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Drag effect of triple junctions on grain boundary and grain growth kinetics in aluminium

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Abstract

The motion and geometry of connected 2D grain boundary systems with triple junctions in aluminium was investigated in situ in an SEM with a specially designed heating stage. The results show that triple junctions can have a marked influence on grain boundary motion and grain growth kinetics. The grain area change with annealing time was recorded in situ in the SEM. An analysis of the experimental data reveals that there is no unique relationship between growth rate and the number *n* of grain sides (von Neumann–Mullins relation). This is attributed to the effect of triple junction drag on grain growth.

Keywords: Grain boundary; Triple junction; Mobility; von Neumann–Mullins relation; Grain growth

1. Introduction

Grain boundaries and grain boundary junctions are the basic microstructural components of polycrystalline materials. Although the number of grain boundary junctions is of the same order of magnitude as the number of grain boundaries, the grain growth kinetics is usually associated only with grain boundary kinetics. The grain boundaries, therefore, are traditionally conceived to dominate the grain growth kinetics. However, recent studies, both experiments and computer simulations, substantiate that under special conditions triple junctions can have an influence on the motion of connected grain boundaries [1–4]. While intensive research on triple junctions in tricrystals was conducted in the past years, only little attention was paid to triple junction kinetics in polycrystals. We report on an experimental study of the behavior of triple junctions in polycrystals in quasi-2D systems.

2. Motion of connected grain boundaries

To determine the effect of triple junctions on the kinetics of grain growth quantitatively, the mobility of triple junctions has to be measured. For this, the steady state motion of a boundary system with a triple junction is required, in which the curved boundaries maintain their shape during motion so that the entire system moves with the same constant velocity. Two geometrical configurations of 2D boundary systems are shown in Fig. 1. In one configuration (Fig. 1(a)), one of the boundaries is extended during motion of the junction, like during the shrinkage of small grains in polycrystalline aggregate. In the other configuration (Fig. 1(b)), one grain boundary is eliminated, like during the growth of large grains. Assuming that all boundaries of the connected grain boundary system possess equal surface

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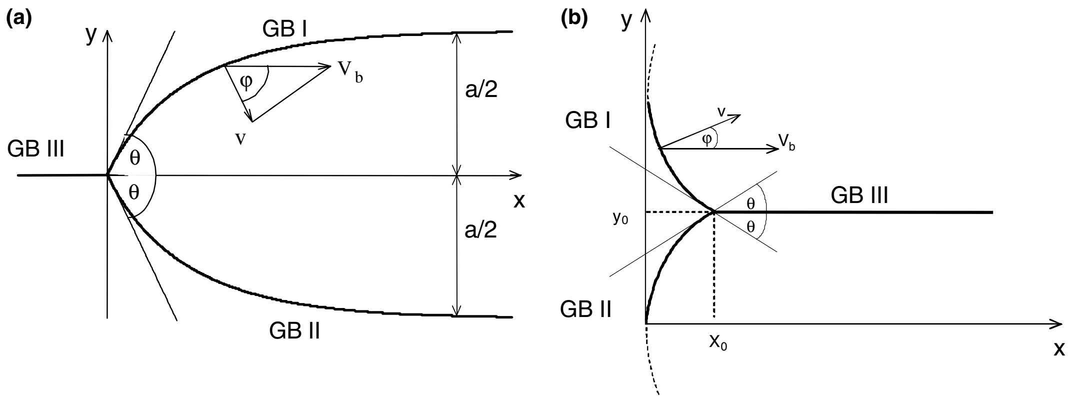

Fig. 1. The two basic configurations of a grain boundary system with triple junction during steady-state motion.

tension and mobility, irrespective of the misorientation and the spatial orientation of the boundaries, the shape and velocity of the moving grain boundaries with triple junction can be calculated analytically [5,6]. The curved boundaries in the first configuration (Fig. 1(a)) experience a force to move toward their center of curvature which results in a constant horizontal velocity V_b, which can be derived from the shape function of the boundaries as [5,6]

$$V_b = \frac{2\theta m_b \sigma}{a}, \tag{1}$$

where θ is half the dihedral contact angle at the triple junction, m_b the boundary mobility, σ the grain boundary surface tension and a the thickness of the shrinking grain. On the other hand, the triple junction will experience a force to move if the three boundary surface tensions acting on it do not add up to zero, or

$$V_{tj} = m_{tj}\sigma(2\cos\theta - 1). \tag{2}$$

Since both boundaries and triple junction have to move jointly, i.e., with the same speed, we obtain from Eqs. (1) and (2)

$$\frac{2\theta}{2\cos\theta - 1} = \frac{m_{tj}a}{m_b} = \Lambda_S. \tag{3}$$

For the second boundary configuration shown in Fig. 1(b), we obtain from the same consideration [9]

$$\Lambda_G = \frac{m_{tj}x_0}{m_b} = -\frac{\ln\sin\theta}{1 - 2\cos\theta}, \tag{4}$$

where x_0 is a characteristic lateral dimension of the grain, which is comparable to the grain size.

