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Influence of transformation temperature on microtexture formation associated with α precipitation at β grain boundaries in a β metastable titanium alloy

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Abstract

The influence of transformation temperature on microtexture development associated with α precipitation at β/β grain boundaries (GB) in the near-β Ti17 alloy was studied using electron backscatter diffraction and considering isothermal treatments. For the alloy studied and the temperature range considered, decreasing the transformation temperature decreased the local microtexture strength within each prior β grain because of a larger number of α_{WGB} colonies (standing for α Widmanstätten GB) formed per β grain, each colony increasing by one the number of α orientations inside each prior β grain. This larger number of α_{WGB} colonies was a consequence of faster formation along β/β GB of their precursors, the allotriomorphic α_{GB} grains (standing for α-GB) at lower transformation temperatures, as evidenced by detailed examination of the first stages of α_{GB} formation. α_{GB} crystallographic orientations frequently followed a variant selection (VS) criterion based on the alignment of $(0\,1\,1)\beta//(0\,0\,0\,1)\alpha_{GB}//(0\,1\,1)\beta$. From a statistically relevant number of observations, VS was found to be more frequent at a lower transformation duration and a lower temperature, but the effect was not significant enough to influence the final α microtexture, considered at the scale of one prior β grain. α_{GB} grains that followed the VS criterion emitted two α_{WGB} colonies on either side of the β/β GB more frequently than those with no particular orientation.

Keywords: Titanium; Microstructure; Microtexture; Phase transformation; Electron backscatter diffraction

1. Introduction

Most studies on titanium alloys dealing with the microstructure and local texture development associated with the $\beta \rightarrow \alpha + \beta$ transformation focus either on the inherited morphology and associated kinetics [1–9] or the crystallographic aspects [10–17]. The present work proposes to link these two.

During cooling of titanium alloys from the β field, the α phase first forms at β/β grain boundaries (GB). This α_{GB} (α GB) morphology consists of nearly continuous α phase layers along β/β GB. Then, Widmanstätten colonies (α_{WGB}) form from those new α_{GB}/β interfaces [1]. They consist of parallel side plates of close orientation separated by remaining layers of β matrix enriched in β-stabilizing elements. Within a colony, there is a tendency to reach the thermodynamic equilibrium values of the respective volume fractions and chemical compositions of both α and enriched β phases. Each α_{WGB} colony needs a parent α_{GB} grain to form (they never form directly on β/β GB). Eventually, if the transformation is not complete and the transformation conditions are achieved, intragranular nucleation of α occurs, leading to a plate morphology identified as the α_{WI} morphology (standing for Widmanstätten

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Table 1
Chemical composition of the Ti17 alloy studied, measured at top and bottom of the billet.

	Al	Cr	Mo	Sn	Zr	O_2 (ppm)
Top	5.03	3.99	4.03	1.99	1.97	1225
Bottom	5.01	4.13	3.90	2.05	2.01	1205

intragranular). This experimental knowledge has recently allowed the modeling of these phase transformations, based either on isothermal (IT) diagrams (Johnson–Mehl–Avrami–Kolmogorov description [6,18]) or on mesoscale approaches based on thermodynamic and diffusion data and describing nucleation and growth processes [19–21]. Using specific models for each morphology makes it possible to predict their growth kinetics and chemical composition as a function of temperature evolution.

The $\beta \rightarrow \alpha + \beta$ phase transformation follows the Burgers orientation relationship (BOR), which implies the parallelism of dense planes and direction of both phases: $(0\,1\,1)_\beta // (0\,0\,0\,1)_\alpha, [1\,1\,-1]_\beta // [1\,1\,-2\,0]_\alpha$. One β orientation can thus give rise to 12 crystallographic α orientations, called variants. Concerning the α_{WI} grains, the 12 variants occur with the same frequency [22]. The α_{GB} grains are mostly in BOR, with only one of the two β grains separated by the β/β GB [12–14,16]. The variant taken by α_{GB} is not random; it depends on the crystallographic parameters of the β/β GB. This phenomenon is called variant selection (VS) [23]. The selection of α_{GB} variants has a direct influence on the α texture obtained at the end of cooling [12,24]. Indeed, the α_{WGB} colonies inherit the orientations taken by their α_{GB} parent grains. Therefore, it is possible to obtain very large areas of the parent β grain with a single orientation of the α phase, which can be detrimental to mechanical properties [12,25].

Better control of the mechanical properties can be achieved by studying the α texture resulting from the $\beta \rightarrow \alpha + \beta$ phase transformation. This has been done extensively at a macroscopic scale (e.g., Ref. [11]) or locally using the electron backscatter diffraction (EBSD) technique [12]. These previous studies focused only on the influence of the β grains orientations (β texture) on the α phase texture. Now one can also use the EBSD technique to start studying the influence of thermal treatment parameters (e.g., cooling rate) on the development of the microtexture, as attempted very recently [26]. First, one has to establish the role of the transformation processes (phase growth, morphologies) and associated kinetics. Little is known on the detailed sequence of events associated with the microtexture construction, because most observations in the literature were done after completion of phase transformation. Second, possible VS influence on final microtexture has to be studied. In particular, it is not known whether the phase transformation temperature can modify the variants selected preferentially, i.e., the nature of VS criteria and their frequency of occurrence.

The purpose of this study is to achieve an experimental investigation of the influence of transformation temperature on α microtexture development. The study was carried out considering a β metastable alloy (Ti17) whose microstructures have been studied extensively [1–3,18–21], but without considering crystallographic orientations. The focus was on α precipitation at β GB giving rise to VS. The α microtexture was characterized using the EBSD technique. The VS was estimated by counting the number of α_{WGB} variants over a statistical number of prior β grains. The influence on the microtexture strength of two distinct features of the α precipitation sequence at β/β GB was established: First, the final number of α_{WGB} colonies per prior β grain, which depends on the phase transformation processes (α_{GB} and α_{WGB} formation). Increasing this number weakened the microtexture. Second, VS was considered, in so far as it can reduce the number of α orientations and strengthen the microtexture. Influence of phase transformation temperature on both aspects was examined. After detailing the experimental method, results are presented according to the chronology of the precipitation sequence of α on β/β GB, and will be discussed in a later section.

Table 2
Thermal treatments and microtexture analysis performed with EBSD.

