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Microstructure stability of Ti_2AlN/Ti−48Al−2Cr−2Nb composite at 900 °C

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Abstract: Microstructure stability of in situ synthesized Ti_2AlN/Ti−48Al−2Cr−2Nb composite during aging at 900 °C was investigated by XRD, OM and TEM, and the unreinforced Ti−48Al−2Cr−2Nb alloy was also examined for comparison. The result showed that in the TiAl alloy, α_2 lamellae thinned and were broken down, and became discontinuous with increasing aging time. The decomposition of α_2 lamella to γ which was characterized by parallel decomposition and breakdown of α_2 lamellae led to the degradation of the lamellar structure. While in the composite, lamellar structure remained relatively stable even after aging at 900 °C for 100 h. No breakdown of α_2 lamellae except parallel decomposition and precipitation of fine nitride particles was observed. The better microstructural stability of the composite was mainly attributed to the precipitation of Ti_2AlN particles at the α_2/γ interface which played an important role in retarding the coarsening of lamellar microstructure in the matrix of composite.

Key words: TiAl composite; Ti_2AlN; microstructure stability; lamellar structure; precipitation

1 Introduction

Intermetallic γ-TiAl-based alloys have been investigated extensively for many years and are considered for higher temperature applications in aerospace and automotive industries due to their attractive properties such as low density, high modulus and good oxidation and creep resistance up to 800 °C [1−4]. However, poor ductility at room temperature and relatively low strength at high temperature limit their application. Many efforts have been made to overcome these shortcomings, and composite technology is regarded as an effective method to improve both toughness and creep resistance [5]. Among various kinds of reinforcements, ternary nitride Ti_2AlN has been used as an attractive candidate for reinforcements in TiAl-based composites due to its metallic and ceramic properties, such as low density, high elastic modulus, easy machinability and high-temperature oxidation resistance [6,7]. Moreover, Ti_2AlN is comparable to TiAl in both density and thermal expansion resistance. Introduction of Ti_2AlN can refine the as-cast microstructure and improve the mechanical properties of TiAl-based alloy remarkably. More attractive thing is that Ti_2AlN/TiAl composites show higher elevated temperature strength than unreinforced TiAl alloy at temperature as high as 900 °C and present great potential in high temperature application [8]. In view of the long-term use at high temperature, the thermal stability of the Ti_2AlN/TiAl composites is necessary to be evaluated. However, few works have focused on this issue up to now.

It is well known that the mechanical properties of composites are associated with their matrix closely. According to the researches, fully lamellar structure consisting of γ and α_2 lamellae provides excellent balanced properties of yield strength, fracture toughness and creep resistance than its counterparts in TiAl alloys [9,10]. However, the lamellar structure would be unstable even at temperature as low as 700 °C due to the reduction of both the chemical free energy and the interfacial energy so that the strength and creep resistance are reduced inevitably [11,12]. In order to improve the microstructure stability, refractory metals such as W, Hf and light elements such as Si and C are added, which causes concentration of refractory metal elements and formation of fine silicide and carbide particles at the α_2/γ lamellar interfaces and stabilizes the lamellar structure [13−16]. The addition of N element is also known as an effective method to enhance creep resistance by nitride (Ti_2AlN, Ti_3AlN) particle

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hardening [17,18]. Nonetheless, the formation of such precipitates was found to induce coarsening of lamellae in fully lamellar TiAl alloy containing N and lead to microstructure degradation [19]. This was mainly attributed to deprivation of Ti during formation of nitrides and left Al rich in the matrix. In the Ti_2AlN/TiAl composites, Ti_2AlN reinforcement was designed with stoichiometry and synthesized in advance. It could be expected that the influence of formation of nitrides on microstructure of TiAl matrix in Ti_2AlN/TiAl composites would be different from that in N-containing TiAl alloy during aging at intermediate temperatures.

Therefore, in this work, the microstructure changes of Ti_2AlN/TiAl composite prepared by an in-situ method, reactive arc-melting, were investigated during long-term aging treatment. For the composite, Ti−48Al−2Cr−2Nb (mole fraction, %), the classic fully lamellar TiAl alloy, was chosen as the matrix. In order to get a better understanding of the microstructure changes, the Ti−48Al−2Cr−2Nb alloy was also studied for comparison.

