Rare Metal Materials and Engineering
Volume 49, Issue 1, January 2020
Available online at www.rmme.ac.cn

Science Press

Cite this article as: Rare Metal Materials and Engineering, 2020, 49(1): 0068-0074.

ARTICLE

Massive Transformation Mechanisms in TG6 Titanium Alloy During Cooling

Sun Feng[1,2], **Liu Xianghong**[1], **Li Jinshan**[2], **Wang Kaixuan**[1], **Zhang Fengshou**[1]

[1] Western Superconducting Technologies Co., Ltd, Xi'an 710018, China; [2] State Key Laboratory of Solidification Processing, Northwestern Polytechnical University, Xi'an 710072, China

Abstract: A kind of massive microstructure α_m along the primary α phase (α_P) has been characterized in the microstructure quenched from the $\alpha+\beta$ phase field. The obvious interface between α_m and α_P can be found under the optical observation, and the concentrations of Al, Sn and Zr elements in α_m are in between that of the α_P and β matrix, but the orientation of the α_m keeps consistent with that of the α_P as evidenced by EBSD analysis. The appearance of the α_m is considered as the result of limited diffusion controlled transformation from the β phase during the cooling, because the α_m initiates from the α/β interface and keeps the consistent orientation with α_P, and the growth is limited around the α/β interface. As the diffusion is limited by decreasing the solution periods from 30 min to 5 min, and the dissolving of α_P is accelerated by increasing the solution temperature from 1040 ℃ to 1060 ℃, the volume fraction of α_m increases from 5.2% to 30.7% significantly.

Key words: near α titanium alloy; massive microstructure; solute redistribution; solution treatment; limited diffusion

Near α titanium alloy TG6, with nominal composition of Ti-6Al-4Sn-4Zr-1.5Ta-0.7Nb-0.4Si-0.06C (wt%), has been widely used in advanced aero-engines to reduce about 40% weight, when replacing the superalloy[1-3]. The alloy can be solution treated in $\alpha+\beta$ phase field to achieve excellent performances[4-6]. During the solution treatment, unsaturated α phase dissolves accompanying with the static recrystallization, and most of the β matrix transforms to α' martensite during quenching[7, 8]. The volume fraction and morphology of α phase are considered as important features closely related to mechanical properties[9,10].

The morphology of α phase in $\alpha+\beta$ alloy during solution treatment has been investigated[11,12]. The secondary α phase (α_s) dissolves at much lower temperature than α_P phase does, because such acicular α_s is enriched with more β stabilized elements than α_P. Besides, the evolution of α_P phase can be classified into three stages. In the initial stage, the separation of α lamellar is dominated by the boundary splitting mechanisms; then the α_P phase becomes more equiaxed in the secondary stage based on the termination migration; in the third stage, the Ostwald ripening will happen if the period of solution is enough [13].

The acicular α' martensite nucleates and grows up in the β matrix during the quenching. The morphology and crystal structure of martensite are mainly determined by the chemical composition of β matrix[14]. In some cases, α_m phase, as parallel slats, has been mentioned as another kind of hexagonal martensite transformed from the β matrix enriched with solute atoms in Ti6Al4V alloy, but the conditions required by its nucleation and growth are still unknown[15,16]. Recently, an epitaxial growth behavior of equiaxed α phase during cooling has been studied, and the results indicates that local composition difference leads to the growth of rim-α phase based on a diffusion-controlled model[17].

The stabilities of α and β depend on the concentration of solute atoms, such as Al and Mo with contrast influence. In this study, the Mo is replaced by Sn to develop the TG6 alloy, and the solution treated microstructure are compared to clarify the influence of Mo on the growth behavior of α phase during cooling. Moreover, the microstructures treated above T_β for several minutes is compared with that of $\alpha+\beta$ solution treatment for hours, to clarify the relationship between epi-

---

Received date: January 25, 2019

Corresponding author: Sun Feng, Ph. D., Senior Engineer, Western Superconducting Technologies Co., Ltd, Xi'an 710018, P. R. China, Tel: 0086-29-86514525, E-mail: yfbsf@c-wst.com

taxial growth of *α* phase and redistribution of solute atoms.