T (°C)	Duration	Precipitation sequence	Equilibrium α volume fraction [2] (%)	α Volume fraction (%)[a]	Fraction of GB decoration by α_{GB}[b] (%)	Technique of analysis	EBSD step size (μm)	No. of α_{GB} grains analyzed with EBSD
830	16 h	Complete	27	27	91	EBSD	1	200
800	200 s	α_{GB}	35	1	12	OM	–	–
	700 s			4	65	EBSD	0.5	107
	1 h	α_{WGB}		17	81	EBSD	0.5	450
	6 h	Complete		35	94	EBSD	0.5	168
750	100 s	α_{GB}	45	0	7.9	OM	–	–
	700 s	α_{WGB}		6.5	81	EBSD	0.4	204
	6800 s	Complete		45	97	EBSD	0.4	158

[a] Estimated from previous phase transformation kinetics measurements [1–3].
[b] Total length of α_{GB} grains divided by total length of β/β GB.

2. Experimental

2.1. Material

Cylindrical specimens 3 mm in diameter and 30 mm in length were spark-machined (electrical discharge machining) from a billet of Ti17 alloy manufactured by TIMET-Savoie. Its composition is shown in Table 1. A β transus of 880 °C was measured through electrical resistivity [27] and confirmed by high energy X-ray diffraction [3].

2.2. Thermal treatments

The samples were first solution-treated at 920 °C in the β phase field for 30 min to obtain a homogeneous β grain size of ~200 μm. Then IT treatments in the α + β field (Table 2) were carried out under secondary vacuum. Finally, the high-temperature α/β microstructure was quenched in less than 10 s by blowing helium gas. On the basis of previous characterizations of IT phase transformation kinetics [1–3], the transformation temperatures and durations were chosen so that only α_{GB} and α_{WGB} were formed.

Three transformation temperatures were chosen (750 °C, 800 °C and 830 °C) to consider the influence of the phase transformation driving force. The following durations were selected corresponding to different stages of the α precipitation sequence: 200 s and 700 s at 800 °C, 25 s and 100 s at 750 °C to examine α_{GB} formation along β/β GB; 1 h at 800 °C and 700 s at 750 °C, at the beginning of α_{WGB} colonies formation; 16 h at 830 °C, 6 h at 800 °C and 6800 s at 750 °C, at phase transformation completion—the equilibrium α amount has nearly been reached, and there is no more microstructure evolution.

2.3. Microstructure characterization

The observation techniques used for each treatment are specified in Table 2. Most measurements were done using scanning electron microscopy (SEM) micrographs obtained in combination with EBSD experiments (see next section), except for the shortest durations of IT treatment, for which optical microscopy was used. For optical microscopy, the samples were mechanically polished and then etched using Kroll's reagent. As shown in Table 2, the α_{GB} nucleation and growth stage correspond to a negligible α volume fraction and partial β/β GB decoration by α_{GB}. At the α_{WGB} growth stage, the GB decoration is almost complete, and the α phase volume fraction is more significant.

Typical optical micrographs are shown in Fig. 1. The 800 °C–1 h microstructure exhibits both α_{GB} and α_{WGB} morphologies. Different degrees of transformation, characterized by the size and amount of α_{GB} grains and α_{WGB} colonies, have been reached on each β/β GB. The 800 °C–700 s microstructure exhibits only a few α_{GB} grains. Finally, the 750 °C–700 s microstructure is similar to the 800 °C–1 h one, with finer α_{GB} grains and less developed colonies of α_{WGB}. Microstructures for shorter IT treatment are shown in Section 3.1.1, and the aspect for phase transformation completion can be found, for instance, in Ref. [2].

2.4. Microtexture characterization

EBSD analyses were performed on the most relevant samples (see Table 2). Samples were first mechanically and then electrolytically polished (in 80% methanol–20% perchloric acid, applying 17 V, 2 A, 20 s, 3 °C). Crystallographic orientation maps were acquired by SEM with JEOL 6490 and FEI Quanta 600 FEG instruments, both equipped with NordlysF cameras and HKL software from Oxford Instruments. Depending on the sample, different maps were acquired and analyzed until at least 200 β/β GB had been sampled, in order to obtain statistically relevant data. The maps were obtained with a step size of 0.4 or 0.5 μm, indexing both β and α phases.

The orientations in the maps and pole figures are colored according to the code of the standard triangle (each color depends on the projection of a macroscopic direction in the crystal reference frame). With this code, different colors indicate different crystallographic orientations, but the inverse is not true.

GB characteristics such as length, transformed fraction, misorientation, VS mechanism criteria, presence of α_{GB}/α_{WGB}, misorientation were systematically inventoried by hand. It was verified that the observations were statistically relevant by making sure that increasing the number of measurements did not change further the average value of the examined parameters.

3. Results

3.1. Precipitation of α_{GB}

Different observations (Figs. 2 and 3) showed that decoration of β/β GB by α_{GB} grains was progressive at both transformation temperatures. In this section, the rate of α_{GB} formation is further analyzed by considering the formation process along the β/β GB, focusing on the first stages in particular.

3.1.1. α_{GB} Grains number per β/β GB

Within each prior β grain, the numbers of α_{GB} grains and of α_{WGB} colonies are closely related, because each colony needs one precursor α_{GB} grain to form. Hence, measuring the number of α_{GB} is important for understanding the microtexture development. Such measurements are used in Section 3.3.2 to interpret the variation of the number of α_{WGB} colonies per β grain with transformation conditions. The average number of α_{GB} grains per β/β GB was counted, denoted N_{GB}. A statistically relevant number of α_{GB} grains were inventoried, using EBDS maps as in Fig. 3, in order to establish the influence of phase transformation temperature and duration on N_{GB}.

N_{GB} measurements are shown in Table 3. Noticeable is the limited N_{GB} variation with transformation duration, which increases from 1.7 to 2.4 at 750 °C and 1.8 to 1.9 at 800 °C. Considering the influence of the phase transformation temperature, it is interesting to observe that N_{GB} was not very different between 830 and 750 °C, despite the different driving force. At phase transformation completion, N_{GB} was equal to 1.8 at 830 °C and 2.4 at 750 °C. For lower transformation durations at 750 and 800 °C, measured N_{GB} values were also similar.

In order to explain the low N_{GB} values (~2) and their limited variation with both transformation temperature and transformation duration, the first stages of α_{GB} grain formation were considered. Observing the microstructure after a short transformation duration (200 s at 800 °C and 100 s at 750 °C) enabled the first α_{GB} grains formed to be located. The optical micrograph in Fig. 2a shows that, at a transformation temperature of 800 °C, the first α_{GB} grains were always connected to a triple junction (TJ). However, for a phase transformation temperature of 750 °C (Fig. 2b), the α_{GB} grains were too thin to confirm this observation without uncertainty in some cases, even using SEM.

