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# Effects of network-distributed TiB on dynamic recrystallization of TiB/(TA15-Si) composites during the deformation in β phase region

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**Abstract.** To study the effects of network distributed TiB on dynamic recrystallization of high temperature β phase of near α titanium matrix composites, the TiB/(TA15-Si) composites were compressed under 1000°C with strain rates ranging from 1 $s^{-1}$~0.01 $s^{-1}$. Results show that the flow stress was sensitive to strain rates. During hot compression, the microstructure evolution included single TiB rotation, network structure changing from circular to oval, and dynamic recrystallization (DRX) of high temperature β phase. The mechanism of DRX was continuous and discontinuous DRX mixed mechanism. Attributed to the accelerative effects of TiB by providing nucleation sites and improving storage energy, DRX preferentially occurred around TiB. After compression, equiaxed grains of high temperature β phase were formed in TiB rich region, while elongated grains were formed in TiB lean region. The dislocations density in elongated grains at higher strain rate was higher than that at lower strain rate. Under high strain rate, high temperature β phase was dominated by DRX in TiB rich region and dynamic recovery in TiB lean region. Under lower strain rates, high temperature β phase was dominated by the growth of DRX grains.

## Introduction

With the rapid development of aero engines, it is urgent to develop lightweight and heat-resistant structural materials with service temperatures above 650°C. Due to the excellent mechanical performance and low manufacturing cost, titanium composites with network structure have received extensive concern [1,2]. Compared with matrix alloy, the service temperature of network structured composites could be improved by 100°C~200°C [3]. Therefore, network structured TiB/(TA15-Si) composites are expected to replace nickel-based alloys to be serviced at 650°C.

Generally, metal materials undergo hot deformation to optimize the microstructure and performance [4,5]. For titanium matrix composites with network structure, the deformation in β phase region has more research value and practical significance. Firstly, the deformation resistance of titanium alloy in α+β phase region is greater than that in β phase region, and the addition of reinforcements could increase the deformation resistance, which makes the deformation of the composites in α+β phase region require higher her tonnage of the press machine. Thus, the composites have more anvantages as deformed in β phase region. Secondly, for near α titanium alloys, it is usually prohibited to forge in β phase region as finished forging of products, which is because fast growth of β phase results to β brittleness. For network structures composites, TiB restricts the grains size within the size of a titanium alloy powder [6]. As well, during the following cooling process, the continuous α phase at the grain boundary can also be avoided to be formed because of TiB, thus it is more likely to prevent the β brittleness. Thirdly, in practice, to avoid β brittleness of near α titanium alloys, the hot deformation is required to be completed in a short time as deformed in β phase region. The total deformation needs to be completed through small deformation in pass with multi-pass, which reduces the production efficiency and wastes energy. For network distributed composites, it has obvious advantages by TiB limiting the growth of high temperature β phase, decreasing the requirements for deformation time, increasing the pass deformation, and then reducing the number of passes. Above all, the deformation of network structured TiB/(TA15-Si) composites in β phase region is necessary to be studied and some investigation has been done [7,8,9]. The results show that the performance was enhanced after deformation in β phase region, and the strength increased with deformation passes. However, those researchers paid close attention to the strength improvement. The deformation behavior and microstructure evolution of the composites in β phase region needs to be further studied.

In this paper, the hot deformation behavior of the composites was investigated through hot compression at 1000℃ with different strain rates. The microstructure evolution after compression was studied by scanning electron microscope (SEM) and electron backscattering diffraction (EBSD) characterization, and the effects of TiB on microstructure were analyzed.

