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Second-order faceting–roughening of the tilt grain boundary in zinc

Dedicated to Professor Dr. Günter Gottstein on the occasion of his 65th birthday

The faceting of the almost stationary $[10\bar{1}0]$ tilt grain boundary with a misorientation angle θ of 84° in the Zn bicrystal has been investigated. The shape of the very slow migrating grain boundary has been studied in situ between 350 and 400 °C using polarized light. Two intersecting facets lie in the closely-packed planes of the constrained coincidence sites lattice with coincidence parameter $\Sigma = 15$. In the as-grown sample flat grain boundary facets form the sharp first-order ridge with the break of the first derivative $\partial y/\partial x$ of the grain boundary shape similar to those observed in our previous works on GB faceting in Zn. However, above 350 °C the sharp first-order ridge is substituted by the smooth grain boundary portion without a $\partial y/\partial x$ derivative break similar to that observed in twin grain boundaries in Mo. The transformation of the as-grown first-order grain boundary ridge into continuous one has been observed for the first time. The critical parameter α has been calculated using the grain boundary shape in the transition region. α increases from $\alpha = 1.7 \pm 0.07$ for 350 °C to $\alpha = 1.9 \pm 0.07$ for 385 °C approaching the $\alpha = 2$ value predicted in the mean-field Andreev model for the continuous surface roughening.

Keywords: Grain boundaries; Faceting; Roughening; Migration; Zinc

1. Introduction

In the pioneering works of Prof. Dr. G. Gottstein and his coworkers the migration of individual grain boundaries (GBs) and triple joints (TJs) has been thoroughly studied both experimentally and theoretically [1]. Several important features of grain boundary faceting–roughening have been observed in the last few years [2–14]. Particularly, GB facets always appear in the so-called special GBs with misorientation angle θ close to the coincidence one θ_Σ. Moreover, the faceting–roughening seems to be the physical reason for the existence of GBs with special structure and properties only in certain angular intervals close to θ_Σ and in a restricted temperature interval [2]. GB facets are almost always parallel to the closely-packed planes of the coincidence site lattice (CSL) in the materials with cubic lattice [3–8] or of the constrained CSL (CCSL) in non-cubic materials (such as Zn) [9–14]. The GB facet becomes shorter with increasing temperature and fully disappears at the GB roughening temperature T_R [9–11]. T_R is higher for the most closely-packed CSL planes and lower for the less closely-packed CSL planes [3–7]. Therefore, the number of crystallographically different GB facets increases with decreasing temperature [3, 4]. GB facets lying in the most closely-packed CSL planes (especially in metals with low stacking-fault energy, such as Cu) can exist up to the melting temperature T_m, in other words their $T_R > T_m$ [3, 4, 7]. GB roughening is reversible: by decreasing temperature below T_R a facet appears again, with small temperature hysteresis [9]. Moreover, a facet disappears above T_R and appears below T_R with some time delay, similar to a particle of the overheated bulk phase above the bulk solvus line or the undercooled one below the solvus line [9]. The presence of GB facets drastically influences the kinetics of GB migration [9–13]. However, all GB faceting–roughening transitions observed in zinc up to now are of the first order [9–13]. In other words, the break of the first derivative $\partial y/\partial x$ of the GB shape is always present between two GB facets [9, 11–13], between the GB facet and the rough (curved) GB portion [9–11], or even between two rough (curved) GB portions (first-order ridge) [9]. Nevertheless, the second-order (continuous) ridges between the GB facet and the rough (curved) GB portion can exist and have been observed in Mo [14] and Al bicrystals [5–7]. There is no principal limitation or theoretical prohibition for the existence of second-order (continuous) ridges in zinc GBs as well. Therefore, this work is devoted to the search and first observation of the second order GB faceting–roughening transitions in zinc.