One can nevertheless deduce the following simple α_{GB} formation sequence from these observations: precipitation starts at TJ, followed by α_{GB} growth along the β/β GB planes, leading to large α_{GB} grains like those in Fig. 3. This

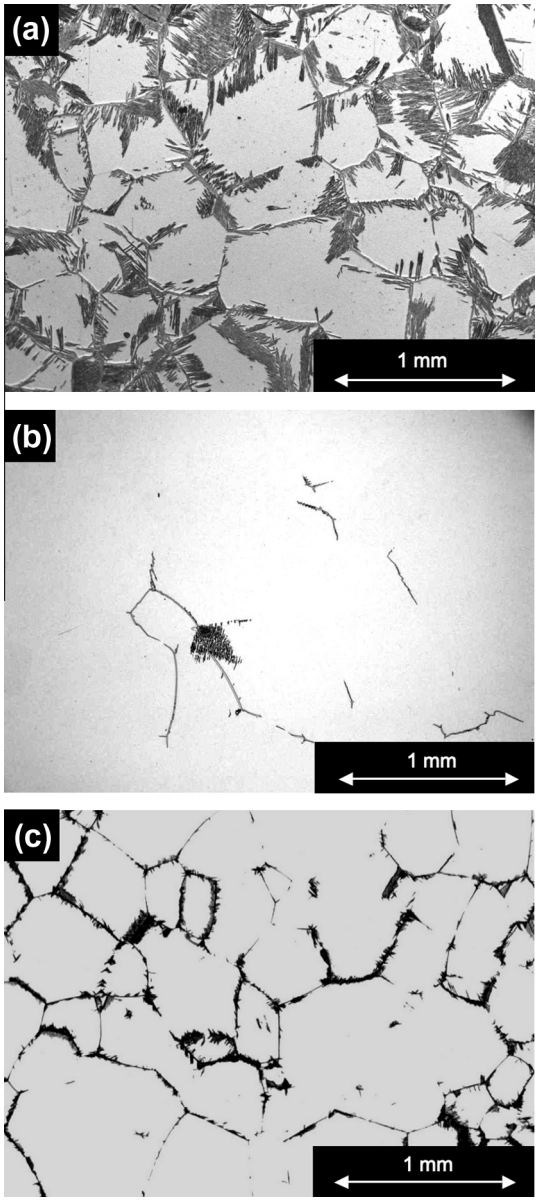

Fig. 1. Micrograph of sample at: (a) 800 °C–1 h; (b) 800 °C–700 s; (c) 750 °C–700 s.

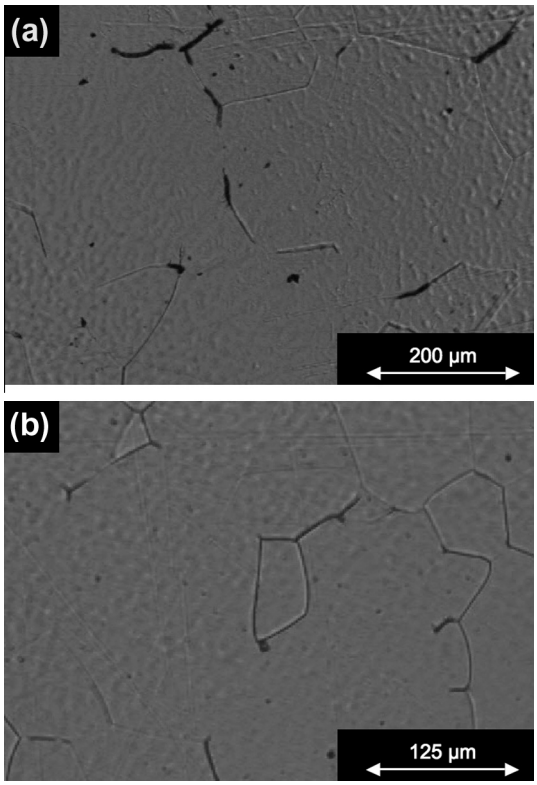

Fig. 2. Optical micrographs: (a) 800 °C–200 s; (b) 750 °C–100 s.

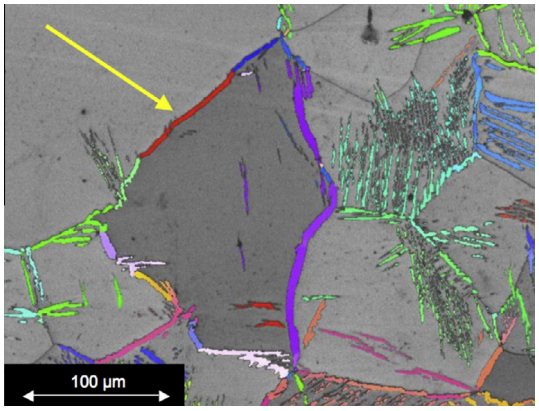

Fig. 3. EBSD orientation map of the 800 °C–1 h microstructure. Arrowed α_{GB} isolated from TJ.

Table 3
No. of α_{GB} grains and α_{WGB} colonies per β grain.

	750 °C		800 °C		830 °C
	700 s	6800 s	1 h	6 h	16 h
α Decorated GB proportion[a] (%)	81	97	81	94	91
N_{GB}	1.7	2.4	1.8	1.9	1.8
N_{WGB}	2.75	7.7	2.8	4.4	3.6

[a] Total length of α_{GB} grains divided by total length of β/β GB.

sequence certainly occurred at 800 °C and probably at 750 °C. This sequence explains the low N_{GB} values measured (~2): nucleation of α was restricted to a very limited number of sites located at TJ, or perhaps more restrictively at β quadruple points (QP, corners). This sequence may also explain why N_{GB} did not vary significantly with the transformation temperature. At 830 °C, the nucleation driving force was already large enough to enable α nucleation on almost all available nucleation sites, and decreasing the transformation temperature to 800 or 750 °C did not activate many additional nucleation sites. N_{GB} is stable with transformation duration, because the nucleation sites were saturated before the transformation durations considered.