In this study, the TG6 alloy is treated at temperatures from T_β-10 ℃ to T_β+10 ℃ for different periods. TEM, EBSD and EDS are used to identify the morphology, crystal structure and chemical composition of *α* phase. The different epitaxial growth behaviors of *α* phase are compared and discussed.

1 Experiment

The as-received TG6 alloy used in the experiment experienced triple self-consumable vacuum arc remelting and *α*+*β* processes. The *β* transus temperature (T_β) is 1050 ℃. As-received microstructure consists of equiaxed *α* grains with about 40μm in size, shown in Fig.1. TG6 alloy specimens were solution treated in the *α*+*β* field from 990 ℃ to 1040 ℃ for 0.5~4.0 h and at 1060 ℃ for 3~15 min followed by water quenching, as shown in Table 1. Then the cross-sections of the specimens were polished and etched with the solution of 8% HF and 92% H_2O for optical micro-observation. Specimens for the EBSD analysis were electro-polished in a solution of 5% perchloric acid and 95% alcohol. The EDS and EBSD scans were carried out on the SUPRA_55 equipment. Phase identification was performed by X-ray diffractometry with a DX-2700 instrument using Cu K*α* radiation. A Tecnai F30 transmission electron microscope was also used, where thin foils were prepared by the twin-jet electro-polishing using a solution of 6% perchloric acid and 35% butyl alcohol in methanol at −20 ºC.

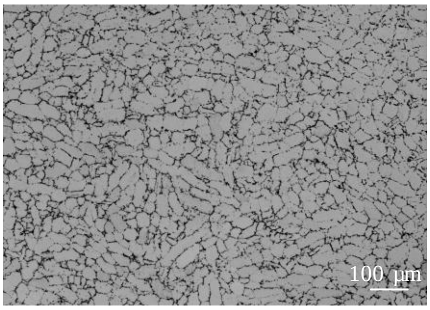

Fig.1 As-received microstructure in the forging bar

Table 1 Solution treatment conditions for TG6 alloy

Condition	Thermal history	
α+*β* solution	1040 ℃/1000 ℃, 30 min/240 min	WQ
β solution	1060 ℃/1040 ℃, 5 min	

2 Results and Discussion

2.1 Microstructures treated at 1040 ℃ and 1000 ℃

The microstructures solution treated at 1040 ℃ and 1000 ℃ are compared in Fig.2. They consist of large white α_p particles and acicular *α*′ laths. At low temperature of 1000 ℃, most of the α_p particles even connect with each other, and the percentage of area approximates to 61.5%, the boundaries of *α*/*β* interface prefers to be straight other than curving. All the retained *β* phase transforms to acicular *α*′ martensite, which has been evidenced by the XRD results shown in Fig.3. None of the *β* peaks can be distinguished from the diffraction patterns. In detail, some short straight *β*/*β* boundaries can be found as connection between adjacent α_p particles. It indicates that the static recrytallization starts at 1000 ℃. It is obvious that not only α_p and *α*′ phase exist in the microstructure, but a gray layer α_m of several micrometers thickness can be observed covering the α_p particles discontinuously.

At 1040 ℃, more α_p particles dissolve, and the original *β* grains grow up. The α_p particles are reserved inside the grain or in the junctions of grain boundaries. In detail, the curvature of the α_p particles increases significantly, which indicates the driving force of spheroidization increases with temperature. Besides, the area of α_m layer increases at high solution temperature, and the *β* matrix away from the α_p particles still transforms to acicular *α*′ martensite. Significantly, the α_m prefers to grow from the *α*/*β* interface with large curvature to the *β* matrix. Such anisotropic growth of the α_m layer can also be observed along the *β* grain boundaries, as shown in Fig.2b. The *β* grain boundaries have been occupied by the α_p particles dissolving during solution treatment. Moreover, the α_m layer still shows no any change in morphology and distribution even when the solution time increases from 30 min to 240 min.