2. Experimental details

Zn $[10\bar{1}0]$ flat bicrystal was grown from Zn of 99.999 wt.% purity using a modified Bridgman technique [1, 15, 16]. The seeds in this bicrystal form the tilt GBs with misorientation angle θ of 84° (Fig. 1). The GB is perpendicular to the surface of the sample. The $[10\bar{1}0]$ axes in both grains are also perpendicular to the surface of the sample. The GB already contained some facets in the as-grown sample (Figs. 1 and 2a). These facets are also perpendicular to the surface of the sample. Such long facets were produced by the programmed change in the thermal regime during bicrystal growth. As a result, the GB started to grow in one close-packed CSL (coincidence site lattice) direction, then changed to another, and later came back to the first one.

Due to its optical anisotropy, zinc allows one to study the shape of the GB with the aid of polarized light. During the heating in the microscope hot stage, the shape of the as-grown facets changes, they also slowly move towards the mean GB position in the bicrystal (from the right to the left in Fig. 2). The steady-state shape of the migrating GB was studied in situ on a hot stage of an optical microscope in the temperature range between 350 and 400 °C. The samples were protected from oxidation by a pure nitrogen atmosphere. Before measurements the samples were electropolished in the $H_3PO_4 + C_2H_5OH$ solution. An additional polarization filter applied in the reflected beam permits distinguishing different orientations of the grains by the different intensity of the reflected light. The GB shape was recorded in the course of experiment by a color video camera connected with the microscope and a video recorder. This method of studying the GB migration was originally developed in Refs. [15, 16].

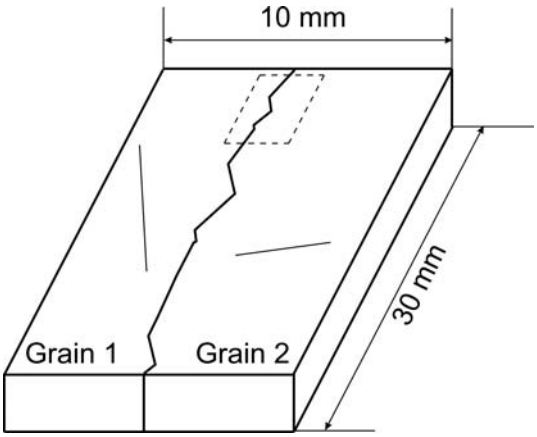
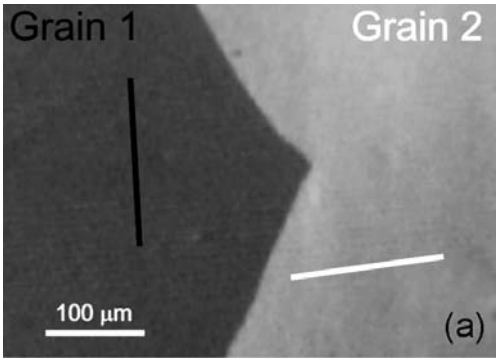


Fig. 1. Scheme of the Zn bicrystal containing the $[10\bar{1}0]$ tilt GB with a misorientation angle of 84°. The orientation of basal plane (0001) in both grains (thick lines) is shown. The positions of facets are shown. The broken line marks where the sample was cut from the as-grown bicrystal.



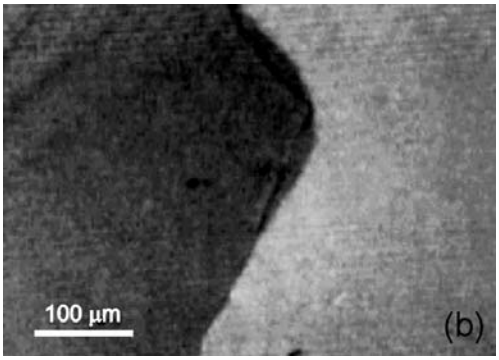


Fig. 2. Micrographs of the GB facets obtained in situ on a hot stage using polarized light. (a) as grown sample (room temperature) and (b) 385 °C.

3. Results and discussion

The micrographs in Fig. 2 obtained in situ on a hot stage using polarized light show typical shapes of the GB, namely (a) the shape of the facets in the as-grown sample and (b) after annealing at 385 °C. In the as-grown sample flat facets 1 and 2 intersect forming the sharp ridge with the break of the first derivative $\partial y/\partial x$ of the GB shape as in Refs. [9, 11–13]. In the temperature range below 350 °C GB remains stationary with the sharp ridge between facets. Starting from 350 °C, the GB ridge between facets 1 and 2 becomes rounded, without the break of the first derivative $\partial y/\partial x$ of the GB shape. The shape of a typical rounded ridge is shown in Fig. 2b.