It is worth mentioning that a very different α_{GB} formation sequence has been proposed in other studies [4,17]: large α_{GB} grains like those of Fig. 3 are believed to result from the impingement by growth of several fine α precipitates that nucleated separately in the middle of the β/β GB plane. These initially separate precipitates are believed to have the same crystallographic orientation because of strong VS at β/β GB. The observations in Refs. [4,17] should not be generalized too readily, because the authors considered alloy compositions, transformation temperatures and rate of diffusion different from those of present study. For instance in Ref. [17], a transformation temperature of 660 °C was considered. The slower diffusion of the alloying elements is likely to have impeded the β/β GB decoration by α_{GB} diffusional growth, leaving enough time, in this case, for α to nucleate in the middle of the GB.

The key difference with the present proposed sequence is that, according to Refs. [4,17], the α phase can nucleate in the middle of the β/β GB planes. For the transformation conditions considered, α grains at the middle of a β/β plane and disconnected from a TJ were almost never observed. Only a few exceptions were observed after longer transformation durations, where some α_{GB} grains can be considered isolated from a TJ, as shown by the arrow in Fig. 3. However, recent 3D characterization of the microstructure [28] revealed that the α_{GB} grains grew, with ramifications. Apparently isolated α_{GB} may, in fact, be the intersection of these ramifications with the observation plane.

The micrographs at short durations in Fig. 2 provide sufficient evidence that α nucleation was restricted to β TJ or perhaps only QP, at least at 800 °C and probably at 750 °C. Fig. 2 is only one example of a statistical number of micrographs that were carefully examined. The low variation in N_{GB} shows that the transformation temperature has an effect mainly on the kinetics of α_{GB} formation (as detailed in the next section), i.e., the number of α_{GB} precursors for α_{WGB} colonies can be considered as nearly constant.

3.1.2. Growth along the GB planes

Table 2 illustrates the fact that the α_{GB} grains decorated the β/β GB progressively (i.e., there was no "immediate" wetting). For instance, at 800 °C, the fraction of GB decoration by α_{GB} increased from 12, 65, 81 and 94% after 200 s, 700 s, 1 h and 6 h of transformation. An analysis of the effect of the β/β GB misorientation angle θ, not detailed here, revealed that the α_{GB} growth was faster on high angle GB ($\theta > 15°$) than on low angle GB ($\theta < 15°$). Hence, the β/β GB energy influenced the α_{GB} growth rate.

The effect of the transformation temperature on the rate of α_{GB} formation shows that the kinetics was faster at 750 °C than at 800 °C. The time to fill ~80% of the β/β GB with α_{GB} grains was 700 s at 750 °C and 1 h at 800 °C (Table 2). By dividing an approximate β/β GB length (~100 μm) by these durations, a crude α_{GB} growth rate along the GB is estimated at 14×10^{-8} m s^{-1} and 2.8×10^{-8} m s^{-1} at 750 and 800 °C. Faster α_{GB} growth at 750 °C than at 800 °C is consistent with previous studies on α_{WGB} morphology [19], which clearly established the diffusion-controlled growth of α. According to modeling [19–21], the α_{WGB} platelets' growth rate at 750 and 800 °C was equal to 2.0×10^{-8} m s^{-1} and 1.0×10^{-8} m s^{-1}. Growth rate orders of magnitude are thus similar for α_{GB} (along the GB) and α_{WGB}, though with higher growth rates for α_{GB} and a bigger difference between α_{GB} and α_{WGB} at 750 °C. This bigger difference will have consequences for further evolution of the microstructure associated with the development of the α_{WGB} colonies, which is highlighted in Section 3.3.2.

3.2. Orientation of α_{GB}; variant selection

The present results confirm that, in the vast majority of cases, the α_{GB} grains are in BOR, with only one of the two β grains adjoining the β/β GB (Table 4), in accordance with numerous previous works. No α_{GB} grain was found to respect the exact BOR with both grains at the boundary,

Table 4
Proportion of α_{GB} grains verifying the BOR with the β grains in contact: maximum deviation to BOR considered for the statistics, 5°.

Thermal treatment	No. of adjoining β grains respecting the BOR with α_{GB} grain		
	0	1	2
830 °C–16 h	31 (15.5%)	169 (84.5%)	0
800 °C–6 h	35 (16.5%)	177 (83.5%)	0
800 °C–1 h	65 (15%)	380 (85%)	0
800 °C–700 s	3 (3%)	104 (97%)	0
750 °C–6800 s	10 (7%)	147 (93%)	0
750 °C–700 s	0	72 (100%)	0

unlike what was observed in Ref. [15]. The α_{GB} grain can be in BOR simultaneously with both β grains only for some special β/β GB [29]. No such GB were found in the microstructures analyzed. Table 4 shows that some proportion of the α_{GB} grains was in BOR with neither of the two adjacent β grains (for instance 15% at 800 °C and 1 h). Similar non-BOR α_{GB} were reported in studies on Ti–8Al–xV [14] and TA6V [16]. This may be due to BOR α formation on TJ followed by non-BOR growth along the GB plane. This interpretation is confirmed by the EBSD maps (no examples are shown here). Table 4 also shows that the proportion increased with exposure time, implying that non-BOR α_{GB} growth is slower.

The α_{GB} orientation was often not selected randomly among the 12 potential variants inherited from β to α transformation. The present authors studied a VS criterion often discussed in the literature [13–16,23]: If adjacent β grains β_1 and β_2 share a common $\{110\}_\beta$ pole, the α_{GB} grain orientation that aligns its $\{0001\}_\alpha$ to this common $\{110\}_\beta$ pole is often preferentially selected. This means that β_1, β_2 and α_{GB} tend to align their dense planes. This VS emphasizes α_{GB} orientations that are simultaneously in BOR with one β grain and not far from the BOR with the other β grain.

The purpose in this study was to examine this criterion further by estimating its frequency of occurrence. To this end, an approach introduced recently in Ref. [23] was followed. It consists in measuring for each α_{GB} grain the angle ω between the closest $\{110\}_\beta$ planes of its two adjacent β grains. ω is different from θ, the misorientation angle. ω can be close to 0, even if θ is large. $\omega = 0$ means exact alignment between two $\{110\}_\beta$ planes. Then it was determined whether or not the α_{GB} grain $\{0001\}$ plane was parallel to the closest $\{110\}$ poles.

The result for the 800 °C–1 h and 750 °C–700 s treatments is given in Fig. 4 as a function of ω. The β GB were categorized into different classes of 4° width, according to their ω values. For each class, the fraction of corresponding α_{GB} grains satisfying the VS criterion was plotted. As expected and as observed in Ref. [23], for both transformation temperatures the VS frequency decreased with increasing ω values, that is when the closest $\{110\}_\beta$ poles on either side of the GB deviated from alignment. This trend was observed for all other transformation temperatures and durations. It was verified that the β grains' orientation distribution was similar for all samples, to avoid any interference from the β texture.