The α_p volume fractions of TG6 titanium alloy at different solution temperatures were plotted and compared with IMI834, Ti-811 and Ti6Al4V such near *α* or *α*+*β* titanium alloys[18-20]. As shown in Fig.4, the volume fraction of α_p phase not including α_m approximates to that of IMI834 alloy. It indicates that the α_m phase is not part of α_p phase retained after $\alpha \rightarrow \beta$ transformation. The transformation of $\beta \rightarrow \alpha_m$ is supposed to occur during the cooling.

The solute concentrations of α_p, *α*′ and α_m phases are also compared in the microstructures experiencing different solution treatments. As shown in Table 2, the α_p phase is enriched with Al element, and Sn and Zr aggregate in *α*′ phase. The concentration of α_m phase falls in between that of the α_p and *α*′ phases. Such a transition area plays the critical role in formation of α_m phase instead of *α*′ phase during cooling. Moreover, the morphology of α_m phase is related to the curvature of *α*/*β* interface and volume fraction of α_p phase.

2.2 Microstructures treated rapidly at 1060 ℃ and 1040 ℃

It is noted that α_m phase nucleates and grows up in the *β* matrix near the *α*/*β* interface with transitional concentration. A diffusion limited mechanism can be connected with the formation of such transitional area. Therefore, the diffusion

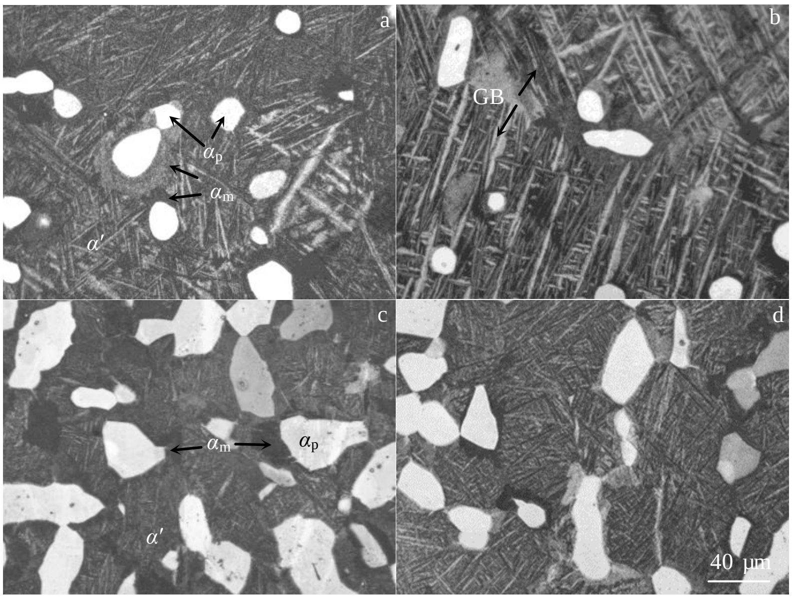

Fig.2 Microstructures solution treated under 1040 ℃, 30 min (a); 1040 ℃, 240 min (b); 1000 ℃, 30 min (c); 1000 ℃, 240 min (d)

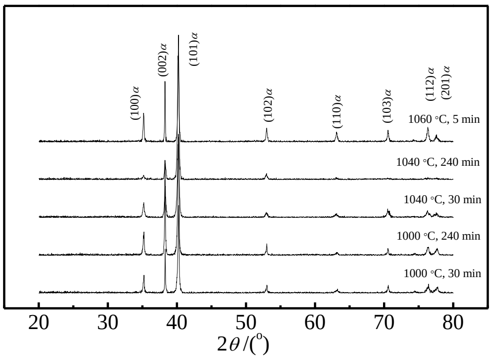

Fig.3 XRD patterns of the microstructures solution treated at 1040 ℃ and 1000 ℃ for 30 min and 240 min, 1060 ℃ for only 5 min