The *c*/*a* ratio of lattice spacing *a* in basal plane (0001) and *c* perpendicular to (0001) is irrational in Zn. In hexagonal close-packed (hcp) crystals, the three-dimensional CSLs can only be obtained when a *c*/*a* ratio of the lattice spacing *a* in the basal plane (0001) and *c* perpendicular to (0001) is rational [17]. Therefore, exact coincidence site lattices exist in Zn only for GBs with [0001] rotation axis. In all other cases, including $[10\bar{1}0]$ tilt GBs, it is necessary to constrain the *c*/*a* value to the closest rational value to obtain a three-dimensional CSL, which is called a constrained coincidence site lattice (CCSL) [18, 19]. Such an approach allowed the successful description of observed GB structures in Zn [19, 20]. The section of CCSLs perpendicular to $[10\bar{1}0]$ tilt axis is shown in Fig. 3 for GB with misorientation angle of 84°. Filled and empty circles mark the sites of

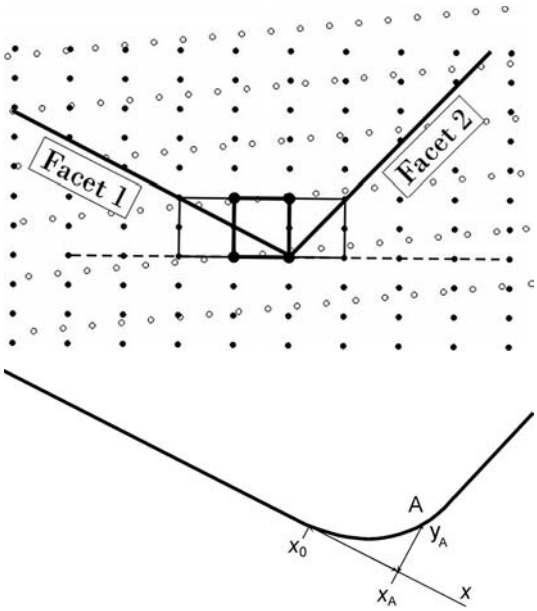


Fig. 3. Section of CCSLs perpendicular to $[10\bar{1}0]$ tilt axis for GBs with misorientation angles θ of 84°. Filled and empty circles mark the sites of two misoriented Zn lattices. Large circles mark the sites of the CCSL. The inverse density of coincidence sites is $\Sigma = 15$. The unit cell of the respective CCSL, position of basal plane (0001) for grain 1 (cf. Figs. 1 and 2a) and position of the most closely packed plane in the CCSL are also shown. In the bottom part of the figure the scheme for the quantification of a curved GB portion close to the facet is shown.

two misoriented Zn lattices. Large circles mark the sites of the CCSL. The sites of the lattices do not coincide exactly, the difference reaches a few per cent of the lattice spacing. This situation is similar to the near-coincidence GBs in materials with cubic lattice, when the misorientation angle is close, but is not equal to the misorientation of exact coincidence θ_Σ, but is still inside of the area of existence for special GBs [2]. The inverse density of coincidence sites is $\Sigma = 15$. The unit cell of the respective CCSL, position of basal plane (0001) for grain 1 (cf. Figs. 1 and 2a) and position of the most closely packed plane in the CCSL are also shown. It can be clearly seen that the facets observed in $\theta = 84^\circ$ GB (cf. Fig. 2) lie parallel to the closely packed planes in $\theta = 84^\circ$ CCSL. The *c*/*a* ratio in Zn is temperature dependent [21]. The CCSL in Fig. 3 is shown for 350 °C.