A higher driving force and a shorter transformation duration were associated with more frequent VS. This is shown in Table 5, where the proportion of α_{GB} that verified the VS criterion is specified for each treatment. For the sake of simplicity, only β/β GB with $\omega < 4°$ were considered, at which VS was most frequent. At phase transformation completion (longest durations in Table 5), the VS frequency varied from 36% at 830 °C to 45% at 750 °C. Reducing the transformation duration increased the frequency up to 79% for 800 °C–700 s treatment. In addition,

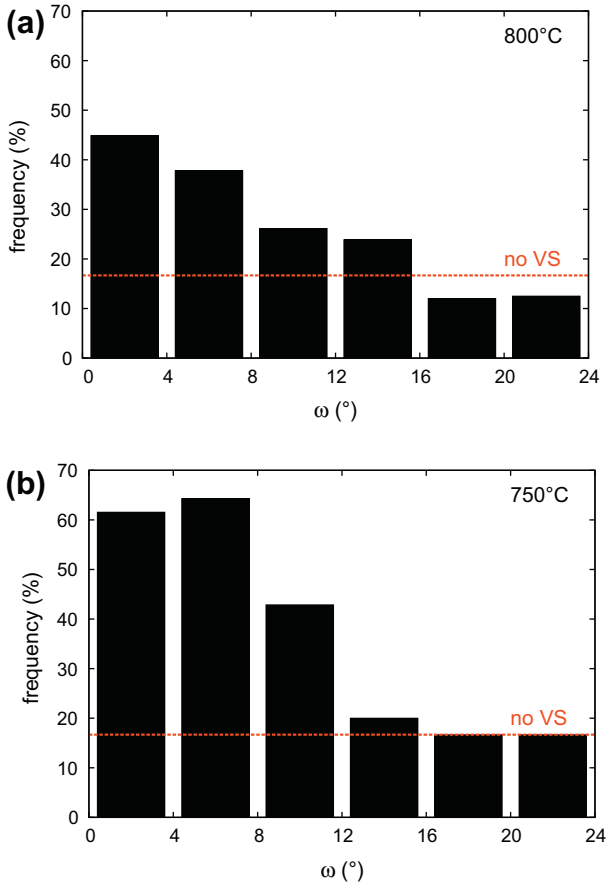

Fig. 4. Proportion of α_{GB} grains respecting the VS criterion based on the alignment of their $\{0001\}\alpha$ pole with closest $\{110\}\beta$ poles of adjacent β grains. The β/β GB were categorized according to their ω angle into classes of 4° range. Each bar indicates the proportion of α_{GB} grains that respected the VS criterion for each class of β/β GB.

Table 5
Frequency of VS and α decorated GB proportions as a function of transformation temperature and duration, taking only β/β GB with $\omega < 4°$ into account.

Temperature	750 °C		800 °C			830 °C
Duration	700 s	6800 s	700 s	1 h	6 h	16 h
α Decorated GB proportion (%)[a]	81	97	65	81	94	91
VS frequency (%)	61	45	79	45	41	36

[a] Total length of α_{GB} grains divided by total length of β/β GB.

it enhanced the temperature influence: comparing the 800 °C–1 h and 750 °C–700 s treatments that exhibit the same proportion of GB decoration by α, the frequency was 45% and 65%, respectively. This influence of transformation temperature is confirmed by a previous study on Ti–LCB [23], where a frequency of 70% was reported at 615 °C, which is equivalent to 705 °C in Ti17 in terms of undercooling (T–T_β).

The larger proportion of α_{GB} verifying the VS criterion at shorter transformation durations is probably due to

Table 6
Number of α_{WGB} colonies emitted by α_{GB} grains at phase transformation completion as a function of transformation temperature.

T (°C)	Duration	Av. No. of α_{WGB} colonies/α_{GB}	α_{GB} Not verifying VS rule				α_{GB} Verifying VS rule			
			Proportion of α_{GB} per No. of α_{WGB} colonies emitted (%)			Av. No. of α_{WGB} colonies/α_{GB}	Proportion of α_{GB} per No. of α_{WGB} colonies emitted (%)			Av. No. of α_{WGB} colonies/α_{GB}
			0	1	2		0	1	2	
750	6800 s	0.99	15	85	0	0.85	4	66	30	1.26
800	6 h	0.69	49	50	1	0.52	9	81	10	1.00
830	16 h	0.85	22	77	1	0.79	18	64	18	1.00

more favorable transformation conditions, considering interface and elastic strain energies. Only longer transformation durations would leave enough time for the formation of α_{GB} with less favorable crystallographic orientations. Another explanation could be that another VS criterion becomes prevalent at longer transformation durations. Such an evolution has been reported in a very different transformation (bainitic ferrite in steel [30]). In fact, another VS criterion for α precipitation on β GB in titanium involving the GB plane orientation has been put into evidence in Ref. [17]. Examining the two VS criteria simultaneously would surely provide a more comprehensive understanding. This could also explain why only a certain proportion of the α_{GB} followed the VS criterion studied when the GB ω angle was small. These results show that the VS criterion studied in the present work (alignment of $(011)_{\beta1}//(0001)_{\alpha}//(011)_{\beta2}$) was often followed by the α_{GB} and at a frequency that depended on transformation temperature and on treatment duration.

Interestingly, more frequent VS at lower transformation temperatures is difficult to explain with the current interpretations of the origin of VS that can be found in the literature. Let us first draw a short overview of these interpretations, focusing on precipitation at GB and diffusion-controlled transformations (not martensitic or massive). In the first studies that put VS in titanium alloys into evidence [16,17], VS has been interpreted qualitatively in the framework of the classical nucleation theory. According to these studies, the variants following VS criteria nucleate more favorably because of a reduced activation energy for critical nucleus formation, due only to α/β interfaces with lower interfacial energy. Other analyses considered exclusively the misfit elastic strain energy required to accommodate the α nucleus inside the volume occupied initially by the β phase and neglected the influence of interfacial energies (e.g., Ref. [31]). In an experimental study on GB nucleated bainitic ferrite in an Fe–9Ni–0.15C steel [30], the effects on VS of both interfacial energies and elastic straining energies were considered simultaneously. Studying the effect of phase transformation duration, it was observed that, at the beginning of the transformation, the variants of bainitic ferrite were restricted to minimize the interfacial energy, while as the transformation proceeded, the VS reducing the deformation energy became more important.