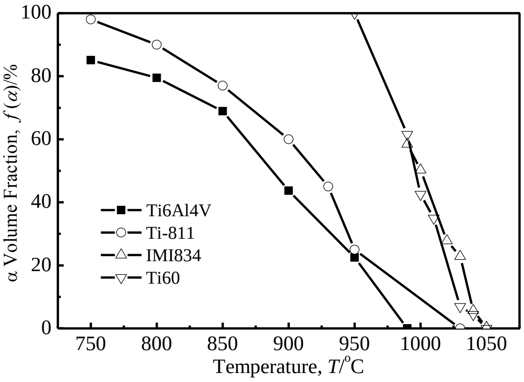

Fig.4 α volume fraction curves of Ti6Al4V, Ti-811, IMI834 and Ti60 alloys [18-20]

Table 2 EDS analysis of the α_p phase, martensite α' matrix and massive α_m phase after solution treatments for TG6 alloy

Solution treatment	Phase	Volume fraction/%	Average chemical composition/wt%		
			Al	Sn	Zr
1040 ℃, 30 min	α_p	4.4	5.76	4.22	2.99
	α_m	5.2	4.98	4.85	3.33
	α'	Balance	4.70	4.79	3.64
1000 ℃, 30 min	α_p	61.5	5.42	3.93	3.02
	α'	Balance	4.76	4.33	3.70
1060 ℃, 5 min	α_p	5.3	5.79	4.14	3.27
	α_m	30.7	5.3	4.25	3.41
	α'	Balance	4.99	4.55	3.64

of solute atom during redistribution can be limited by shortening the solution time, and the $\alpha \rightarrow \beta$ transformation kinetics can be accelerated by increasing the solution temperature. In order to clarify the mechanism of nucleation and growth of α_m phase, the alloy is solution treated at 1040 ℃ and 1060 ℃ for 5 min.

As shown in Fig.5, the α_p particles dissolve rapidly at 1060 ℃, and the percentage of α_p phase decreases to 5.3% after 5 min solution treatment. Similarly, nearly 4.4% α_p phase is retained after solution treatment at 1040 ℃ for 30 min. However, 30.7% α_m phase forms around the α_p particles after rapid treatment, but only 5.2% α_m phase can be observed in the microstructure treated at 1040 ℃ for 30 min.

As for the treatment at 1040 ℃, the amount of α_m phase also increases as the period decreases, although the amount of α_p particles is more than that at 1060 ℃.

It is evidenced that the α_m phase prefers to nucleate and grow in the concentration transition area where the redistribution of solute atom is limited by reducing the solution period.

2.3 Characteristics of α_m phase under TEM

It can be found that both of the α_m and α' phases nucleate and grow up during quenching, but the morphologies of them are various. The interface of α_m and α' phases is magnified under TEM, and the selected diffractions of them and the interface are compared in Fig.6b, 6c and 6d. The hcp structure of the α_m is evidenced as the α' phases, and no relationship can be found between their orientations. Two α' lamellar related to the pattern marked with "DF" in Fig.6d are indicated in dark field, as shown in Fig.6e. The patterns in Fig.6d include both of α_m and α' phases. As shown in Fig.6f, the tip of the acicular α' phase is embedded in α_m phase. The lattice mismatch between the two phases induces lots of dislocations aggregated around the tip. Besides, an interface as subgrain boundary in α_m phase marked as "SGB" can be observed in Fig.6a.