Eqs. (3) and (4) define the criterion Λ which describes the influence of the triple junction on the motion of grain boundaries [5,6]. In case of the configuration in Fig. 1(a), for $\Lambda_S \to 0$, the angle θ tends to zero and the steady state velocity is entirely controlled by the mobility of the junction. For $\Lambda_S \to \infty$ the junction easily adjusts to the motion of the boundary system, and the angle θ approaches its equilibrium value $\pi/3$. For the other configuration (Fig. 1(b)), again $\theta \to \pi/3$ if the boundary mobility determines the kinetics of the system, i.e., $\Lambda_G \to \infty$. When a low mobility of the triple junction starts to drag the motion of the boundary system, Λ decreases, and the steady state value of the angle θ increases to approach $\pi/2$.

The state of motion of the entire grain boundary system can therefore be determined experimentally for both configurations by only measuring the contact angle θ.

3. Effect of triple junctions on grain growth in polycrystals

In the classical approach [6–8] the rate of grain area change of a 2D granular system is described by the von Neumann–Mullins relation, which constitutes a basis for practically all theoretical and experimental investigations and computer simulations of microstructural evolution in 2D polycrystals in the course of grain growth. According to this approach the rate of the grain area change is given by the equation

$$\frac{dS}{dt} = -\frac{A_b\pi}{3}(n - 6), \tag{5}$$

where S is the grain area, $A_b = m_b\sigma$ is the reduced grain boundary mobility and n is the number of triple junctions for the respective grain, i.e., the topological class of the grain. Thus, the rate of area change is independent of the shape of the boundaries and determined by the topological class n only. Grains with $n > 6$ will grow, and those with $n < 6$ will disappear [7]. Fundamental assumptions of the respective model are: (1) all grain boundaries possess equal mobilities and surface tensions, irrespective of their misorientation and the crystallographic orientations of the boundaries; (2) the mobility of a grain boundary is independent of its velocity; (3) triple junctions do not affect grain boundary motion; therefore, the contact angles at triple junctions are always in equilibrium and, due to the first assumption, are equal to 120.

However, the latter assumption of the theory is at variance with the reported experimental evidence of a finite mobility of triple junctions and the corresponding marked deviation of the vertex angle θ from its equilibrium value. This requires to include the influence of triple junction drag on grain boundary motion for a consideration of the grain growth kinetics.

When the triple junctions exert a drag on the adjoining grain boundaries owing to their low mobility, it can be derived [9]

for $n < 6$

$$\frac{\mathrm{d}S}{\mathrm{d}t} = -\frac{A_\mathrm{b}}{1 + \frac{2\cos\theta - 1}{2\theta}}[2\pi - n(\pi - 2\theta)] \tag{6}$$

and for $n > 6$

$$\frac{\mathrm{d}S}{\mathrm{d}t} = \frac{A_\mathrm{b}[n(\pi - 2\theta) - 2\pi]}{1 - \frac{1 - 2\cos\theta}{\ln(\sin\theta)}}. \tag{7}$$

For both cases, $n < 6$ and $n > 6$, the expressions (6) and (7) converge to the von Neumann–Mullins relation (Eq. (1)) when Λ tends to infinity, i.e., θ approaches $\pi/3$. It is, however, seen from Eqs. (6) and (7) that in case of triple junction drag the growth rate $\mathrm{d}S/\mathrm{d}t$ of a grain of topological class n is not uniquely defined by n anymore, as predicted by the von Neumann–Mullins relation, but by θ, respectively, Λ, as well.

4. Experimental procedure

The current study addressed the effect of triple junctions on grain growth in polycrystalline Al. For this the velocity of individual triple junctions, their vertex angles and the growth rate of individual grains were measured. The used material was high purity aluminum (99.9999%) doped with 10 ppm magnesium to slow down the growth kinetics and to facilitate in situ grain growth measurements.

The experiments were carried out on polycrystalline Al sheet of thickness 120 μm. The material was recrystallised to a mean grain size of 150 μm. This allowed to realise an approximately 2D microstructure.

EBSD measurements were carried out before and after in situ grain growth experiments to determine the misorientation of the grains and to confirm the quasi-two-dimensionality of the grain structure. These EBSD checks were necessary to ascertain that no grains from beneath surfaced and interfered with the measurement. The crystallographic characteristics of the junctions studied are given in Table 1.

The in situ experiments were conducted in a JEOL JSM820 containing a specially designed heating stage. The experiments were performed in a temperature range from 250 to 300 °C. The SEM was upgraded by a digital image scanning system, which collected the images and saved them in AVI format. In post-processing the images were analysed by a special routine that measured the displacement of the triple junctions and the vertex angles at the triple junctions.