It is thought that none of the results from the literature summarized above can fully account for the increased VS frequency at larger undercooling that was put into evidence in the present work. According to these analyses, increasing the phase transformation chemical driving force could allow less VS because of the larger energy amount available for interfacial energies and elastic energy. Those latter are not expected to depend significantly on temperature (ignoring the effect of plasticity). A thorough investigation of the origin of VS would require a numerical approach considering both interfacial energy and deformation energy with accounting of the neighborhood of the forming grain (i.e., nucleation site β/β GB plane, but also TJ or corners [30,32,33]). Calculations of the deformation energy should also consider this neighborhood with the anisotropy of the elastic properties and certainly the occurrence of plasticity.

3.3. α_{WGB} Microtexture and microstructure

3.3.1. Microtexture

The α_{GB} orientation (and associated VS mechanism) influenced the orientation of the α_{WGB} colonies that developed from it. Colonies were emitted in one or both β grains at each side of the α_{GB}. When only one α_{WGB} colony was formed, it was always inside the β grain in BOR, with α_{GB} and the α_{WGB} colony inherited, in this case the exact α_{GB} orientation, according to EBSD precision, ~1° or 2°. Only one example (not shown) was observed of an α_{WGB} colony that did not respect the BOR with its host β grain. When α_{GB} grains emitted a colony in the other β grain, it was also in BOR with its respective host β grain. This second colony was misoriented from the α_{GB} by a low angle, always inferior to 5°, but clearly detectable. It seems that the emergence of the α_{WGB} sideplates inside the β grain not in BOR with the parent α_{GB} was accompanied by partial adaptation of their crystallographic orientation. Interestingly, this adaptation was the same for each sideplate inside the colony. In almost all cases, the variant selected inside the second β grain was the one that minimized the misorientation angle between α_{GB} and α_{WGB}.

It was also found that the α_{GB} grains that verified the VS criterion studied in Section 3.3.2, i.e., alignment of $\{110\}_{\beta1}$, $\{0001\}_{\alpha}$ and $\{110\}_{\beta2}$, had emitted on average more α_{WGB} colonies than other α_{GB} (Table 6). For

instance, at 800 °C the average number of α_{WGB} colonies emitted by the α_{GB} grains was equal to 0.52 for α_{GB} not following VS and equal to 1.00 for α_{GB} following VS. The trend was similar for transformation temperatures 750 and 830 °C. Table 6 also shows the proportion of α_{GB} that emitted 0, 1 or 2 α_{WGB} colonies on either side of the GB. The proportion of α_{GB} with 0 colony was higher inside the population of α_{GB} that did not follow the VS rule. It can also be seen that almost none (0–1%) of the α_{GB} that did not follow the VS rule emitted two α_{WGB} colonies, while a significant proportion (10–30%) of the α_{GB} that followed the VS rule emitted two colonies. Finally, a noticeable result was the absence of temperature influence on the number of α_{WGB} colonies emitted by the α_{GB}.

Two mechanisms may explain why the proportion of α_{GB} having emitted at least one or even two colonies was much larger inside the population of α_{GB} that followed the VS criterion. (i) These α_{GB} formed faster, as concluded in Section 3.3.2; as a result, these α_{GB} had more time to form colonies. (ii) It is likely that the α_{GB} verifying $\{1\,1\,0\}_{\beta 1}$, $\{0\,0\,0\,1\}_{\alpha}$ and $\{1\,1\,0\}_{\beta 2}$ alignment reduces the crystallographic orientation adaptation required at the transition from α_{GB} to α_{WGB} inside the β grain not in BOR with α_{GB}. This is confirmed by the low α_{GB}/α_{WGB} misorientation angles reported above. The branching phenomenon has been attributed to either interface instability during growth (e.g., Ref. [34]) or to α sympathetic nucleation at the α_{GB}/β interface [35]. In this latter process, the α_{GB}/β interface is replaced after nucleation by a low angle α_{GB}/α_{WGB} boundary, and the sympathetic nucleation rate should be increased for a higher α_{GB}/α_{WGB} misorientation angle. Interestingly, the opposite trend was obtained in the present work, since increasing the misorientation angle between α_{GB} and potential α_{WGB} platelets seemed to reduce the possibility to emit colonies. Additional characterization of α_{GB}/α_{WGB} misorientation should be done at a lower scale using, for example, TEM to investigate further the mechanism of α_{WGB} sideplate emission.

3.3.2. *Microstructure; number of α_{WGB} colonies per β grain*

The average number of α_{WGB} colonies (N_{WGB}) contained by the prior β grains is an important microstructure parameter for microtexture and mechanical properties, because each colony represents a potentially large volume of the material where the platelets of the anisotropic α phase exhibit one single orientation. In a previous work [7], N_{WGB} was measured from SEM micrographs and considering morphological aspects at IT phase transformation completion for the β-Cez alloy with composition Ti–5Al–4Mo–2Cr–1Fe–4Zr–2Sn (wt.%), which is close to that of the Ti17 alloy. It was found that N_{WGB} depended strongly on the transformation temperature, with a value of 7 and 16 for transformation temperatures of 790 and 700 °C, respectively. The larger value of N_{WGB} at larger undercooling (700 °C) had been ascribed to a larger driving force and a greater number of active nucleation sites for α_{GB}, leading to the formation of more colonies. Indeed, the formation of an α_{WGB} colony is not possible without the presence of a parent α_{GB} grain (as the colonies cannot form "directly" on β/β GB). However, in the previous work, the assumption of a larger density of nucleation sites was not analyzed further.