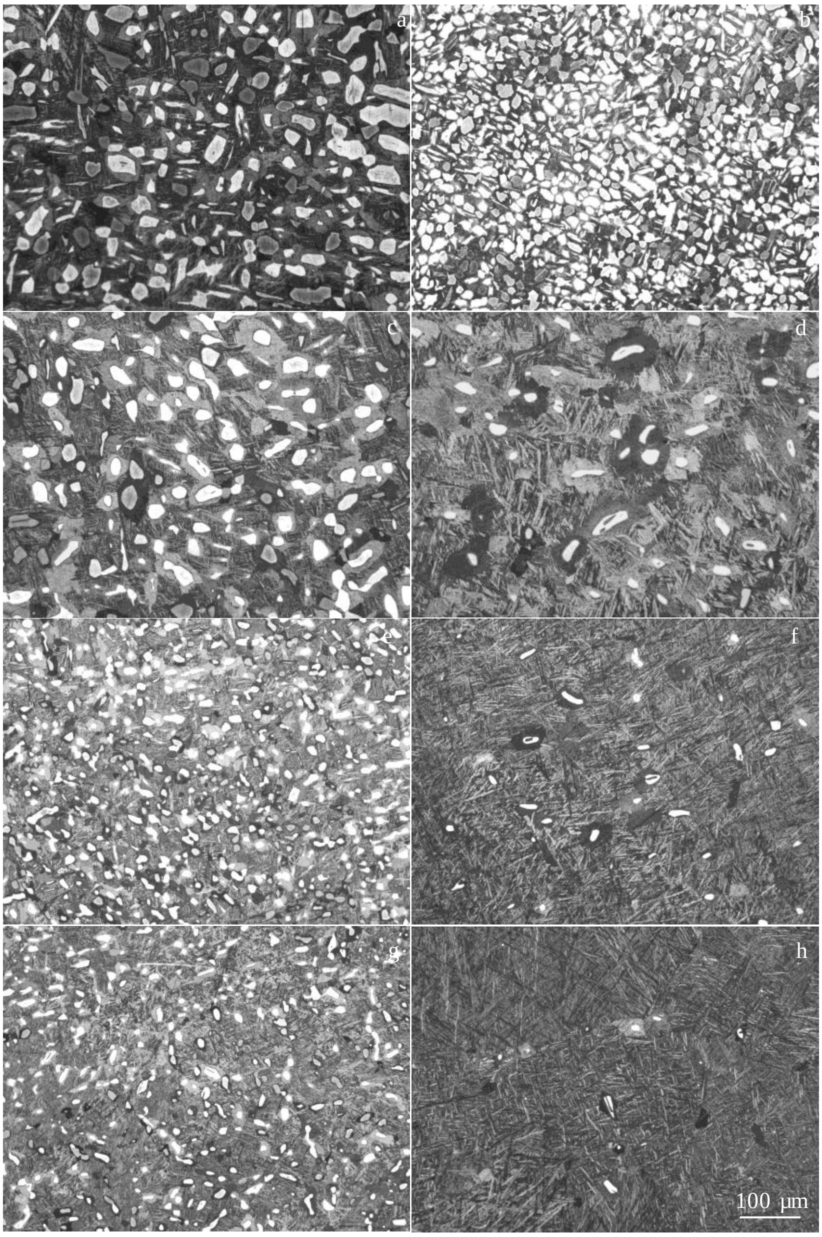

Fig.5 Microstructures solution treated at 1040 ℃ (a, c, e, g) and 1060 ℃ (b, d, f, h) for 3 min (a, b), 5 min (c, d), 8 min (e, f) and 15 min (g, h) followed by quenching