Table 1
Misorientations of three contiguous grains at investigated junctions

Junction	GB I	GB II	GB III
TP-S2	19°$[0 2 \bar{1}]$	43°$[\bar{4} 3 \bar{3}]$	39°$[\bar{2} \bar{1} \bar{2}]$
TP-S3	35°$[3 1 0]$	53°$[\bar{4} \bar{3} \bar{4}]$	46°$[\bar{1} \bar{1} \bar{2}]$
TP-G1	28°$[\bar{2} \bar{2} \bar{3}]$	31°$[\bar{1} 0 0]$	30°$[\bar{1} 2 \bar{3}]$
TP-G3	26°$[0 1 4]$	23°$[\bar{1} 0 0]$	12°$[0 \bar{3} \bar{2}]$

5. Results and discussion

5.1. Motion of individual grain boundary triple junctions

In order to analyse triple junction kinetics in a polycrystalline material the junctions have to satisfy the following criteria. First, the junctions have to be formed by grain boundaries with the same energies and mobilities. This holds for random high angle grain boundaries where the properties vary only slightly with changing misorientation. In this case the connected grain boundary system can be regarded as uniform. Second, the junction geometry and the direction of motion have to be of the same configuration as assumed in theory (Fig. 1). Third, the triple junction must move in a steady state. Only when these criteria are fulfilled the triple junction can be used for analysis, and the dimensionless criterion Λ can be calculated.

The motion of four selected boundary systems with junction (Table 1) which fulfilled the criteria mentioned above, were measured and analyzed. Junctions TP-S2 and TP-S3 moved in configuration of Fig. 1(a), while junctions TP-G1 and TP-G3 demonstrated steady-state motion in configuration of Fig. 1(b). The change of the vertex angle 2θ with temperature was measured, and the criterion Λ was calculated according to Eqs. (3) and (4).

The measured displacement over time at 250 and 300 °C for junction TP-S2 is shown in Fig. 2. The junction displacement increased in proportion to time for a constant temperature. Hence, the triple junction moved in steady-state, and its velocity for each temperature could be calculated (Table 2). With rising temperature the velocity increased. At 300 °C the velocity suddenly increased after about 350 s (Fig. 2(a)). This increase could be associated with a change of the number of triple junctions of the grain. First the grain had $n = 4$ triple junctions but after some time the number decreased to $n = 3$, and the velocity of the considered triple junction increased. The corresponding vertex angle measurements are shown in Fig. 2(b). The angles

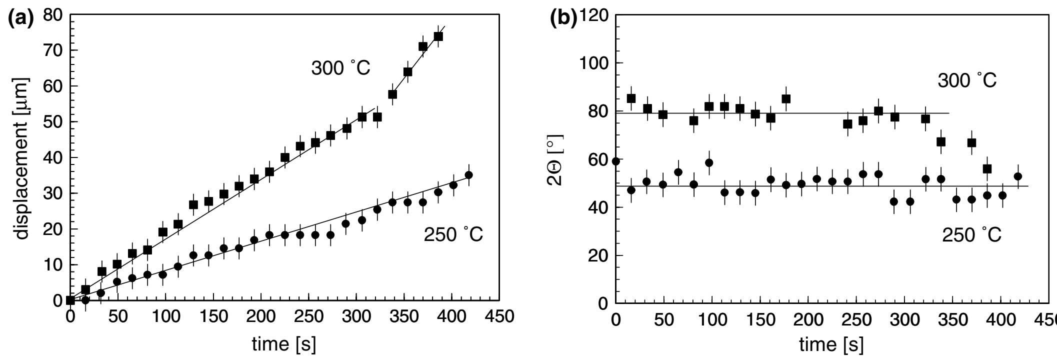

Fig. 2. (a) Displacement and (b) vertex angle 2θ versus time for triple junction TP-S2 with a configuration of Fig. 1(a) at 250 and 300 °C.

Table 2
Parameters of motion of the investigated boundary systems with triple junction

Junction	T (°C)	v (µm/s)	2θ	Λ
TP-S2	250	0.08	49	1
	300	0.17 (0.35)	79	2
TP-S3	250	0.10	82	3
	300	0.15	116	37
TP-G1	250	0.05	127	0.9
	300	0.08	127	0.9
TP-G3	300	0.15	129	0.7
	320	0.35	124	2

remained constant over the measured time interval (Table 2). The measured values of the angle 2θ for junction TP-S2 were 49° at 250 °C and 79° at 300 °C, which in both cases reflected a conspicuous deviation from the equilibrium angle of 120°. At 300 °C the angle decreased towards the end of the measurement owing to the change in the number of sides of this grain as mentioned above. The criterion Λ was remarkably low for the whole investigated temperature regime (250 °C: $\Lambda_S = 1$, 300 °C: $\Lambda_S = 2$), but it increased slightly with rising temperature. In essence, in this temperature interval the grain boundary system was strongly dragged by the triple junction.

