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Faceting and migration of twin grain boundaries in zinc

Dedicated to Professor Dr. Duk Yong Yoon on the occasion of his 65th birthday

Faceting and migration of twin tips in slightly deformed Zn $[11\bar{2}0]$ flat single crystals have been investigated. The stationary shape of the slowly migrating tip of the twin plate has been studied, and the migration rate has been measured between 592 and 692 K *in situ* using polarized light. Below 632 and above 682 K, the twin tip contains only one facet which is parallel to the $(\bar{1}10\bar{2})_2$ and $(1\bar{1}00)_1$ plane of the matrix single crystal, respectively. The non-physically high values of the apparent migration activation enthalpy between 632 and 682 K are discussed using the model of simultaneous migration of co-existing grain boundary facets based on the concept of weighted mean curvature. The experimentally determined temperature dependence of the twin tip mobility can be rationalized under the assumption that the migration of individual facets is characterized by identical activation enthalpies of 46.6 kJ/mol, but different pre-exponential factors (1.1×10^{-11} and 7×10^{-9} m^2/s, respectively).

Keywords: Grain boundaries; Faceting; Migration; Single crystals; Twins; Zinc

1. Introduction

Both faceting and roughening are well known for surfaces and interfaces, particularly, for grain boundaries (GBs) [1–3]. GB faceting mainly occurs close to the so-called coincidence misorientations, when the GB structure can be described using the coincidence site lattice (CSL). With decreasing temperature, the GB faceting can be observed for CSLs with lower and lower *density of coincidence sites* [4]. If the CSL is fixed, the less and less densely packed CSL *planes* appear with decreasing temperature in the faceted GB structure [5]. Yoon and coworkers have shown that the presence of GB faceting correlates also with the phenomenon of abnormal grain growth in alumina [6], stainless steel [7] and silver [8]. Above a certain temperature, faceted GBs cannot be observed in the microstructure and abnormal grain growth does not occur [5–8]. It is known that capillary-driven GB migration is the basic process controlling the grain growth in polycrystals. Therefore, for understanding of the interrelationship between the GB faceting and grain growth, the effect of the former on the GB migration has to be investigated in details. Unfortunately, in most of the works performed until now the isotropic approximation has been used in the investigations of capillary-driven GB migration [9]. In recent computer simulations, the fundamental role of GB anisotropy in determining the shape of a shrinking circular grain has been emphasized [10]. However, using the model of Ref. [10] for discussing of experimental data is problematic since it requires the full knowledge of the inclination dependence of GB energy. The best way to investigate the influence of GB faceting on the GB migration experimentally is to study the migration of individual fully faceted GBs.

2. Experimental details

Zn $[11\bar{2}0]$ flat single crystals were grown from Zn of 99.999 wt.% purity using a modified Bridgman technique [9, 11, 12]. Individual elongated twin plates exhibiting very uniform thickness were produced with the aid of slight deformation of single crystals. The produced twin plates were perpendicular to the surface of the sample. The $[11\bar{2}0]$ axes in both grains were also perpendicular to the surface of the sample. The parallel elongated sides of the twin plate are formed by the coherent symmetric twin grain boundaries (STGBs) of the type $(1\bar{1}02)_1\|(1\bar{1}0\bar{2})_2$. Due to its optical anisotropy, zinc allows one to study the shape of the GB with the aid of polarized light. The stationary shape of the slowly migrating tip of the twin plate was studied *in situ* in a hot stage of an optical microscope in the temperature range 592–692 K. In the same temperature interval, the GB migration rate has been measured and, subsequently, the GB mobility has been calculated. The samples were protected from oxidation by pure nitrogen atmosphere. Before measurements, the samples were electropolished in a $H_3PO_4 + C_2H_5OH$ solution. An additional polarization filter applied in the reflected beam permits to distinguish 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 colour video camera connected with the microscope and a video recorder. This method of studying the GB migration was originally developed in Refs. [11, 12]. It has been shown previously that the driving forces resulting from GB phase transitions (such as faceting) are usually rather low to cause a shape change of stationary GBs in a reasonable time [13–15]. On the contrary, migrating GBs are very sensitive to slightest variations of