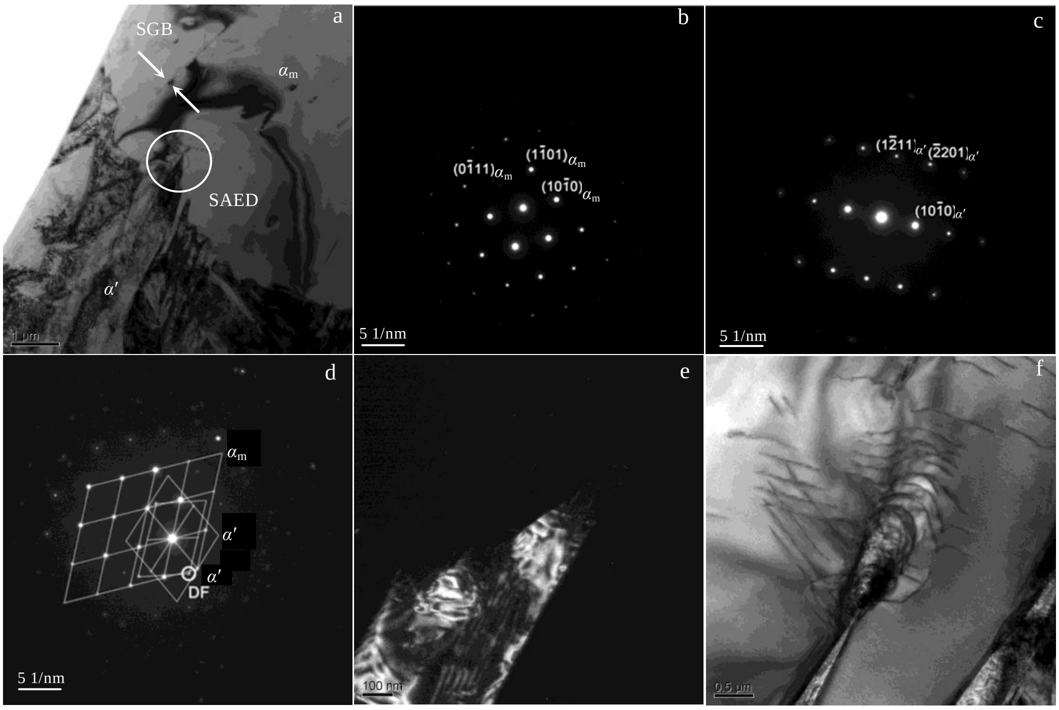

Fig.6 TEM observation of the α_m and α' phase interface microstructure solution treated at 1040 ℃, including bright field image (a), selected area electron diffraction (SAED) patterns of α_m (b), α' phases (c) and their interface marked in Fig.6a (d), and the corresponding dark field (DF) image (e) for a single martensite lath, dislocations aggregated around the tip (f)

3 Discussions

During the fast cooling from the high temperature in $\alpha+\beta$ phase field, a common type of $\beta\rightarrow\alpha$ transformation is the formation of α' martensite phase with similar composition as β matrix phase. The start of α' martensite phase transformation temperature (M_s) approximates to 900 ℃ evidenced in previous research[9]. The great driving force induced by the undercooling is high enough to encourage a large amount of α' acicular to nucleate inside the β matrix and grow up with Burgers orientation relationship (OR) with parent β phase. The interfaces with such OR dominate the growth and result in acicular profile of α' phase due to the lower interface energy compared with other ORs.

As for the slow cooling condition, the α phase prefers to grow from the grain boundaries to the grain inside to form the Widmannstatten microstructure[21]. The growth orientation of the parallel α lamellar is related to the grain boundary α phase (α_{GB}), and also keep Burgers OR with β matrix resulting from the low interface energy. The kinetics of such $\beta\rightarrow\alpha$ transformation is determined by the diffusion velocity of solute atoms. The driving force of nucleation reduces due to low undercooling. The number of nucleation sites decreases, and they are limited along the grain boundaries.

After the solution treatment, when the diffusion of solute atoms cannot accompany with the spheroidizing of α_p phase, a transient zone forms around the α_p particles with middle concentration of solute atoms. During the quenching, the driving force of $\beta\rightarrow\alpha$ transformation in this zone is lower than that of grain inside. Such $\beta\rightarrow\alpha$ transformation starts earlier than the M_s in TG6 alloy. The α phase grows from the α_p particles to the grain inside similar to the Widmannstatten microstructure, and it keeps the same orientation with parent α_p phase, which is evidenced by EBSD results indicated in Fig.7.

However, the diffusion of solute atoms is limited by the fast decreasing of temperature and short period during quenching. The α phase can not grow up to form the crossed acicular or parallel lamellar, and the migration of α/β interface is limited not exceeding the concentration transient zone. Finally, the α_m phase only appears around the α_p particles, and the growth is related to the area of the transient zone. Besides, the profile of α_m phase is not as smooth as observed under TEM and EBSD, which also can be considered as the tendency of the growth of α lamellar. Once the temperature approximate to the M_s, the retained β phase will transform to α' martensite as soon as possible. The tips of α' phase contact with α_m phase inducing inner stress and the formation of dislocations.