In the real polycrystals, the ideal tilt GBs (similar to those studied in this work) form a rather small sub-population of the whole GB array. Tilt GBs possess a high average interplanar spacing. Therefore, according to the criterion of Wolf and co-workers, they belong to the low-energy ones [22–24]. Tilt GBs also fulfil the criterion proposed by Rohrer and co-workers, since they have a low average surface energy of two surfaces forming the GB [25–27]. The temperature of the GB wetting phase transformation T_w permits judgement about GB energies [28–30]. For example, the minimal temperature of the GB wetting phase transformation in Al–Zn alloys is $T_{wGB0\%} = 440$ °C [29]. Below $T_{wGB0\%} = 440$ °C completely wetted GBs in the Al–Zn polycrystals do not exist. $T_{wGB0\%}$ corresponds to the GBs with maximum energy [30]. The maximum temperature of the GB wetting phase transformation in Al–Zn alloys is $T_{wGB100\%} = 565$ °C [29]. Above $T_{wGB100\%} = 565$ °C all GBs in the Al–Zn polycrystals are completely wetted. $T_{wGB100\%}$ corresponds to the GBs with minimum energy [30]. Indeed, the whole interval of T_w for the [110] tilt GBs in Al is very narrow. All wetting temperatures for tilt GBs are also very close to $T_{wGB100\%}$ [28]. For example $T_{wGB35^\circ} = 530$ °C for the low-energy tilt [110] GB with misorientation angle $\theta = 35^\circ$. $T_{wGB15^\circ} = 525$ °C for the high-energy tilt [110] GB with $\theta = 15^\circ$. Misorientation angles 15° and 35° nearly correspond to the maximum and minimum energies of [110] tilt GBs in Al [31]. It is noteworthy that the whole T_w interval for the [110] tilt GBs is about 15 times narrower than that for the all GBs in a polycrystal [28, 29]. Nevertheless, the CSL concept remains the most reliable one to describe the energy relations among the tilt GBs. Therefore, tilt GBs are most attractive for studying the GB faceting–roughening phenomena. In addition, the concidence GBs with misorientations close to the CSLs with low Σ also both fulfill Wolf and Rohrer criteria [22–27].

The idea of faceting–roughening transition for the free crystal surfaces was firstly proposed by Burton with coworkers [32]. They supposed that the free energy of an elementary step for each crystal surface decreases with increasing temperature due to the thermal fluctuations (configurational enthropy). As a result, for each singular crystal face a roughening temperature T_R exists. At T_R the free energy of a step at a surface becomes zero. As a result a flat surface becomes unstable above T_R. T_R is different for crystallographically different surfaces, the highest T_R corresponds to the surfaces with lowest Miller indices. For GBs the CSL (i.e. superlattice formed by the lattices of two grains) plays the role similar to a crystal lattice for the free surface. In other words, GB facets almost always coincide with CSL planes densely-packed with coincidence sites. GB faceting correlates to the so-called special GB properties close to the coincidence misorientations [2]. According to the idea of Burton with coworkers a surface facet has to gradually disappear by $T \to T_R$. However, due to huge experimental troubles the surface roughening (i.e. the vanishing of surface facets) was only directly observed for the first time more than 30 years later by growth of solid ^{4}He crystals [33]. The gradual disappearance of a GB facet has been observed for the first time in situ in measurement of the facet length in Zn [11].

In Refs. [9, 11] the last stage of the GB roughening was observed in situ, namely full disappearance of GB facet with increasing temperature. In this work the early stage of the GB roughening is observed, namely only the ridge between facets 1 and 2 becomes rounded (Fig. 2), the facets themselves remain flat. The studied GB migrated only above 350 °C. Below 350 °C the existing driving force was not high enough to force the GB to migrate. As a result, when the as-grown first-order ridge became rounded, it remained rounded in the whole temperature interval above 350 °C independent of heating or cooling. This means that the actual T_R value lies below 350 °C. Another important difference is the shape of the transition line between the remaining facet and rounded GB portion. In Refs. [9, 11] the break of first derivative $\partial y/\partial x$ of the GB shape was always present along the contact line between GB facet and rounded GB portion. In this work the transition between GB facets and the rounded GB portion is continuous, i.e. without a break in the derivative $\partial y/\partial x$. Similar smooth edges between flat surface facets and rounded rough regions have been observed in lead [34–38] and helium crystals [39]. Near a smooth edge the shape of the curved interface varies as (see Fig. 3, bottom part)

$$y = A(x - x_c)^\alpha + \text{higher order terms} \quad (1)$$

In our case the x axis was chosen parallel to the GB facet 1 (or facet 2). The y axis was perpendicular to the x axis. Both x and y axes lie in the sample plane. The position of the edge is given by x_0. The exponent α is a critical index. The curved GB regions close to facets 1 and 2 have been quantified and the respective values were calculated (Fig. 3). $\alpha = 1.7 \pm 0.07$ for 350 °C, $\alpha = 1.8 \pm 0.07$ for 375 °C and $\alpha = 1.9 \pm 0.07$ for 385 °C (Fig. 4).