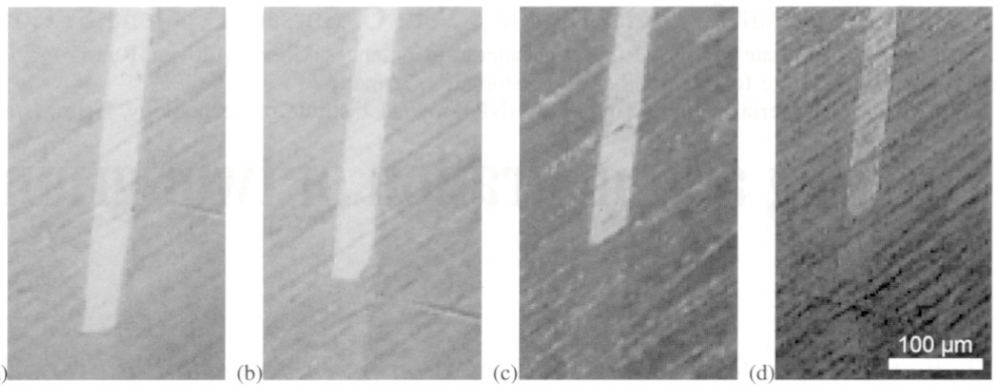

Fig. 1. Shape of the twin plates in Zn [11$\bar{2}$0] flat single crystals at various temperatures: (a) 632 K, (b) 652 K, (c) 682 K, (d) 692 K. Length of the scale bar is 100 μm.

the driving or braking forces [9], so that the steady-state shape characteristic for a given temperature can be readily established.

3. Results

The micrographs in Fig. 1 show the change of the shape of a GB at the tip of the twin plates in Zn at various temperatures. The shape of the twin tip differs drastically from the rounded shape of the GB half-loops in Zn bicrystals containing non-special GBs [9, 12, 16, 17]. At low temperature, the twin tip contains one flat facet 1 lying almost perpendicular to the STGBs (Fig. 1a). With increasing temperature a second facet 2 appears at the tip (Fig. 1b). Facet 2 has an angle of approximately 46° both with STGB and facet 1. When the temperature increases further, the length of facet 2 also increases and that of facet 1 decreases. Above a certain temperature, only facet 2 is present at the twin tip having rather sharp edges with STGBs (Fig. 1c). Close to the melting temperature of Zn, T_m, the edges at the intersection of the facets become rounded, the GB roughening transition proceeds (Fig. 1d). Previously, it has been shown that the angles between STGB and facets 1 and 2 do not depend on temperature [13]. The mean value of the angle for facet 1 is 84°, and for facet 2 is 46°. Below 632 K, only facet 1 is present in the twin tip. Between 632 and 677 K, facets 1 and 2 coexist. Above 677 K, only facet 1 is present. Since the ratio of the lattice spacings a and c [i. e., perpendicular and parallel to the (0001) plane] is irrational in Zn, exact CSLs exist for the Zn lattice only for rotations about the c axis. In all other cases, the so-called constrained CSLs (CCSLs) are used for the description of the coincidence in Zn GBs. Schemes in Fig. 2 demonstrate the CCSLs and crystallography of the observed facets. STGB is coherent and coincides with $(1\bar{1}0\bar{2})$ planes in both lattices (Fig. 2a). Facet 1 is nearly parallel to the $(\bar{1}10\bar{2})_2$ plane, and the coincidence is much poorer than in case of the coherent STGB (Fig. 2b). Facet 2 is nearly parallel to the $(1\bar{1}00)_1$ plane (Fig. 2c).