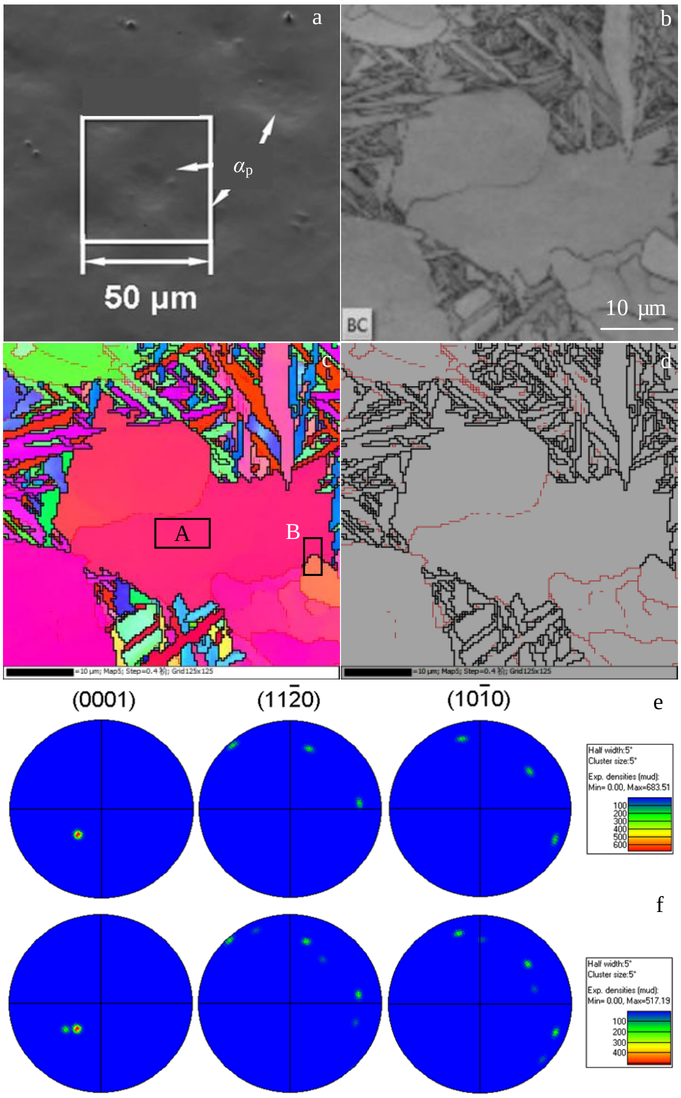

Fig.7 EBSD images of one single α_p particle treated at 1040 ℃ for 30 min, including the surface morphology (a), diffraction contrast image (b), orientation relationship (c) and statistic of boundaries (d), and the pole figures belonging to the marked area A (e) and area B (f) in Fig.7c

4 Conclusions

1) The massive α_m phase is evidenced in the microstructure of TG6 alloy after the cooling from the temperature in $\alpha+\beta$ phase field.

2) The growth of α_m phase starts from the α_p/β interface and is limited in the concentration transition zone around the α_p phase. The orientation keeps the same as the α_p phase. Once the volume fraction of α_m phase increases, the growth area of α_m phase reduces since the concentration transition zone become smaller.

3) If the solution temperature increases to the 1060 ℃ above T_β from 1040 ℃, and the time is compressed from 30 min to 5 min, the volume fraction of α_m phase increases from 5.2% to 30.7%, and that of α_p phase keeps in 5% level. The diffusion between α_p and β phase is limited during redistribution due to the reduction of time, and the growth area of α_m phase increases.