For sample TP-S3 the angles were measured with 82° at 250 °C and 116° at 300 °C, hence only at 250 °C there was a noticeable deviation from the equilibrium angle of 120°. With increasing temperature the value 2θ increased as well and approached 120° (Fig. 3). This becomes obvious from the criterion Λ, which had a low value at 250 °C, namely $\Lambda_S = 3$, but increased significantly with increasing temperature up to $\Lambda_S = 37$. As a result, in this sample the triple junction had a drag effect on the grain boundary system only at the lower temperature.

The measured displacements and vertex angles over time at two different temperatures for junctions of grains with $n > 6$ are shown in Fig. 4. Both measured boundary systems possess the geometry shown in Fig. 1(b). At a given temperature boundaries and junctions moved at a constant rate. The calculated velocity rose with increasing temperature (Table 2). For the system TP-G1 at both temperatures (250 and 300 °C) the angle 2θ was measured to be about 127° with $\Lambda_G = 0.9$. The vertex angle for the system TP-G3 was 129° ($\Lambda_G = 0.7$) at 300 °C and 124° ($\Lambda_G = 2$) at 320 °C, respectively. The dependency θ (Λ) for the motion of the boundary system in this configuration (Fig. 1(b)) noticeably changed in a much smaller Λ-interval than in the case of the configuration of Fig. 1(a) as obvious from Fig. 5. While the Λ – values of TP-S2 for 250 and 300 °C and TP-S3 for 250 °C reflected substantial junction drag ($\Lambda \sim 1$), the triple junc-

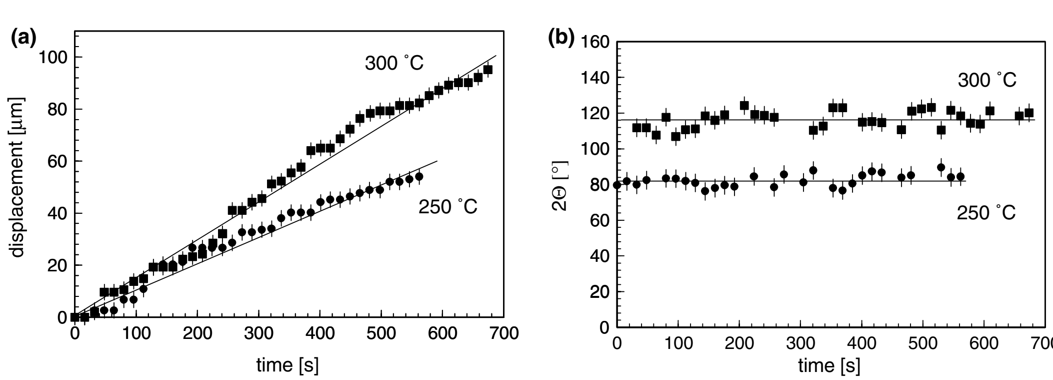

Fig. 3. (a) Displacement and (b) vertex angle 2θ versus time for triple junction TP-S3 with a configuration of Fig. 1(a) at 250 and 300 °C.