**Material and experimental procedures**

Initial material. TiB/(TA15-Si) composites with network structure were utilized in the present study. The as-received composites were sintered billet with 3.5% volume fraction of TiB and 0.5% weight fraction of Si. The microstructure of the as-sintered sample is shown in Fig. 1(a), which consisted of lamellar α phase, β phase and network distributed TiB. In order to study the deformation behavior of the composite in single β phase region, the β transformation temperature was determined to be 995℃ by metallographic method. As the sample was heated to 1000℃ and held for 20 min, the α phase disappeared and transformed to β phase completely, as shown in Fig. 1 (b). The high temperature β phase of the as-sintered sample was reconstructed, as shown in Fig. 1 (c). The grain boundary of the reconstructed β phase coincides with the position of network-structured TiB, and the size of the prior β grain is about 120 μm.

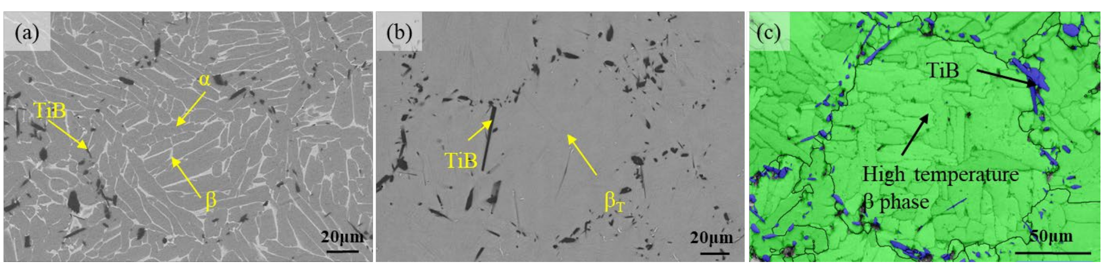


*Fig. 1. SEM of the sample with different conditions: (a) as-sintered; (b) after heat treatment at 1000℃ for 20 min; (c) phase maps of reconstructed β phase of as-sintered.*

Experimental procedures. The cylindrical samples with ϕ8 × 12 mm were compressed on a universal material testing machine (Japan Shimadzu AG-X Plus 250 kN/50 Kn). To reduce friction, the samples' surface was uniformly coated with the antioxidant of glass protective lubricant, and a graphite sheet was placed between the samples and indenters. The samples were held at 1000℃ for 20 min before deformation and then were compressed with a reduction of 60% at strain rates of 1 $s^{-1}$ and 0.01 $s^{-1}$ respectively. After the compression, the samples were water quenched immediately.

To investigate the microstructure evolution during the deformation, the microstructure was characterized by SEM (Hitachi Scanning Electron Microscope SU 5000) and EBSD. The deformed samples were ground mechanically then electrolytic polished for 90 s at a voltage of 30V and current of 0.68A ~ 0.72A. The electrolytic solution was composed of 60% methanol, 34% butyl alcohol and 6% perchloric acid and kept at -30℃. The polished samples were etched in Kroll solution for 5 seconds for TiB observation. AZtecCrystal software was used to analyze the EBSD data. The reconstruction of high temperature β phase was accomplished using the function of Parent Grain Analysis in AZtecCrystal software. The main parameter setting were as follow: the parent phase and child phase was selected as β phase and α phase of titanium based on the acquired data information, the orientation relation was the Burgers relationship including $\{110\}_{\beta}//\{0001\}_{\alpha}$ and $<\text{-}11\text{-}1>_{\beta}//<2\text{-}1\text{-}10>_{\alpha}$, the refinement range was 10°, and the window size, step width, voting thresh, and parent thresh were 20 pix, 25%, 5° and 10°, respectively.

## Results and Discussion

Hot deformation behaviors. Fig. 2 shows the true stress-strain curves of the samples compressed at 1000℃ with different strain rates. It is found the flow stress increases rapidly at the initial stage of the deformation due to work hardening effect, then the increase rate of stress decreases, and the flow stress gradually increases until the peak stress is reached. After that, softening of flow stress decreases gradually. The softening behavior may be related to the DRX of matrix [10] and evolution of the reinforcements [11]. It is noted that when the strain rate is $0.01s^{-1}$, the flow stress shows a slow increase trend in the late stage of the deformation. By comparing the curves under different strain rates, the flow stress is positively correlated with the strain rate when the composite is formed in β phase region.