References

1 Banerjeea D. *Acta Materialia*[J], 2013, 61(3): 844

2 Winstone M R, Partridge A, Brooks J W. *Proceedings of the Institution of Mechanical Engineers Part L: Journal of Materials Design & Applications*[J], 2001, 215(2): 63

3 Eylon D, Fujishiro S, Postans P J *et al. The Journal of the Minerals, Metals & Materials Society*[J], 2013, 36(11): 55

4 Wang Tongbo, Li Bolong, Wang Zhenqiang *et al. Materials*

Science and Engineering A[J], 2018, 731(25): 12

5 Sun Feng, Li Jinshan, Kou Hongchao *et al. Materials Science and Engineering A*[J], 2014, 626(25): 247

6 Ji Zhe, Chen Yuhao, Qiang Yinghuai *et al. Materials Science and Engineering A*[J], 2018, 723(18): 29

7 Balachandran S, Kumar S, Banerjee D. *Acta Materialia*[J], 2017, 131(1): 423

8 Sun Feng, Li Jinshan, Kou Hongchao *et al. Journal of Alloys & Compounds*[J], 2013, 576(5): 108

9 Li C, Chen Jianlin, Li W *et al. Journal of Alloys and Compounds*[J], 2015, 627(5): 222

10 Sun Jianfeng, Zhang Zhongwu, Zhang Milin *et al. Journal of Alloys and Compounds*[J], 2016, 663(5): 769

11 Zherebtsov S, Murzinova M, Salishchev G *et al. Acta Materialia*[J], 2011, 59(10): 4138

12 Ma Xiong, Zeng Weidong, Tian Fei *et al. Materials Science & Engineering A*[J], 2012, 548(30): 6

13 Wu Chuan, Yang He, Li Hongwei *et al. Computational Materials Science*[J], 2013, 79(1): 944

14 Hammond C, Kelly P M. *Acta Metallurgica*[J], 1969, 17(7): 869

15 Ahmed T, Rack H J. *Materials Science & Engineering A*[J], 1998, 243(1-2): 206

16 Pilchak A L, Broderick T F. *The Journal of The Minerals, Metals & Materials Society*[J], 2013, 65(19): 636

17 Gao Xiongxiong, Zeng Weidong, Zhang Saifei. *Acta Materialia*[J], 2017, 122(1): 298

18 Malinov S, Markovsky P, Sha W. *Journal of Alloys & Compounds*[J], 2002, 314(1-2): 181

19 Malinov S, Guo Z, Sha W *et al. Metallurgical & Materials Transactions A*[J], 2000, 32(11): 879

20 Malinov S, Sha W, Markovsky P. *Journal of Alloys & Compounds*[J], 2003, 348(1-2): 110

21 Appolaire B, Héricher L, Aeby-Gautier E. *Acta Materialia*[J], 2005, 53(10): 3001

TG6 钛合金冷却过程的块状转变机制研究

孙 峰[1,2]，刘向宏[1]，李金山[2]，王凯旋[1]，张丰收[1]

(1. 西部超导材料科技股份有限公司，陕西 西安 710018)

(2. 西北工业大学 凝固技术国家重点实验室，陕西 西安 710072)

摘　要：在近 α 型 TG6 钛合金两相区淬火过程中，对初生 α 相 $\alpha_{\rm P}$ 周围形成的块状组织 $\alpha_{\rm m}$ 进行表征研究，块状组织 $\alpha_{\rm m}$ 与 $\alpha_{\rm P}$ 存在明显的界面，合金元素 Al、Sn 和 Zr 浓度介于 $\alpha_{\rm P}$ 和 β 基体之间，EBSD 分析结果表明 $\alpha_{\rm m}$ 取向与 $\alpha_{\rm P}$ 保持一致。在冷却过程中，$\alpha_{\rm P}$ 周围 β 基体中合金元素扩散受到限制，进而在局部过渡成分区域通过扩散转变形成 $\alpha_{\rm m}$ 组织。当固溶温度从 1040 ℃提高至 1060 ℃，同时保温时间从 30 min 减少为 5 min，溶质元素扩散受限，$\alpha_{\rm m}$ 体积分数从 5.2%提升至 30.7%

关键词：近α钛合金；块状组织；溶质再分配；固溶处理；有限扩散

作者简介：孙 峰，男，1986 年生，博士，高级工程师，西部超导材料科技股份有限公司，陕西 西安 710018，电话：029-86514525，E-mail: yfbsf@c-wst.com