In this work as well as in papers [3–13, 15, 16] we restricted ourselves to the phenomena observable in situ by optical microscopy, i.e. in the scale range between 1 μm and 1 mm. The details of GB faceting–roughening on the lower length scale are also very interesting. Unfortunately, their in situ observation using transmission electron microscopy (TEM) (as, for example, in the classical work of Hsieh and Balluffi [40]) is restricted by the fact that the equilibrium GB shape is seriously disturbed by the influence of both sample surfaces in the very thin TEM samples. There are two main models predicting α values. The mean field Andreev approach to the problem of crystal shape neglecting the fluctuations employs a Landau-type expansion of the free energy near the roughening transition of II order [41]. It delivers $\alpha = 2$, which is the signature of the square-law effective interaction between steps. The value $\alpha = 2$ has never been observed experimentally before. Pok-

rovsky and Talapov discussed the structure of a monolayer deposited on a periodic substrate incommensurate with the periodicity of the monolayer itself [42]. The steps occurring in the vicinal surface play the role of the boundaries separating individual commensurate regions in the model [42]. This theory predicts the $\alpha = 3/2$. Earlier, we found for the GB roughening in Mo α values which were distinctly below the mean-field prediction and are more consistent with Pokrovsky–Talapov (PT) theory [14].

In our case the α value slightly increases with increasing temperature and becomes rather close to the prediction $\alpha = 2$ of the Andreev mean-field theory. In the investigations of equilibrium shape of Pb particles it has been observed that the critical exponent α measured near a (111) facet is not completely universal and varies with azimuthal angle [36]. The α values distinctly fall into two groups: the first mean value is about 1.7 (similar to our value for Mo twin GB [14]) and the second is almost equal to 3/2. It has been shown that the α value depends on how the steps interact at the vicinal surface [36]. Scanning tunneling microscopy demonstrates that these steps can expose either (100) or (111) microfacets. Higher exponents are connected with (100) step ledges. In our case the difference between the measured α and theoretical values can be due to similar GB steps.