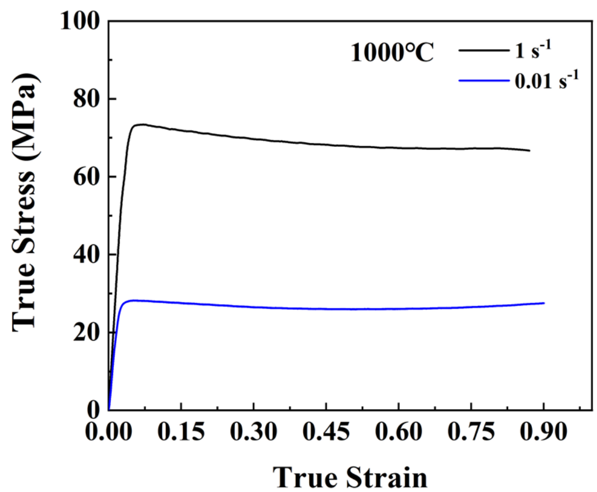


*Fig. 2. Stress-strain curves of the samples compressed at 1000℃ with different strain rates.*

Microstructure evolution. Fig. 3 shows the SEM microstructure of the deformed samples in the center position under different strain rates. It is found that the TiB is still distributed in a circular network structure in the center of the cross section, while in a oval network structure of the longitudinal section. Moreover, the direction of a single TiB is perpendicular to the compression direction. The reason is the lower resistance of high temperature β phase to deformation, resulting in the rotation of TiB with the flow of matrix. In addition, fine β grains are found, as marked in yellow circle in Fig. 5(c) and (f), which indicates the occurrence of dynamic recrystallization of high temperature β phase during the compression.

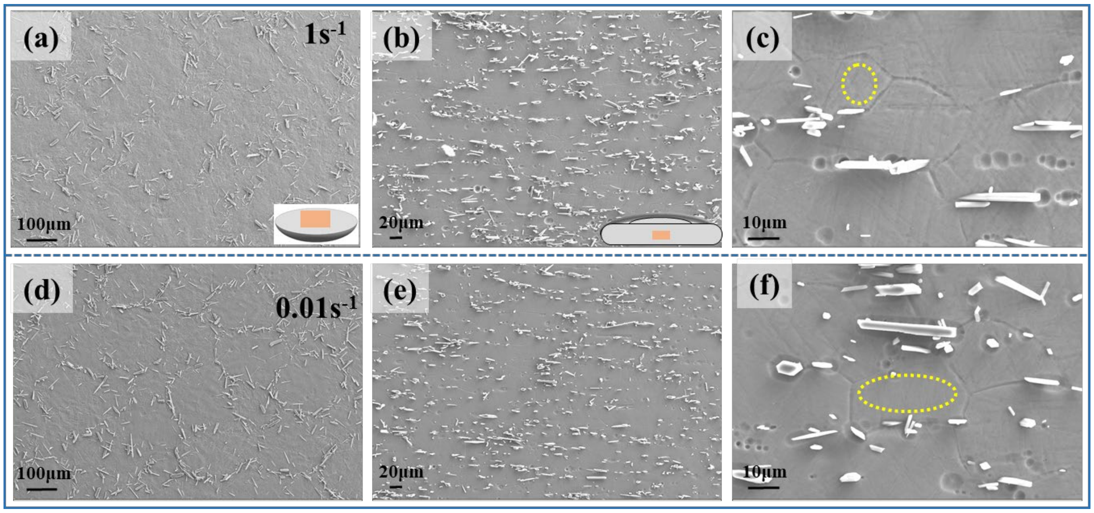


*Fig. 3. SEM microstructure of the deformed samples in the center position under different strain rates: (a, b and c) 1 $s^{-1}$; (d, e and f) 0.01 $s^{-1}$; (a, d) in the cross section; (b, e) in the longitudinal section; (c, f) high magnification of figure (b) and (e).*