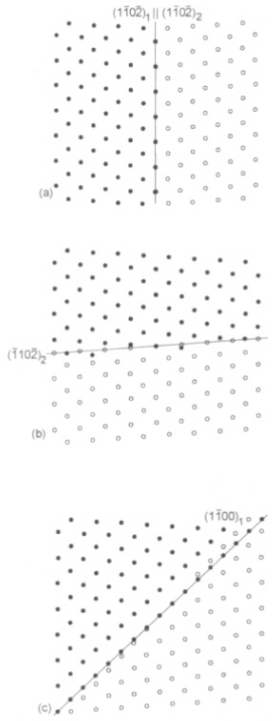

Fig. 2. Crystallographic features of the observed facets. (a) STGB $(1\bar{1}0\bar{2})_1 \| (1\bar{1}0\bar{2})_2$. (b) Facet 1 is nearly parallel to the $(\bar{1}10\bar{2})_2$ plane and has an angle of 84° with the STGB. (c) Facet 2 is nearly parallel to the $(1\bar{1}00)_1$ plane and has an angle of 46° with the STGB.

Figure 3 demonstrates the geometry of twin plate migration. The temperature dependence of the normalized lengths of facets 1 and 2 are shown in Fig. 4. Two typical dependences of the GB position on annealing time are shown in Fig. 5. The GB mobility has been calculated from the least-square linear fit of this dependence under the conditions of steady-state GB migration with a constant shape. In a previous work [13], the phase diagram for the faceting

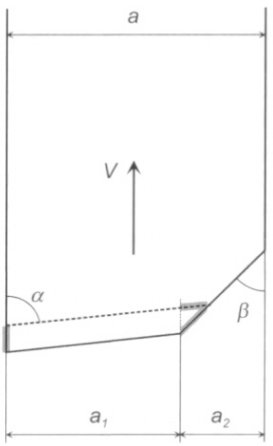

Fig. 3. Schematic diagram showing the geometry of twin plate migration. The new position of facet 1 after an infinitesimal displacement used in the calculation of its weighted mean curvature is shown by the dashed line. The newly created or annihilated sections of the interfaces that need to be taken into account in calculating the weighted mean curvature are marked in gray.

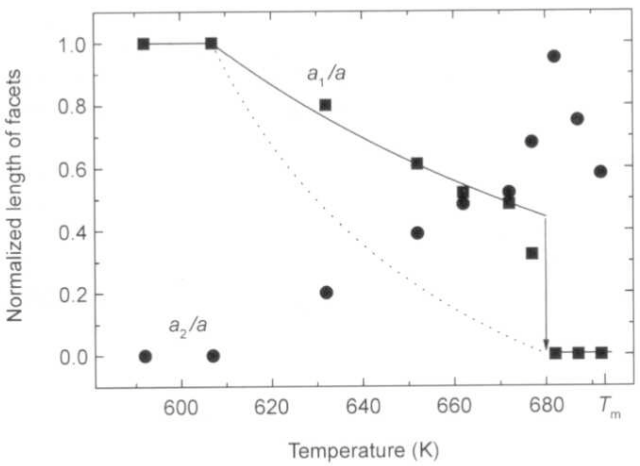

Fig. 4. Temperature dependence of the normalized length of the facet 1 (a_1/a, squares) and facet 2 (a_2/a, circles) of the twin plates in a Zn $[11\bar{2}0]$ flat single crystal. The dotted line represents a fit using Eqs. (6a) and (9) with $T_C = 607$ K and $A = 0.0274$. The solid line is plotted under the assumption that an abrupt decrease in the length of facet 1 occurs at 680 K (kinetic phase transition). In this case, $A = 0.01$.

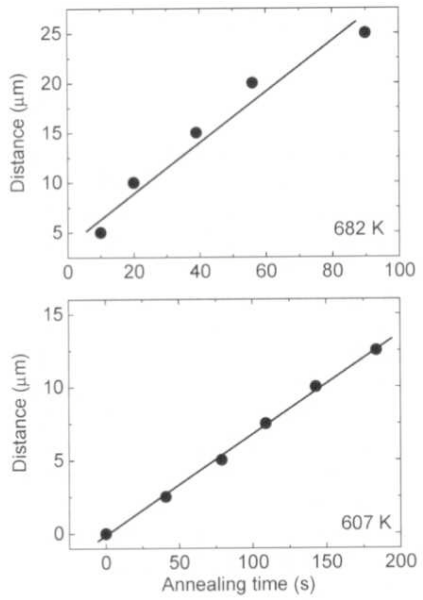

Fig. 5. Two typical dependences of the GB position on annealing time. The GB shape remains constant during stationary GB motion.