2 Experimental

The Ti_2AlN/Ti−48Al−2Cr−2Nb (Ti_2AlN/TiAl) composite was prepared by in-situ method of reactive arc-melting and the Ti−48Al−2Cr−2Nb (TiAl) alloy was produced by arc melting technique. To improve the chemical homogeneity, the two ingots were remelted four times. The microstructure of as-cast TiAl alloy shows coarse columnar dendrites and Ti_2AlN/TiAl composite exhibits a fine near lamellar structure in the matrix and rod-like reinforcement particles dispersed in the matrix. After that, the as-cast materials were subjected to a solution treatment at 1400 °C for 10 min and furnace cooling to room temperature so as to obtain fully lamellar microstructure. Then, the treated samples were aged at 900 °C for 50 h and 100 h in air, respectively. For each aging step, the samples were air cooled to room temperature.

The phase analysis was carried out on an X-ray diffractometer (DX−2700) using Cu K_α radiation for an angle range of 20°−90° (2θ), with a step of 0.02° (2θ). The microstructural features were observed by optical microscope (OLYMPUS GX71) and the samples for optical metallographic observation were prepared by standard mechanically techniques and etched using Kroll's reagent. Transmission electron microscopy (TEM) investigation was carried out on an FEI Tecnai F30 G^2 microscope operated at 200 kV. Thin foil specimens for TEM observation were first ground mechanically from both sides to about 50 μm and then thinned by standard double-jet electropolishing technique with an electrolyte consisting of 60% methanol, 35% butanol and 5% perchloric acid (volume fraction) operated at 35 V and −30 °C. For the evaluation of grain size and lamellar spacing of each sample, more than ten micrographs were analyzed and a mean linear intercept method was used to achieve the average value.

3 Results and discussion

3.1 As-annealed microstructures

Figure 1 shows the X-ray diffraction patterns of TiAl alloy and Ti_2AlN/TiAl composite solution-treated at 1400 °C for 10 min. According to the analysis results, γ-TiAl and α_2-Ti_3Al phases are identified in both materials. It should be noted that Ti_2AlN phase cannot be found in the composite. The main reasons are that, on one hand, a nominal Ti_2AlN content of 3% (volume fraction) is designed to introduce into TiAl matrix in this work, which is too little to be detected by XRD with limited precision; on the other hand, Ti_2AlN phase was found to be susceptible to decomposition above 1400 °C [20].

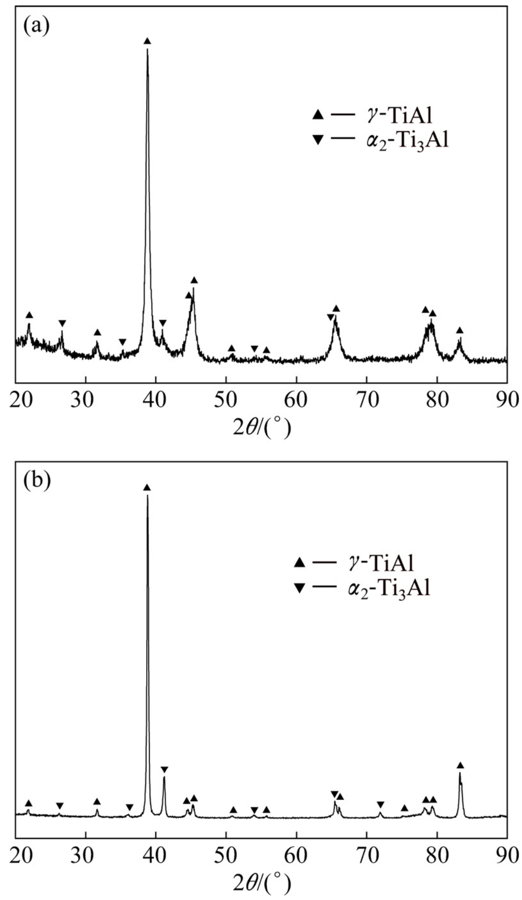

Fig. 1 XRD patterns of TiAl alloy (a) and Ti_2AlN/TiAl composite (b) solution-treated at 1400 °C for 10 min

The microstructures of the solution-treated materials are shown in Fig. 2. The TiAl alloy displays a coarse fully lamellar microstructure consisting of α_2+γ lamellar colonies with an average grain size of 1026 μm (Fig. 2(a)) and well interlocked grain boundaries which are caused by incursion of lamellae into the neighboring grains. By contrast, the Ti_2AlN/TiAl composite shows a refined microstructure with a grain size about 486 μm (Fig. 2(b)) and much finer and shorter lamellar incursions of the grain boundaries. The refined microstructure was inherited from the as-cast one, which was refined due to the introduction of Ti_2AlN particles. They acted as nucleation cores during melting and solidification.