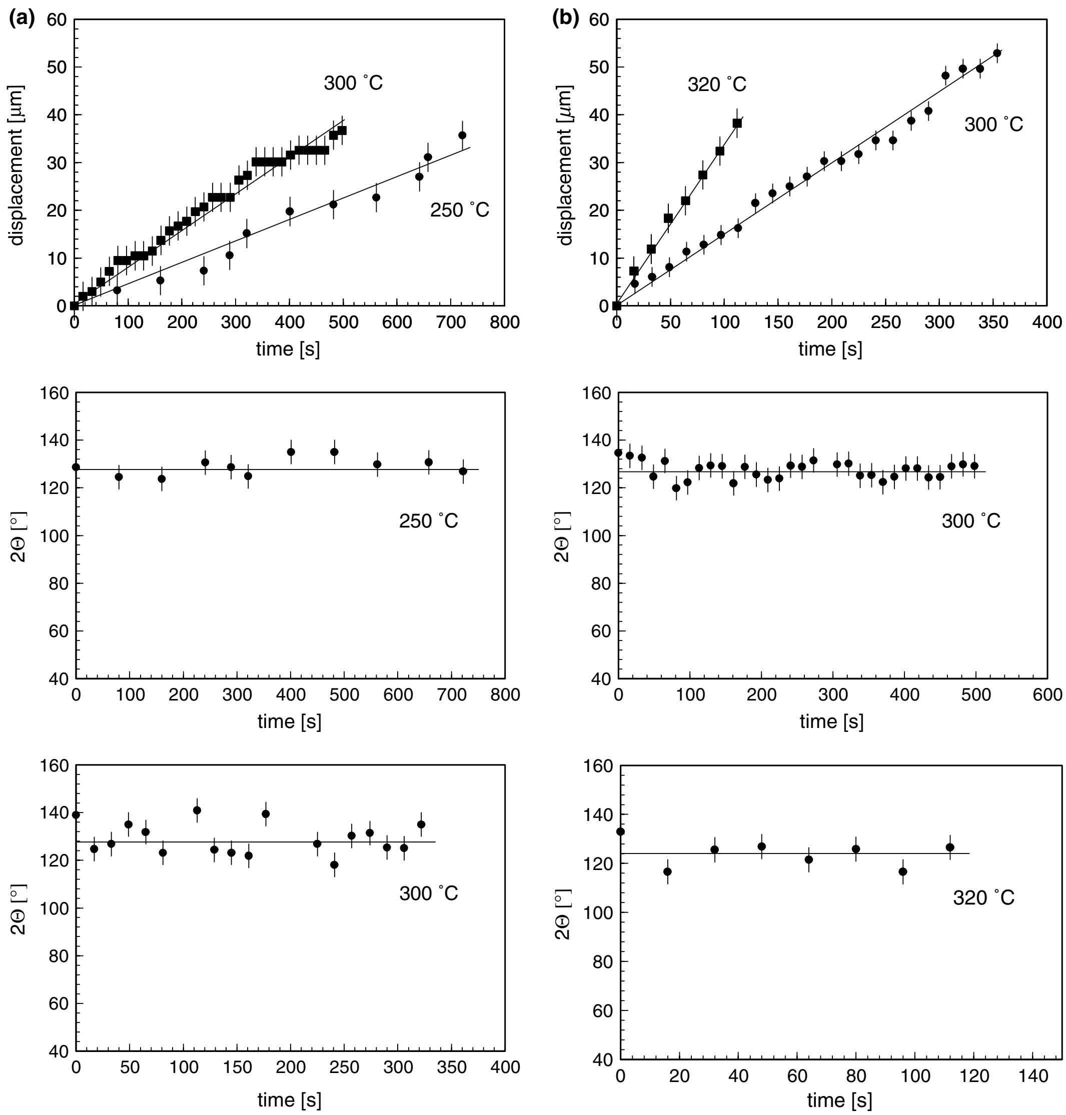

Fig. 4. (a) Displacement and (b) vertex angle 2θ versus time for triple junction (a) TP-G1 and (b) TP-G3 with a configuration of Fig. 1(b) at two different temperatures.

tion TP-S3 had no influence anymore on boundary migration ($\Lambda \gg 1$) at 300 C. The Λ-values for the TP-G1 and TP-G3 were all in the regime ($\Lambda \sim 1$), where they had a dragging influence on the motion of the boundaries.

From these measurements the conclusion can be drawn that with increasing temperature the triple junctions lose their impact on boundary motion. It is noted that the current investigation did not address the cause of a low triple junction mobility, which may be affected by its structure and segregation in the presence of impurities. To accurately measure the effect of impurity drag on grain boundary and junction mobility it is necessary to determine triple line and grain boundary segregation on tricrystals containing the same set of grain boundaries, for instance by atom probe tomography. Such experiments are currently in progress.

5.2. Grain growth kinetics

Another measure to assess the influence of triple junction drag on grain growth is the rate of area change of individual grains. For such analysis the grains have to satisfy the following criteria. First, the grain boundaries have to move toward their center of curvature. Second, the triple junctions must move in steady state. Third, a triple junction was not allowed to appear or disappear. In such case the grain was regarded as a new grain and analyzed separately.

The rate of the grain area change dS/dt was measured at three different temperatures for a number of grains with various number of sides n (topological class of the grain) from $n = 3$ up to $n = 10$ (Tables 3 and 4). The results in Fig. 6 show that at a constant temperature the grain area S decreased or increased in proportion to

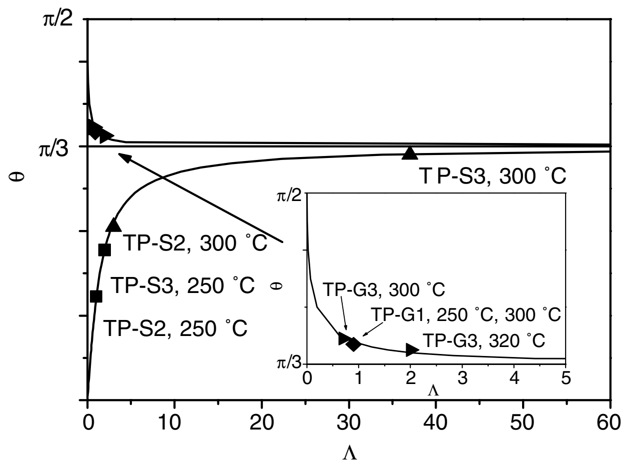

Fig. 5. Vertex angle θ versus criterion Λ. The symbols denote the measured values for the analyzed junctions, the lines are calculated according to Eqs. (3) and (4).