of twin GBs in Zn and schematic Wulff–Herring plots (polar plots describing the dependence of GB energy γ on boundary plane inclination) illustrating the possible energetic reasons for both GB faceting and roughening transitions have been constructed. There are two GB faceting phase transitions in this system: (1) Between 677 and 682 K facet 1 disappears. (2) Between 607 and 632 K facet 2 appears. STGB is stable at all temperatures. The temperature dependence of the migration rate of the twin plate tip is shown in Fig. 6. The temperature dependence is strongly non-linear (in Arrhenius co-ordinates) indicating that the twin tip migration cannot be described by a unique value of the migration activation enthalpy in the case in which the steady-state GB shape changes drastically with increasing temperature.

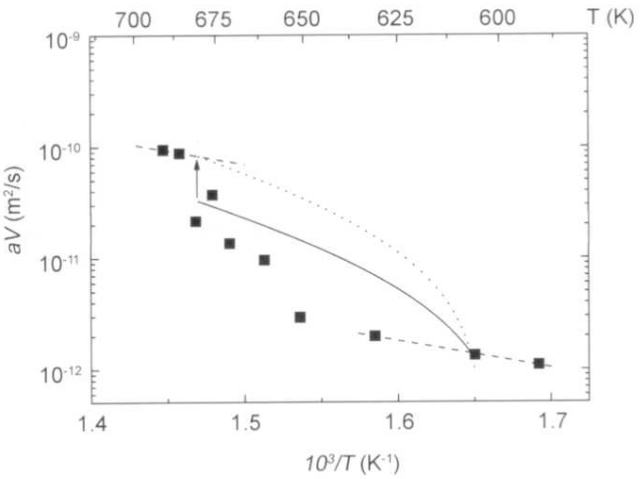

Fig. 6. Temperature dependence of the migration rate of the twin plate tip in a Zn $[11\bar{2}0]$ flat single crystal. The dashed lines represent Arrhenius-type fits to the low-and high-temperature data and characterize the intrinsic mobilities of the facets. The solid and dotted lines are plotted using Eqs. (6b) and (9) and have the same meaning as in Fig. 4.

4. Discussion

Our results clearly indicate that there is a strong effect of the GB faceting on the migration rate of the twin tip. To understand this phenomenon, we need to go beyond the classical models of isotropic migration of GBs [9]. The shape of a faceted GB migrating under the action of capillary forces can be understood in the framework of the weighted mean curvature concept [18]. In the case of isotropic behaviour, the velocity of the GB normal motion, v_n, is given by [19]

$$v_n = Mr \tag{1}$$

where M and r are the GB mobility and the radius of curvature, respectively. For a flat GB facet trapped in the local minimum of the GB Wulff–Herring plot, the velocity of normal motion is determined by the weighted mean curvature, r_m:

$$v_n = Mr_m \tag{2}$$

The latter is defined as a negative of the total interfacial energy change of the system after infinitesimal displacement of the given facet while all other interfaces are immobile, divided by the volume swept by the facet [18]. The facet mobility, M, depends on the atomic mechanisms of the facet motion. For example, under certain conditions, M can be inversely proportional to the facet length [20]. However, consideration of facet migration mechanisms is beyond the scope of the present work, and in what follows, the facet mobilities will be considered as phenomenological length-independent quantities that depend on temperature according to the Arrhenius law. For the receding twin considered in this work (Fig. 3), the weighted mean curvatures of facets 1 and 2 can be easily calculated:

$$r_{m,1} = \frac{\gamma_0 + G}{a_1} \tag{3a}$$

$$r_{m,2} = \frac{\gamma_0 - G}{a_2} \tag{3b}$$

where

$$G = \frac{\gamma_2 \sin\alpha - \gamma_1 \sin\beta}{\sin(\alpha - \beta)} \tag{4}$$

and γ_0, γ_1 and γ_2 are the interfacial energies of the STGB, facet 1, and facet 2, respectively. Taking into account Eq. (2) and noting that the normal velocities of the facets V_1 and V_2 are projections of the global velocity of twin shortening, V, onto the facet normals [9], we arrive at the following equations that give a full description of the geometry and kinetics of the system:

$$V_1 = \frac{M_1}{\sin\alpha}\frac{\gamma_0 + G}{a_1} \tag{5a}$$

$$V_2 = \frac{M_2}{\sin\beta}\frac{\gamma_0 - G}{a_2} \tag{5b}$$

$$a = a_1 + a_2 \tag{5c}$$

Equation (5c) is a trivial geometric relationship (see Fig. 3). The solution of Eqs. (5a)–(5c) can be easily found:

$$\frac{a_1}{a} = \frac{1}{1 + \dfrac{(\gamma_0 - G)\, M_2 \sin\alpha}{(\gamma_0 + G)\, M_1 \sin\beta}} \tag{6a}$$

$$aV = \frac{(\gamma_0 + G)}{\sin\alpha} M_1 + \frac{(\gamma_0 - G)}{\sin\beta} M_2 \tag{6b}$$

An interesting feature of Eq. (6a) is that the shape of the receding twin is determined by a combination of both thermodynamic (γ_0, G) and kinetic (M_1, M_2) parameters. It is known that the faceting geometry of a planar GB of fixed inclination is determined exclusively by the topology of the γ-plot, i. e., by purely thermodynamic factors [21]. At the same time, the shape of the growing or dissolving crystal is determined by the Wulff construction on the v_n (**n**) plot (polar plot of the growth velocity), i. e., by kinetic factors only. The receding twin investigated in this work represents an intermediate situation: while the shortening of the twin resembles the dissolution of the crystal in which kinetics plays a dominant role, the driving force for this shortening is of capillary origin, which introduces the dependence of shape on interfacial energies.

Equation (6b) shows that the effective mobility of the tip of the twin plate during concerted motion of facets 1 and 2 is a linear combination of their respective mobilities (weighted by interfacial energies). This means that the rate of twin plate shortening is determined by the mobility of the fastest of two facets. This is in apparent contradiction with the conventional wisdom claiming that the slowest element of microstructure is responsible for its overall dynamics [10]. However, a closer look at Eqs. (5a, b) reveals that the slow facet 1 can keep pace with the highly mobile facet 2 by decreasing its length a_1. Shorter facet length means also a higher driving force for migration, which to some extent compensates the facet slowness.

The analysis of Eqs. (3) and (6a) shows that, depending on the values of G and γ_0, the following morphologies of the migrating twin plate tip are possible:

$G > \gamma_0$: Only facet 1 is stable. (7a)

$-\gamma_0 < G < \gamma_0$ Both facets are stable. (7b)

$G < -\gamma_0$: Only facet 2 is stable. (7c)

The analyses of weighted mean curvatures for the cases when facet 1 or 2 alone is stable, performed in full analogy with the above analysis of concerted motion, lead to the following relationships for the twin plate tip migration rates:

$$aV = \frac{2\gamma_0}{\sin\alpha} M_1\text{: Only facet 1 is stable.} \tag{8a}$$

$$aV = \frac{2\gamma_0}{\sin\alpha} M_1 + (\gamma_0 - G)\left\{\frac{M_2}{\sin\beta} - \frac{M_1}{\sin\alpha}\right\}:$$

Both facets are stable. (8b)

$$aV = \frac{2\gamma_0}{\sin\beta} M_2\text{: Only facet 2 is stable.} \tag{8c}$$

Equation (8b) represents Eq. (6b) rewritten in a form that is more convenient for further analysis.

The ordered structure of the STGB (see Fig. 2a) implies that its specific Gibbs excess entropy is small and, hence, the temperature dependence of γ_0 is very weak. Therefore, the changes in the relative values of G and γ_0 in the narrow temperature interval of about 100 K can be attributed to facets 1 and 2 that exhibit an atomic structure less ordered than that of the STGB (see Figs. 2b, c). Neglecting the dependence of specific the Gibbs excess entropies on temperature, the $G(T)$ dependence can be represented in the form:

$$G = \gamma_0[1 - A(T - T_C)] \tag{9}$$

Above T_C, facet 2 appears in addition to facet 1. Assuming $T_C = 607$ K and $A \approx 0.0274$ leads to the fulfillment of Eqs. (7a–c) in the temperature ranges $T < 607$ K, 607 K < $T < 680$ K, and $T > 680$ K, respectively. Therefore, the thermodynamic criterion Eq. (9) correctly describes the morphology of the twin plate tip in the whole investigated range of temperatures (see Fig. 4). For a quantitative description of the experimental data by the suggested theory, we will fit the mobility data of Fig. 6 for the three lowest temperatures studied (at which facet 2 is either not appearing in morphology or is very short) by the Arrhenius law: $aV_1 = 1.41 \times 10^{-8}$ m^2/s $\times$ exp (−46.5 kJ mol^{-1}/RT), where R is the gas constant. The obtained activation enthalpy, 46.5 kJ/mol, for the migration of facet 1 is very close to the activation enthalpy of Zn self-diffusion along random GBs, 54.4 kJ/mol [22]. Therefore, it is reasonable to assume that the migration of facet 1 is controlled by GB diffusion. If facet 2 migrates by the same diffusion mechanism the data of Fig. 6 for the two highest temperatures studied (at which facet 1 does not appear in morphology) can be fitted by the Arrhenius law with the same value of the activation enthalpy: $aV_2 = 3.1 \times 10^{-7}$ m^2/s $\times$ exp (−46.5 kJ mol^{-1}/RT). The corresponding values of the mobilities can be obtained from Eqs. (8a, c):

$$\gamma_0 M_1 \approx 7 \times 10^{-9}\ \text{m}^2/\text{s} \times \exp(-46.5\ \text{kJ mol}^{-1}/RT) \tag{10a}$$

$$\gamma_0 M_2 \approx 1.1 \times 10^{-9}\ \text{m}^2/\text{s} \times \exp(-46.5\ \text{kJ mol}^{-1}/RT) \tag{10b}$$

With the known facet mobilities [Eqs. (10a) and (10b)] and the thermodynamic factor G [Eq. (9)] the dependences $a_1(T)$ and $aV(T)$ for the temperature interval 607 K < $T_C < 680$ K of concerted motion of two facets can be calculated with the help of Eqs. (6). The results of calculations are shown in Figs. 4 and 6 by the dotted lines. The theory predicts a continuous hyperbolic variation of the length of

facet 1, a_1, from the maximal value $a_1 = a$ at $T_C = 607$ K down to zero at 680 K. The disagreement between predicted and experimentally measured dependences is alarming. For the twin plate tip velocity, aV, the theory predicts a continuous transition between the two Arrhenius dependences characterizing the migration processes of facets 1 and 2 (Fig. 6). The rate of migration velocity variation with temperature is maximal at T_C. Also in this case, the agreement between theory and experiment is quite poor. There are several possible reasons for this discrepancy:

(i) The faceting–faceting transition at $T_C = 607$ K is of second or higher order and, consequently, the dependence of G [see Eqs. (4) and (9)] on temperature is nonlinear;
(ii) The activation enthalpy in the Arrhenius equation for M_2 is different from that of M_1;
(iii) The facet mobilities depend on their length;
(iv) The roughening of the twin tip above 680 K has a major influence on the migration rate.

All four hypotheses allow a better fitting of the experimental data, however, at the expense of introducing new fitting parameters. In what follows, we will show that a better agreement between theory and experiment is possible without introducing any new parameters. Indeed, Eq. (6) just describes the relative lengths of the facets and the velocity of their concerted motion and do not actually force the twin plate tip to be composed of two facets at $T > T_C$. Even at these temperatures and with the condition Eq. (7b) fulfilled, the twin tip can continue migrating as facet 1 or can fully transform into facet 2. The only reasonable criterion for selection of the actual morphology of the tip is the requirement that this morphology should provide the maximal rate of decrease of the Gibbs energy during migration. A similar principle was successfully applied to the problem of the morphology of the discontinuous precipitation reaction product [23], however, its rigorous proof has yet to be found. Cahn [23] pointed out that "it should be possible and would be desirable to justify this assumption on more basic grounds, but how this should be done has so far eluded the author". In the case of the twin plate considered in this work, the rate of Gibbs energy decrease upon migration is directly proportional to the migration velocity of the twin tip. A close look at Eq. (8) shows that if the condition