3.2 Microstructure evolution during aging at 900 °C

The X-ray diffraction patterns of TiAl alloy and Ti_2AlN/TiAl composite aged at 900 °C for different time are shown in Fig. 3. Both γ and α_2 phases are detected in TiAl alloy after aging for 50 h, while only γ phase remains as time extends to 100 h (Fig. 3(a)). In the aged composite, α_2 phase is still remained even after 100 h aging, and some diffraction peaks of Ti_2AlN phase can be confirmed (Fig. 3(b)).

The microstructures of TiAl alloy aged at 900 °C for different time are presented in Fig. 4, and some noticeable changes can be observed. After aging for 50 h, the perfectly aligned lamellar structure is disturbed. Some α_2 lamellae underwent decomposition and transformed into γ phase. In consequence, thin γ lamellae laid in the remaining α_2 lamellae and fine alternating arrangement of γ and α_2 laths was formed, which is called "parallel decomposition". The generated α_2/γ interfaces are parallel to the original interfaces (Fig. 4(a)). In addition, boundary migration is observed. In Fig. 4(b), the α_2/γ interface is bulged and the α_2 lamella is even broken up, which leads to coalescence of the γ lamellae. The interface bulging was believed to only present at the 120° rotational fault interfaces [21]. The lamellae migration caused the coarsening of the γ lamellae. In these two cases, the α_2/γ lamellar interface keeps straight basically. Nevertheless, remarkable microstructure degradation appears at the interface, as displayed in Fig. 4(c). The interface between γ and α_2 lamellae is not straight anymore and the γ lamellae become coarse. Many dislocations are observed in the γ lamellae and at the α_2/γ interface even without applying stress. The generation of these dislocations is caused by loss of α_2 phase which transforms into γ phase. It is known that the Shockley partial associated with the transformation of α_2 interacts with the Frank partial associated with interface misorientation on the interface plane and gives rise to ordinary hard-mode dislocations within the γ matrix via reactions [22], i.e.,

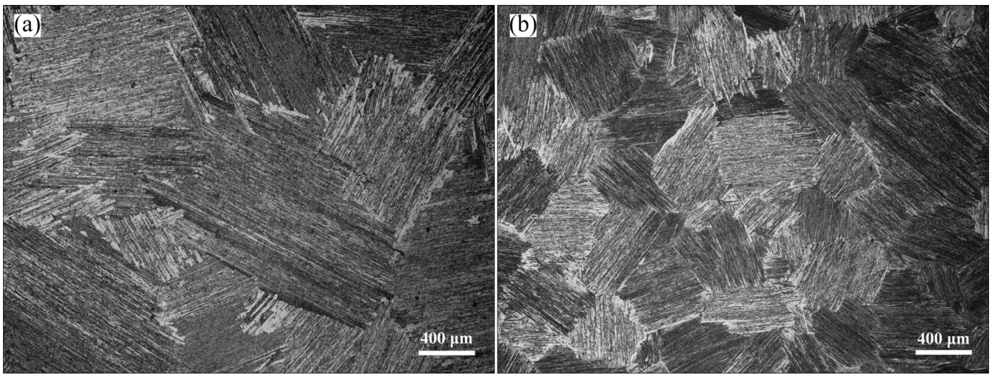

Fig. 2 Optical images showing microstructures of TiAl alloy (a) and Ti_2AlN/TiAl composite (b) solution-treated at 1400 °C for 10 min and furnace cooled

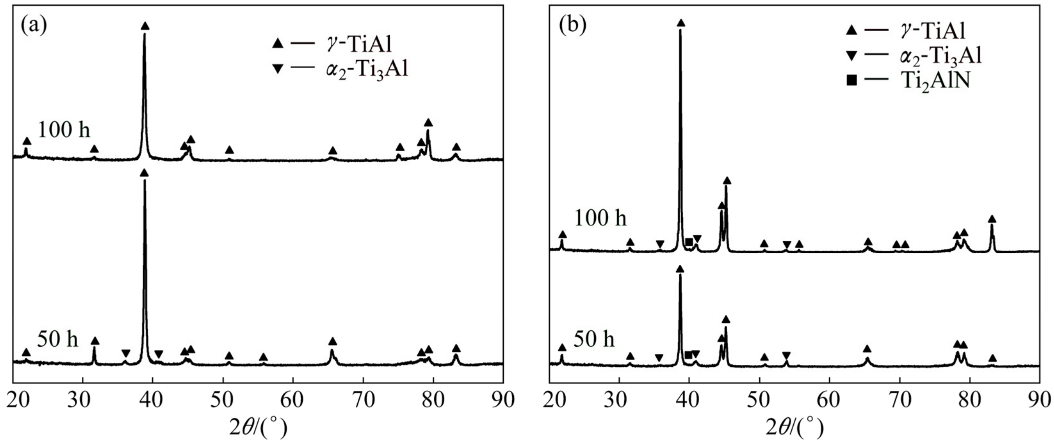

Fig. 3 X-ray diffraction patterns of TiAl alloy (a) and Ti_2AlN/TiAl composite (b) aged at 900 °C for different time