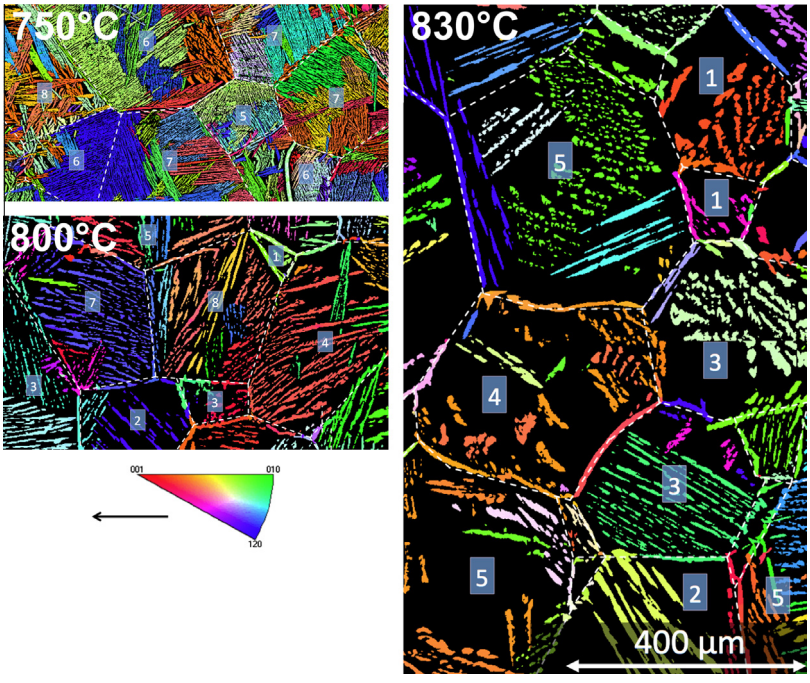

Fig. 5. IPF EBSD maps at the same scale showing the α phase microtexture at phase transformation completion: 750 °C–6800 s; 800 °C–6 h; 830 °C–16 h. β phase represented in black, and β/β GB represented by bold white lines. The α phase pixels are colored according to the color key of the standard triangle. The number of α_{WGB} colonies inside each β grain is indicated.

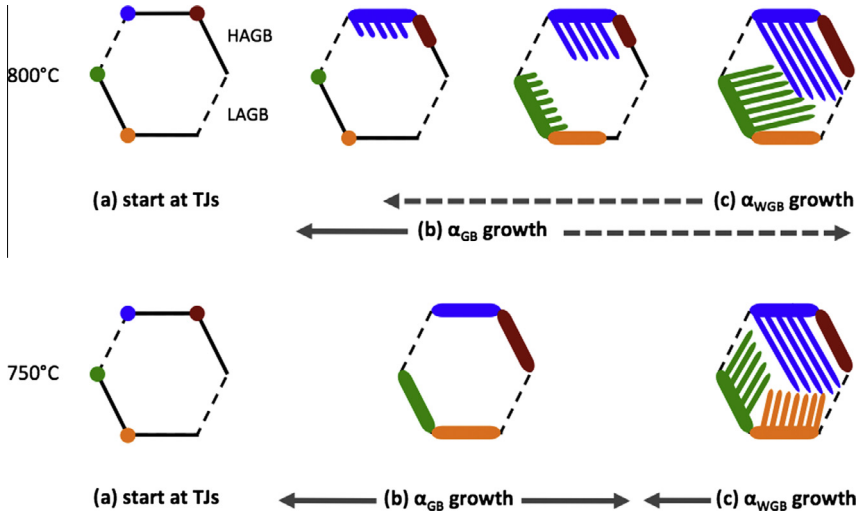

Fig. 6. Schematic of the α precipitation sequence on β/β GB at 800 °C and 750 °C according to three consecutive stages: (a) start of α formation at TJ; (b) α_{GB} growth mostly along HAGB; (c) α_{WGB} growth inside the β grains. At 800 °C, N_{WGB} is lower due to slower α_{GB} formation.

The present results on α_{GB} and α_{WGB} allow these observations to be more precise, with counts based on EBSD maps instead of SEM. Fig. 5 shows representative examples of the α phase microtexture at phase transformation completion at 750, 800 and 830 °C. The main qualitative observation shown by these EBSD maps is the increase in N_{WGB} for decreased transformation temperature. The lower amount of α phase at higher temperatures is in accordance with the thermodynamics of this alloy [2,36]. A statistical number of such EBSD data were analyzed. Table 3 shows the measured average number of α_{WGB} colonies per β grain (N_{WGB}) as well as the number of α_{GB} grains, their precursors, per β GB (N_{GB}), for phase transformation completion at 750, 800 and 830 °C. N_{GB} and N_{WGB} increased with decreasing temperature from 830 to 750 °C: N_{GB} by a factor of 1.3, and N_{WGB} by a factor 2.1. This result is in accordance with Ref. [7]. However, according to the small N_{GB} variation, the N_{WGB} increase cannot simply be explained by a larger number of available precursor α_{GB} per β grain. Neither can this be explained by more frequent emission of α_{WGB} colonies by α_{GB}, as shown in the previous section.

A probable explanation for the larger N_{WGB} at lower transformation temperature is schematized in Fig. 6. One has to compare the detailed evolution of the microstructure at 800 and 750 °C: α_{GB} nucleation, growth and emission of α_{WGB} colonies. The difference between the two temperatures is the kinetics of α_{GB} formation and the α_{WGB} growth stage. Fig. 6 illustrates the consequence at 800 °C. The slow kinetics of α_{GB} formation will lead to a limited number of α_{WGB} colonies that start to grow while some α_{GB} are still growing. The predominant growth of these first colonies will prevent other colonies from forming by occupying the β grain, and this will reduce N_{WGB}. In contrast, at 750 °C, the kinetics of α_{GB} formation is more rapid, leading to earlier simultaneous growth of α_{WGB} on existing α_{GB}. More colonies will be able to grow in the parent β grain, resulting in final larger N_{WGB}. Table 7 illustrates the differences between the duration of α_{GB} and α_{WGB} growth stages. These durations, estimated as the time necessary to fill 80% of the GB by α_{GB} grains (see Section 3.1.2) and to fill 100% of the β grains by α_{WGB} colonies (estimated from previous studies on kinetics [19–21]), respectively, are summarized as follows. It can be seen that, at 750 °C, the time necessary to fill the GB by α_{GB} nucleation and growth is seven times greater than the time for one colony to grow over the grain, while this time is 18 times greater at 800 °C. It can be mentioned that these times are dependent on the β grain size: i.e., for a large grain size, the time necessary for α_{WGB} to fill the grain will increase, allowing other colonies to grow.

4. Summary and discussion

In this contribution, microstructure and microtexture evolution during IT β → α phase transformation was studied in the Ti17 β-metastable alloy with undercooling of 50 °C, 80 °C and 130 °C ($T = 830$, 800 and 750 °C), using

Table 7
Approximate duration to fill the GB with α_{GB} grains and the β grains with α_{WGB} colonies.