Fig. 4 shows the EBSD results of the reconstructed β phase of the deformed samples under different strain rates. As the strain rate is $1s^{-1}$, fine β grains can be seen around TiB. As marked in yellow arrows in Fig. 4(a), the fine grains exhibit a distinct misorientation with the near and elongated grains, which is a typical characteristic of discontinuous dynamic recrystallization (DDRX). Additionally, sub-grains with low-angle grain boundaries are found within the deformed grains, as shown in the white arrows in Fig. 4(a). The misorientation of the sub-grains is close to that of the surrounding grain, which is a characteristic of continuous dynamic recrystallization (CDRX). For near α titanium alloy, the microstructure evolution during the deformation in β region is dominated by the recovery (DRV) of the β phase. However, TiB can promote the DRX of β phase by supplying the nucleation sites [12]. In this paper, the DRX of β phase occurs in TiB rich region. In the TiB lean region, it can be found that a slight difference in color within the grains, as shown in the yellow frame in Fig. 4(a), which suggests there are certain dislocations inside the grain. Thus, the elongated grain is dominated by DRV. Thus, at the strain rate of $1s^{-1}$, DRX of high temperature β phase occurs in TiB rich region, while DRV of high temperature β phase in TiB lean region.

When the strain rate is $0.01s^{-1}$, the size of the DRX grains around TiB increases as shown in yellow arrows in Fig. 4(b). Moreover, the color within the elongated β grains in TiB lean region is consistent, as shown in the yellow frames in in Fig. 4(b). It means a lower density of dislocations within the elongated β grains, which suggests the elongated β grains at the strain rate of $0.01s^{-1}$ are DRX grains. This is because the migration of grain boundaries reduces dislocation density. Thus, at the strain rate of $0.01s^{-1}$, the DRX grains around TiB grow to a certain extent along the direction to TiB rich region due to a limited effect of TiB, but the DRX grains grow rapidly along the direction to TiB lean region fastly due to the absence of the restriction of TiB.

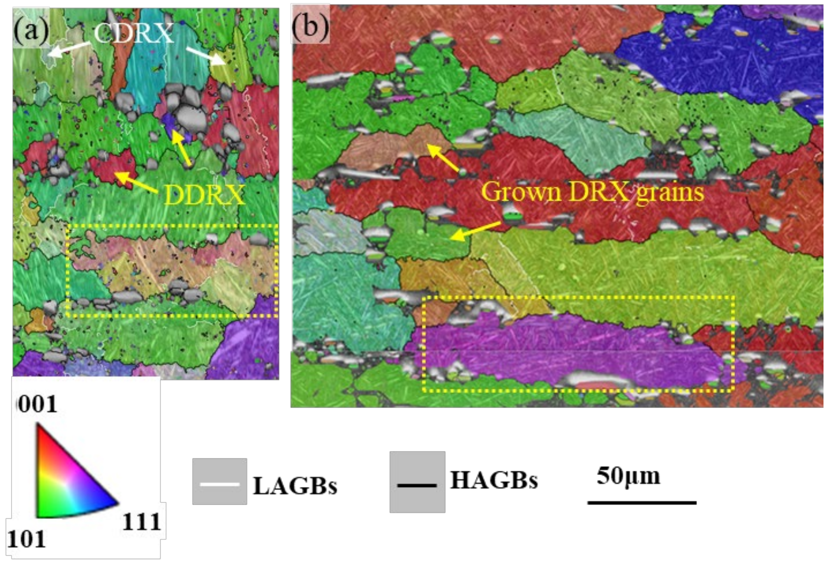


*Fig. 4. EBSD results of the reconstructed β phase of the deformed samples under different strain rates: (a) 1 $s^{-1}$; (b) 0.01 $s^{-1}$.*