Table 3
Measured rate of area change at 250 and 320 °C and calculated parameter Λ for grains with different topological class n

Grain	n	dS/dt (10^{-12} m^2/s)	Λ
250 °C			
15_K5	3	−0.37	1.1
15_K6	3	−10.74	1.3
15_K6	5	−15.0	12.5
16_KB	5	0.13	10.4
16_KD	5	4.24	9.8
15_K4	6	4.18	
16_KC	7	9.72	0.36
16_KE	7	−0.71	0.22
15_K2	7	−1.06	0.21
16_KA	8	11.09	0.19
15_K2	8	−0.29	0.12
320 °C			
10_KE	3	−5.75	1.2
10_KG		−151.8	18.4
10_KE	4	0.359	3.5
10_KB		−47.5	5.2
10_KCA		−73	6.5
10_KG		−45.8	20.2
10_KE	5	−68.6	34.3
10_KC		−1.96	10.6
10_KB		−62.6	29.1
10_KU	6	−3.4	
10_KC		7.8	
10_KD	9	36.8	0.244

Table 4
Measured rate of area change at 300 C and calculated parameter Λ for grains with different topological class n

Grain	n	dS/dt (10^{-12} m^2/s)	Λ
14_K5		−8.2	1.2
14_K6	3	−3.2	1.2
14_K7		−74.4	2.3
15_KD		−6.1	1.2
10_KT		−12.0	3.8
10_KN		−105.0	9.2
10_KP		−13.0	3.9
14_K5		−6.0	3.6
14_K6	4	−151.4	18.1
15_KB		−4.7	3.6
15_KC		−14.0	3.8
16_KB		−4.4	3.6
16_KD		0.1	3.5
15_KA		−1.2	10.4
15_KB		−10.0	11.7
16_KB	5	−0.9	10.5
16_KC		3.9	10.0
16_KE		0.2	10.4
13_K4		−5.9	11.1
10_KT		−7.8	11.41
10_KO		−78.11	47.27
10_KQ		4.7	9.84
10_KR	5	−6.6	11.25
14_K6		−14.2	12.37
14_K7		−98.9	224.0
10_KH		−9.5	
10_KR		−8.0	
14_K9		−16.2	
15_KR	6	−6.6	
13_K3		3.0	
16_KE		3.0	
10_KD		110.0	117.98
14_K6	7	1.8	0.25
16_KC		−25.7	
16_KA	8	11.5	0.19
16_KA		7.4	0.17
10_KF	10	65.8	0.28

the annealing time t. This confirmed that the observed grain growth kinetics were controlled by curvature driven grain boundaries and not by triple junction motion that would have resulted in $S \sim t^2$. From these results the rate of area change for grains with different n at different temperatures could be extracted (Fig. 6).

The measured rate of area change at 300 °C versus the topological class of the grain (Fig. 7) revealed that the rate dS/dt was not constant for a given n, as expected according to the conventional theory of grain growth kinetics, but ranged between zero and some maximum value. The classical approach of von Neumann–Mullins [7,8] predicts a rate of area change according to Eq. (5), i.e., the grain area change depends only on the number of sides n of a grain. For a grain with 6 sides the area change is zero, and the grain is stable. These predictions, however, are at variance with the experimental results of this study.

We attribute the observed variation of the dS/dt – values to the drag effect of triple junctions on the grain growth. Since a vertex angle θ is a function of criterion Λ, the expressions for the rate of grain area change in