$$\frac{M_2}{\sin\beta} > \frac{M_1}{\sin\alpha} \tag{11}$$

and condition Eq. (7b) are fulfilled, the migration velocity of the twin tip composed of facet 2 alone [Eq. (8c)] is always higher than the migration velocity of the tip composed of two facets [Eq. (8b)]. This means that at the temperature T_C both thermodynamic [Eq. (9)] and kinetic transitions in the morphology of the twin tip should occur simultaneously, with facet 2 replacing completely facet 1. However, the kinetic transformation of the twin tip morphology may require some overheating, in full analogy with the usual thermodynamic phase transformations that often require some overheating to proceed. With this scenario in mind, we can argue that while the appearance of facet 2 at T_C is of thermodynamic nature [and, hence, the function G should be described by Eq. (9)], the disappearance of facet 1 at 680 K is of kinetic nature and the condition (7c) has not to be fulfilled at this temperature. The corresponding dependences $a_1(T)$ and $aV(T)$, calculated for $A = 0.01$, are shown in Figs. 4 and 6 by bold solid lines. At 680 K, the kinetic transition in the morphology occurs and facet 1 abruptly disappears. The dependence $a_1(T)$ exhibits an excellent agreement with the experimental data. For the $aV(T)$-dependence, the correspondence between theory and experiment is reasonable, and certainly better than for the theory based on the assumption that the transition at 680 K is of thermodynamic nature.

In conclusion, we would like to note that the activation enthalpy for twin tip migration, formally determined from the Arrhenius plot in the temperature range 607 K $< T_C <$ 680 K, would be unreasonably high. Our theory clearly shows that this is because in this range of temperatures the actual mobility of the twin tip is a weighted average of the mobilities of two facets. The resulting temperature dependence is very strong in spite of the fact that the activation enthalpy for migration of each of the two facets is quite normal. It is well possible that high activation enthalpies for GB migration often reported in the literature are actually related to the faceting of these boundaries on the microscale [10, 24].

5. Conclusions

We studied the migration kinetics of the tip of a twin plate in the temperature range 592–692 K. The twin plate was produced by a slight plastic deformation of a Zn single crystal. Parallel elongated sides of the twin plate formed by the coherent symmetric twin $(1\bar{1}0\bar{2})_1 \parallel (\bar{1}10\bar{2})_2$ grain boundary facets remained immobile at all temperatures studied. Below 607 K, the migrating twin tip contained only one plane facet 1 which is nearly parallel to the $(\bar{1}10\bar{2})_2$ plane; in the temperature range 607–680 K, facet 1 at the twin tip co-existed with facet 2 which is nearly parallel to the $(1\bar{1}00)_1$ plane, and above 680 K, only facet 2 was present at the tip. The temperature dependence of the twin tip migration rate exhibited a strongly non-Arrhenius-type behaviour. However, it could be rationalized with the aid of the theory of faceted grain boundary migration, based on the concept of the weighted mean curvature. The theory predicts that in the temperature range 607–680 K of coexistence of two facets, the integral mobility of the twin tip is a weighted average of the mobilities of two facets. The experimentally determined activation enthalpy for migration of facet 1, 46.5 kJ/mol, is very close to the activation enthalpy of grain boundary self-diffusion in Zn, which indicates that grain boundary diffusion plays an important role in migration of flat facets. We assumed that the activation enthalpy for migration of facet 2 is identical with that of facet 1, though not enough experimental data were available to support this assumption. Without introducing any further adjustable parameters, we considered two alternative hypotheses:

1. The observed changes in twin tip morphology are related to the relative changes of interfacial energies;
2. The nucleation of facet 2 at 607 K is governed by interfacial energies, while the disappearance of facet 1 at 680 K is determined by the thrust of the system to increase the rate of the Gibbs energy reduction (kinetic phase transformation).

We demonstrated that the second hypothesis allows achieving a better agreement between the theory predictions and experimental data. Finally, it was argued that the high acti-

vation enthalpies reported in some studies for grain boundary migration may be attributed to the boundary faceting on a microscale.

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