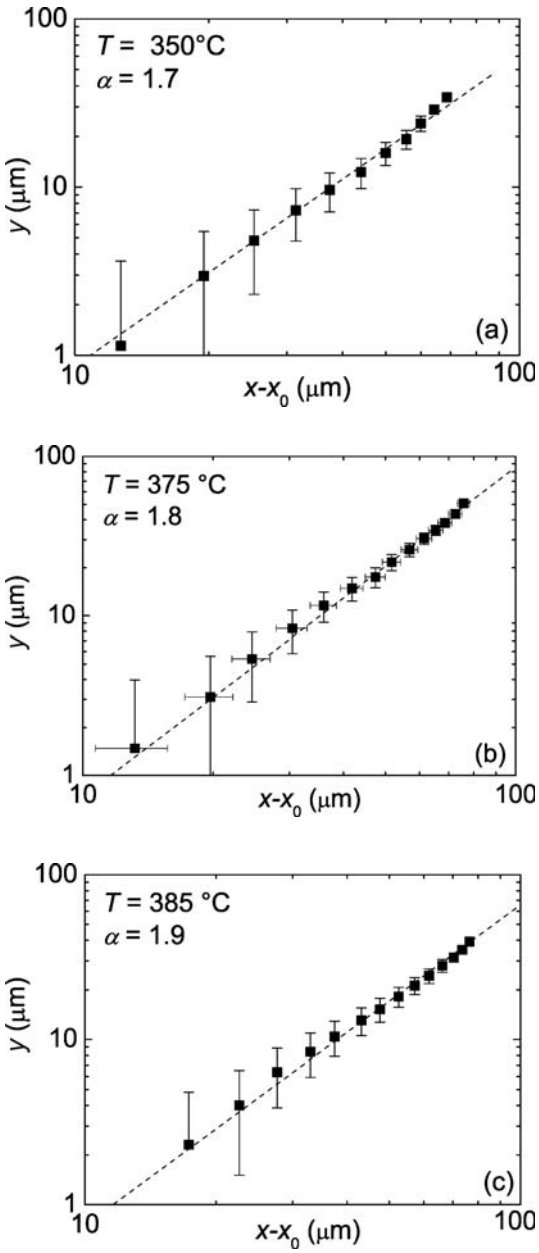


Fig. 4. Shape of the rounded GB portion close to the facets 1 and 2 (cf. Figs. 1 to 3) in scaling coordinates.

In this work we interpret the changes in GB morphology in Zn in terms of second-order roughening transformations. Previously, we observed the faceting-roughening phenomena in the moving GBs in Zn [9–13, 15, 16]. Exactly speaking, our samples contained two parallel straight stationary GBs connected by the GB half-loop. This GB half-loop moved during the annealing in the microscope hot stage driven by the capillary force. In such experiments the interplay of thermodynamic and kinetic influence on the shape of moving GB has to be carefully considered. In this work the sample geometry is much more advantageous. Namely, we analyzed the shape of the extremely slow migrating GB part which was practically pinned by the long stationary straight GB parts (see Fig. 1). In this case the kinetic influence on the GB shape analyzed in [10, 12] can be practically neglected.

Moreover, in our previous works we tried to quantify the shape of the moving curved GB portion which immediately contacted the straight stationary GB part using the scaling approach (as in Fig. 4). It has been observed that the slope of the lines plotted in scaling coordinates scattered in the very broad interval (between 3 and 10) even at constant temperature and GB migration rate. This is a definite indication that the shape of this GB part was controlled not by the faceting–roughening transformation but by the kinetic factors. In the moving GB half-loop, all ridges between the GB facets or GB facets and rounded GB portions were always of the first order (i.e. discontinuous) when observed by optical microscopy in the 1 μm to 1 mm scale [9–13, 15, 16]. Moreover, when GB facets disappeared with increasing temperature, the first order ridge continued to exist between two rounded GB portions [9].

As we already mentioned, the *c*/*a* ratio in Zn is temperature dependent [31]. Therefore, with increasing temperature, the deviation from the CCSLs shown in Fig. 3 would take place. This process is equivalent to the deviation from the ideal coincidence misorientation θ_Σ with changing $|\theta_\Sigma - \theta|$ in the materials with cubic lattice [2]. In some cases the change of *c*/*a* ratio in Zn with increasing temperature can lead to the transition from one CCSL to another [18–20] and can be the reason for changing faceting in Zn (as in the case observed in Ref. [12]. This reason for GB phase transformations is unique due to the irrational *c*/*a* and does not exist in materials with cubic lattices. In our case the temperature dependence of the *c*/*a* ratio can lead to fine changes in the strucructure of GB steps and, therefore, to the increase in α (Fig. 4).

5. Conclusions

The faceting of the almost stationary $[10\bar{1}0]$ tilt grain boundary (GB) with misorientation angle θ of 84° in the Zn bicrystal has been investigated in situ between 350 and 400 °C using polarized light. Two intersecting facets lie in the closely-packed planes of the constrained coincidence sites lattice with coincidence parameter $\Sigma = 15$. In the as-

grown sample flat GB facets form the sharp first-order ridge with the break of the first derivative $\partial y/\partial x$ of the GB shape. Above 350 °C the sharp first-order ridge is substituted by the smooth GB portion without a $\partial y/\partial x$ derivative break. The transformation of the first-order GB ridge into a continuous one has been observed for the first time. The critical parameter α increases from $\alpha = 1.7 \pm 0.07$ for 350 °C to $\alpha = 1.9 \pm 0.07$ for 385 °C. It approaches the $\alpha = 2$ value predicted in the mean-field Andreev model for the continuous surface roughening.

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