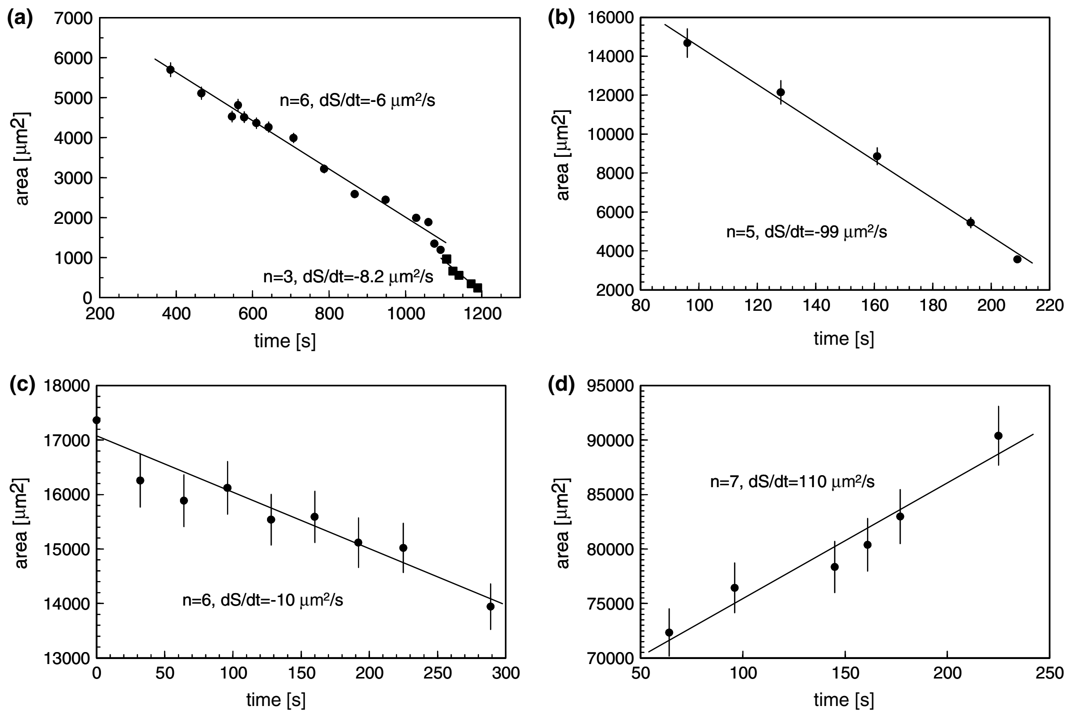

Fig. 6. Grain area versus time at a constant temperature of 300 C for grains with (a) $n = 4$ (3); (b) $n = 5$; (c) $n = 6$; (d) $n = 7$. The grain with $n = 4$ exhibited a constant rate of the grain area change up to about 1150 s. Then the shrinkage rate changed due to a decrease of the topological class of the grain from $n = 4$ to $n = 3$. From this time on the grain was defined as a new one and was accounted for separately.

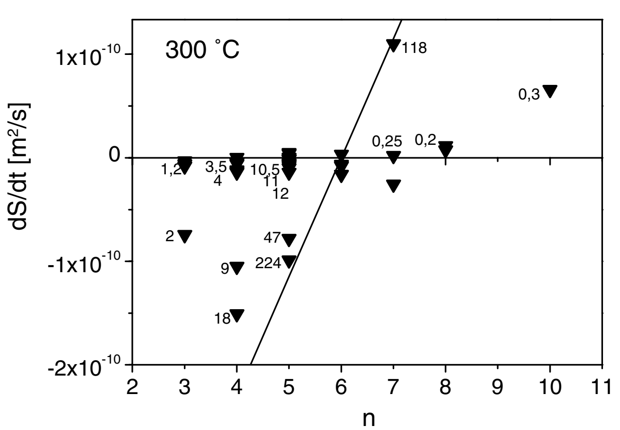

Fig. 7. Rate of grain area change dS/dt versus topological class n of a grain for 300 °C. The values of Λ are given next to the data point.

cases of $n < 6$ and $n > 6$ (Eqs. (6) and (7)) can be rewritten as

$$\frac{\mathrm{d}S}{\mathrm{d}t} = \frac{\pi A_b}{3(1+1/\Lambda)}\left(n\frac{6+\sqrt{3}\Lambda}{2+\sqrt{3}\Lambda} - 6\right) \quad (n < 6) \tag{8}$$

and

$$\frac{\mathrm{d}S}{\mathrm{d}t} = \frac{\pi A_b}{3(1+1/\Lambda)}\left[n\left(1+\frac{6\ln\sin\pi/3}{\pi\sqrt{3}\Lambda}\right) - 6\right] \quad (n > 6), \tag{9}$$

respectively. The advantage of the expressions (8) and (9) is the explicit dependence of the rate of grain area change dS/dt on the parameter Λ. It is stressed, however, that these relations only hold in the vicinity of $\Theta = 60°$.

Apparently, the grains which exhibited higher rates dS/dt, were not (or less) affected in their growth or shrinkage by triple junctions. This corresponds to large values of Λ, for which both expressions (8) and (9) approach the von Neumann–Mullins relation (Eq. (5)). The von Neumann–Mullins limit is represented in Fig. 7 by the straight line connecting the maximum values of dS/dt for $n = 5$ and $n = 7$ and the point dS/d$t = 0$ for $n = 6$. According to Eq. (5) the slope of this line is determined by the reduced grain boundary mobility A_b which was determined from Fig. 7 as $A_b \sim 10^{-10}$ m^2/s. This value is in a good agreement with mobility data obtained on aluminum bicrystals [10].

The Λ-value for each experimental data point in Fig. 7 can then be calculated using measured values of dS/dt (assuming $A_b \sim 10^{-10}$ m^2/s) according to Eq. (8) for $n < 6$ and Eq. (9) for $n > 6$ (Tables 3 and 4). The more a measured value of dS/dt deviated from the theoretical prediction (von Neumann–Mullins limit) the stronger the drag effect exerted by triple junctions, and correspondently, the smaller the respective Λ-value. For example, for grain 14_K7 with $n = 5$ the area shrinkage rate was measured to be dS/d$t = 98.9 \times 10^{-12}$ m^2/s. The