The microstructure evolution of the composites during the deformation in β phase region with different strain rates is summarized, as shown in Fig. 5. The network distribution of TiB changes from circular to oval, accompanied by rotation of the single TiB, whose direction is gradually perpendicular to the compression direction. At the initial stage of deformation, a large number of dislocations are generated in the high temperature β phase. Due to TiB providing sufficient nucleation sites and higher storage energy, the nucleus of crystallization is preferentially formed around TiB. At a high strain rate (1$s^{-1}$), the nucleus of crystallization develops into refined DRX grains in TiB rich region. In TiB lean region, since the nucleus of crystallization has no sufficient time to grow to TiB lean region, the high temperature β phase is mainly dominated by the DRV process, and a certain density of dislocations are still distributed. At a low strain rate (0.01$s^{-1}$), the nucleus of crystallization has sufficient time to grow. In TiB rich region, it can only grow to a certain extent due to a restriction of TiB and elongate along the direction perpendicular to the compression direction, but it can grow fastly to TiB lean region due to the absence of the restriction of TiB. Thus, the dislocation density in TiB lean region is lower, and the corresponding high temperature β phase mainly occurs DRX process.

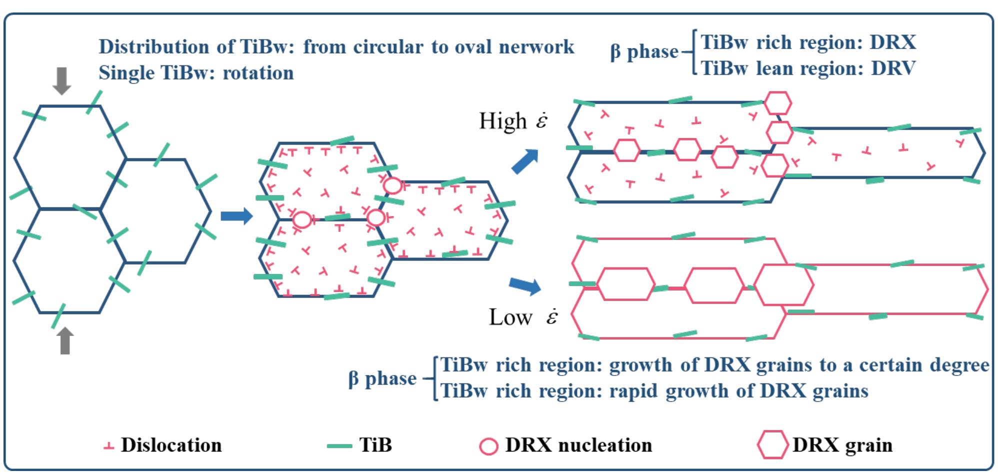


*Fig. 5. Schematic diagram of the microstructure evolution during the deformation in β phase region.*

## Conclusions

In summary, the as-sintered TiB/(TA15-Si) composites with network structure have been compressed with different strain rates in β phase region. The effects of network distributed TiB on dynamic recrystallization of high temperature β phase was investigated. The main conclusions were as follows:

1. After compression, the single TiB rotated, network structure evolved from circular to oval on longitudinal section, and high temperature β phase around TiB occurred dynamic recrystallization preferentially due to TiB supplying nucleation sites and higher storage energy. The mechanism of dynamic recrystallization included continuous and discontinuous dynamic recrystallization.
2. After compression, equiaxed grains of high temperature β phase were formed in TiB rich region due to dynamic recrystallization, while elongated grains were formed in TiB lean region. The dislocations density in elongated grains at strain rate of 1 $s^{-1}$ was higher than that at strain rate of 0.01 $s^{-1}$ due to their different envolution mechanism.
3. At the strain rate of 1 $s^{-1}$, high temperature β phase was dominated by dynamic recrystallization in TiB rich region and dynamic recovery in TiB lean region. At the strain rate of 0.01 $s^{-1}$, it was dominated by the growth of dynamic recrystallization grains with a certain extent in TiB rich region and rapid growth in TiB lean region.

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## Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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