth or dissolution on the surfaces of ionic crystals. However, using scanning tunneling microscopy, Langford et al. [6] observed segments of stable {027} planes on the fractures faces of MgO single crystals. On the (001) face of GaAs, Hata et al. [16] observed the evolution of step bunches composed of high-index (216) facets as high as 56 nm by annealing the samples at elevated temperatures in AsH_3 and H_2 ambient. In contrast to this, Ohkuri et al. [17] reported that the bunched steps formed after thermal treatment in an AsH_3/H_2 atmosphere on the (001) terrace of GaAs did not have a constant slope and thus were not facets. Annealing of polished and cleaved (100) faces of MgO in O_2, however, does not lead to step bunches [10].

There exists a voluminous literature on the experimental and theoretical aspects of the formation of facets on planar surfaces of single crystals in equilibrium, growth, dissolution and annealing (see, for example, Refs. [16–18]). Using the Monte Carlo method, Vlachos et al. [18] examined the dynamics of the formation of facets on the (100) plane of a cubic lattice as a function of surface temperature, crystallographic orientation, and adsorption–desorption and surface diffusion processes. These authors found that faceting does not evolve on orientations deviating from the (100) surface by a slope of less than about 0.1, i.e. vicinal planes with indices exceeding about (10 1 0) orientation, although these orientations are thermodynamically unstable. They attributed this observation to the importance of mass-transfer limitations over long distances for small orientations from low-index planes when transport from one ledge to its neighbors through the terrace requires long times. This explanation is also valid in the case of cleavage steps on MgO crystals.

Using the self-consistent quantum approach for the investigation of the structure and stability of steps and kinks on MgO surfaces, Goniakowski and Noguera [19] found that the {301} vicinal surface with steps is more stable than the {3 1 10} surface with periodic kinks. Physically, this means that the distance between steps in the former case is smaller than that in the latter case. This inference is in agreement with the observation that the range of slopes of the ⟨010⟩ multilayer steps is smaller than that of the inclined ⟨01l⟩ steps.

The plots of h^*/d^* against h/d and $(h^*-h)/h$ against $(d^*-d)/d$, shown in Figs. 7a and b, respectively, reveal that both these dependencies are linear and essentially pass through the origin. This means that basically there is no difference between ⟨010⟩ steps of different categories, and that their configuration is determined by the ratio between h and d.

An important correlation may be established between d^* and d from the dependencies of Fig. 7. Neglecting the initial small deviation from the zero value, the dependence of Fig. 7b may be written as

$$h^*/h=k_1 d^*/d+(1-k_1), \tag{3}$$

where the constant $k_1=0.4642$. Upon rearrangement, this equation may be rewritten in the form

$$h^*/d^*=(1-k_1)h/d^*+k_1 h/d. \tag{4}$$

Assuming that $d^*=k_2 d$, where k_2 is a constant characteristic of the crystal and cleavage step orientation under consideration, one may express Eq. (4) as

$$h^*/d^* = [(1-k_1+k_1k_2)/k_2]h/d. \quad (5)$$

Eq. (5) represents the relationship illustrated by Fig. 7a with the constant $[(1-k_1+k_1k_2)/k_2] = 0.7443$. Using the value of k_1 given above, one obtains the constant $k_2 = 1.913$ and $(d^*-d) = 0.913d$. In other words, the curved parts of a multilayer cleavage step in its upper and lower regions extend to a total distance equal to the width of the step. However, the physical interpretation of this relationship remains unknown.

5. Conclusions

From the present study on the slope of cleavage multilayer steps on the (100) face of MgO single crystals, the following conclusions can be drawn.

(1) The multilayer cleavage steps of a width d between 40 and 600 nm and of a slope m between 0.02 and 0.14 are stable.

(2) The multilayer steps on the cleavage faces of MgO are composed of high-index vicinal planes with indices like (701), (42 0 1) and (77 0 1) for the [010] steps with respect to the (100) cleavage. The terminal planes of cleavage multilayer steps do not correspond to the low-index planes of the equilibrium form of MgO crystals.

(3) The curved parts of a multilayer cleavage step in its upper and lower regions extend to a total distance equal to the width of the step.

Acknowledgements

The authors express their gratitude to the "Serveis Cientifico-Tecnics" of the University for technical facilities. One of the authors (K.S.) is also grateful to the authorities of the Universitat de Barcelona for extending an invitation to work as visiting professor, and to the Generalitat de Catalunya for granting him a maintenance grant. This work was supported financially by the Comision Interministrial de Ciencia y Technologia under Research Project MAT94-1938, and by the Comissarat de Generalitat de Catalunya under Research Project SGR344,1995.

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