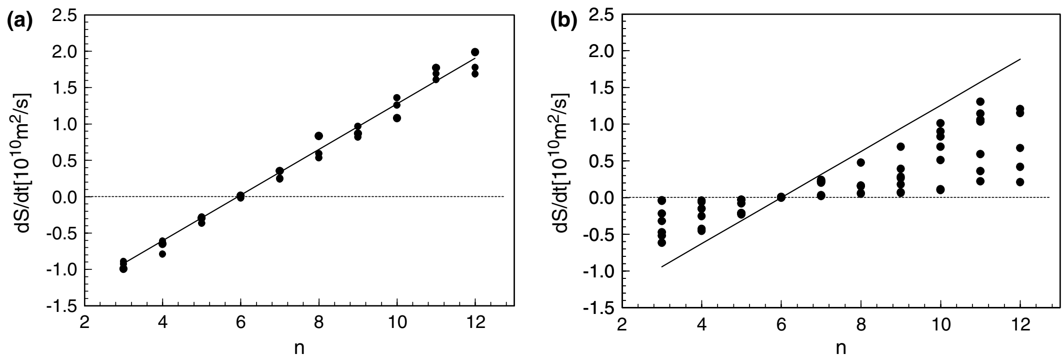

Fig. 8. Results of a vertex-simulation (a) pure von Neumann–Mullins case (Λ-infinite), (b) with junction drag ($0.1 < \Lambda < 10$), assuming $m_b \cong 10^{-10}$ m^4/J s and $\sigma \cong 0.3$ J/m^2 [11].

corresponding value of $\Lambda = 224$ demonstrates that the effect of triple junctions on this grain is negligibly small. In contrast, the much smaller value $\Lambda = 3.6$ for grain 14_K5 with $\mathrm{d}S/\mathrm{d}t = 6.0 \times 10^{-12}$ m^2/s indicates that the growth kinetics of this grain are substantially affected by its junctions.

For grains with different number of sides, Λ is different despite small $\mathrm{d}S/\mathrm{d}t$ values. For grains with $n = 3$ Λ the criterion reached values of 1.5 we determined whereas for $n = 5$ $\Lambda \approx 11$ despite small $\mathrm{d}S/\mathrm{d}t$ values. This can be understood from the modified von Neumann–Mullins approach by Gottstein and Shvindlerman [11]. When $\mathrm{d}S/\mathrm{d}t = 0$ then Λ is 10.4 for a grain with $n = 5$. This means that for this value $\Lambda_0 = \Lambda$ ($\mathrm{d}S/\mathrm{d}t = 0$) a grain is stable and does neither shrink nor grow. For Λ values adjacent to Λ_0 the triple junction had a large influence on grain boundary motion, which reflected the transition from grain boundary kinetics to triple junction kinetics. This transition is not defined by a fixed value of Λ because it depends on the number of sides of a grain. For $\mathrm{d}S/\mathrm{d}t = 0$ a grain is stable since it is bordered by straight (zero curvature) grain boundaries. For a 4 sided grain $\Lambda_0 = \Lambda = 3.5$.

The experimental results are in good agreement with predictions of vertex simulations of grain growth [11] (Fig. 8). In the case of Fig. 8(a) the simulation was carried out with perfectly mobile triple junctions. The spread due to statistical scatter reflects the drag effect of the triple junctions on grain growth. The straight line represents the ideal von Neumann–Mullins limit. Large deviations from this line are obvious, i.e., the simulated values are much smaller than predicted values by the von Neumann–Mullins theorem. Such behavior was also found in the current experimental study and confirms the modified von Neumann–Mullins relation. If is stressed again, the spread of the data is no artifact of the measurement but results from the dragging influence of the triple junctions on boundary motion, and the observed behavior substantiates that triple junctions can substantially slow down microstructure evolution.

6. Summary

In situ observations of grain growth were carried out on aluminum-10 ppm magnesium sheet in order to investigate the influence of triple junctions on grain growth kinetics. EBSD-analysis of the investigated area served to characterise the triple junctions by the crystallography of the adjoining grain boundaries. The steady state velocity of triple junctions, the contact angle at the triple junctions and the rate of grain area change were measured. The following conclusions can be drawn.

1. Triple junction mobility can be low which manifests itself in a deviation of the dihedral contact angles at triple junctions from the equilibrium angles. If its mobility is low, a triple junction can exert a dragging influence on the motion of the adjoining grain boundaries. This influence depends on temperature, i.e., with increasing temperature the triple junctions lose their impact on the boundaries.
2. For the first time it has been shown experimentally that due to triple junction drag there is no unique linear relationship between growth rate and the number n of grain sides, as proposed by the classical von Neumann–Mullins relation. The drag influence of the triple junctions causes the growth rate to depend not only on n but on the criterion Λ as well, i.e., $\dot{S}(\Lambda, n)$ with the limits $0 \leqslant \dot{S} \leqslant \dot{S}_{\mathrm{vNM}}$, where $\dot{S}_{\mathrm{vNM}}$ represents the von Neumann–Mullins limit.

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