Growth stage duration	750 °C	800 °C
α_{GB}	<700 s	<3600 s
α_{WGB}	~100 s	~200 s
α_{WGB}/α_{GB} Stage duration ratio	~7	~18

EBSD and conventional microscopy. Emphasis was put on examining α precipitation at β/β GB. The precipitation sequence and the influence of transformation temperature, schematized in Fig. 6, can be summarized as follows: α formation starting at TJ and/or QP followed by α_{GB} growth along the β/β GB planes, then growth of α_{WGB} colonies inside the β grains at a slower rate than α_{GB}. New observations regarding this sequence were the following.

(1) The number of α_{GB} grains per β/β GB, which was limited to a value of ~2, did not vary significantly with the transformation temperature. The α_{GB} formation started at β TJ.
(2) The α_{GB} formation was faster at lower transformation temperatures. α_{GB} growth was faster on high angle GB (misorientation angle $\theta > 15°$).
(3) As observed in Ref. [7], the number of α_{WGB} colonies per prior β grain was larger at lower transformation temperatures: 7.7 at 750 °C, 4.4 at 800 °C and 3.6 at 830 °C. The present study showed that this was a consequence of faster α_{GB} formation at lower temperatures.
(4) The α_{GB} orientations frequently followed an already established VS criterion based on the alignment of $(0\,1\,1)_{\beta1}$, $(0\,0\,0\,1)_{\alpha}$ and $(0\,1\,1)_{\beta2}$ planes. New observation is that the VS frequency following this criterion was higher at a lower transformation temperature and shorter transformation duration.
(5) Many α_{GB} grains that followed the VS criterion (10–30%) emitted two α_{WGB} colonies on either side of the β/β GB: not only into the β grains in BOR with α_{GB}, but also into the other β grains without or with α_{GB}. In contrast, α_{GB} without particular orientation almost never emitted one colony into the non-BOR adjoining β grains.
(6) The α_{WGB} colonies were always in BOR with their host β grains, as already observed. The present study showed that colonies forming inside the non-BOR β grains were misoriented from their precursor α_{GB} grain by an angle of nearly 5°, while colonies forming inside the β grains in BOR with α_{GB} inherited its exact orientation, with the precision of EBSD measurement.

The purpose of these observations was to examine the influence of the transformation temperature on the microtexture development associated with α precipitation at β/β GB. Interpreting the α microtexture at the scale of the prior β grains in terms of the average number of α orientations per β grain and considering their orientations, the main conclusion is that decreasing the transformation temperature tends to weaken the α microtexture, because of more α_{WGB} colonies forming inside the β grains, thus increasing the number of α orientations. Each colony is associated with one specific orientation, according to the observations. As mentioned above, the number of colonies varied from 3.6 when transformation occurred at 830 °C to 7.7 when transformation occurred at 750 °C. The dominant factor controlling the microtexture was the variation in number of colonies, although VS occurred extensively, often making the individual orientation of the colonies depend on the crystallographic parameters of the β/β GB from which they emanated. If decreasing the transformation temperature increased the frequency of VS occurrence (from 36% at 830 °C to 45% at 750 °C, at phase transformation completion, at β/β GB with $\omega < 4°$), the quantitative effect was not significant enough to expect noticeable α microtexture strengthening compared with the number of α_{WGB} colonies variations. It can be mentioned that a similar limited effect of VS has been put into evidence recently in Ref. [37], but considering the macroscopic α phase transformation texture instead of the microtexture. In this study, the authors made the prior β grain texture vary, and they showed that the α phase transformation texture was more controlled by this parameter than by the occurrence of VS.

In addition, the present study showed that, to understand the formation of the microtexture associated with α_{WGB} colonies, one has to examine their precursors, i.e., the α_{GB} grains that form at early stages of precipitation. By studying the kinetics of α_{GB} formation, it was found that the increased number of colonies at a lower transformation temperature was due to the more rapid α_{GB} formation along the β/β GB compared with the growth rate of α_{WGB}. One notable finding was to clarify the common interpretation, based on enhanced nucleation. Focusing on α_{GB} grains was also necessary in order to study VS, because the α_{WGB} colonies cannot form directly on β/β GB. A striking result was the more frequent occurrence of VS at shorter transformation duration and increased undercooling. Current interpretations of VS in the literature did not make it possible to explain this trend satisfactorily. Probably, the energy barrier associated with α_{GB} with favorable orientations is lower. Numerical simulations will help in further analyses of VS for heterogeneous α precipitation at β/β GB. The other result was the more frequent emission of α_{WGB} colonies by α_{GB} that followed the VS criterion. As a result, a large proportion of the α phase emanated from β/β GB with close $(0\,1\,1)\beta1$ and $(0\,1\,1)\beta2$ planes.

Finally, it may be emphasized that some observations in the present study should not be generalized too fast if one is considering a titanium alloy with a chemical composition different from that of Ti17. Depending on the alloy composition, the nucleation of α may be possible at the middle of GB instead of being restricted to TJ and/or QP. This would modify the α_{GB} formation sequence from the beginning, compared with the sequence that the present authors observed. The VS frequency of occurrence as a function of temperature and exposure time also probably depends on the alloy composition, as it was shown that it depends on the transformation driving force. The prior β grain size also is an important parameter, as it influences the number of α_{WGB} colonies per β grain.

5. Conclusion

The transformation temperature influence on the α microtexture development associated with the α precipitation at β/β GB in the near β Ti17 alloy was studied using EBSD. By interpreting the α phase microtexture at the scale of prior β grains (~200 μm), it was found that, for the alloy studied and the temperature range considered, decreasing the transformation temperature decreased the microtexture strength because of a greater number of α_{WGB} colonies, each colony increasing by one the number of α orientations inside the β grains. The greater number of colonies was due to faster formation of their precursors, the α_{GB} grains, as revealed by a detailed examination of their formation. α_{GB} crystallographic orientations followed extensively a VS criterion based on the alignment of $(0\,1\,1)_{\beta}//(0\,0\,0\,1)_{\alpha GB}//(0\,1\,1)_{\beta}$. From a statistically relevant number of observations, VS was found to be more frequent at a lower transformation temperature, but the effect was not significant enough to influence the α microtexture, at the scale of one single prior β grain. The α_{GB} precursors that followed the VS criterion emitted on average more α_{WGB} colonies than those without particular orientation. Only such α_{GB} grains emitted two colonies on either side of the β/β GB. As a result, a large proportion of the α_{WGB} colonies (but not all) emanated from β/β GB with close $(0\,1\,1)\beta1//(0\,1\,1)\beta2$.

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