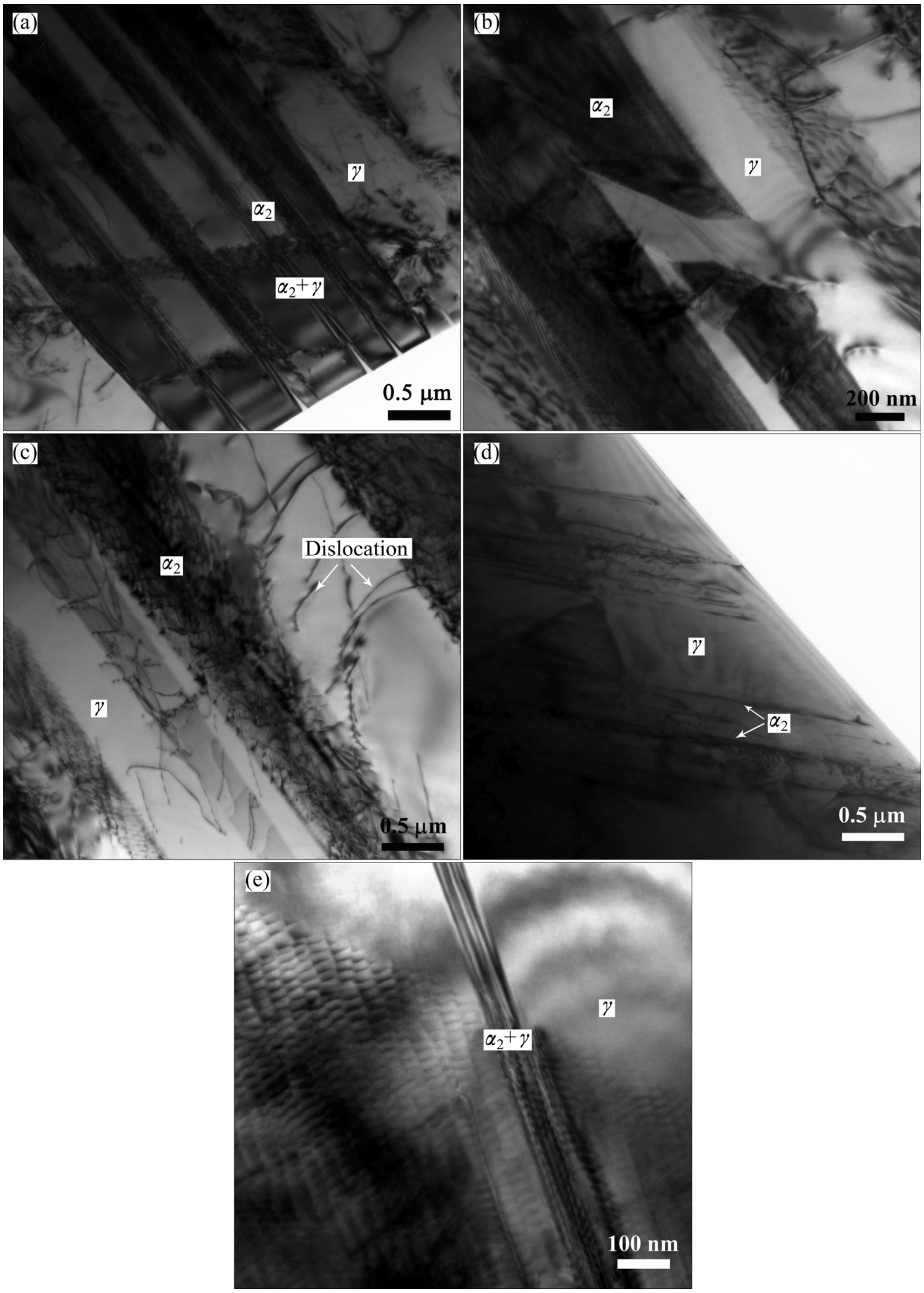

Fig. 4 TEM images showing parallel decomposition (a), breakdown of α_2 lamellae (b), dislocation in γ lamellae and at α_2/γ interface (c) after aging for 50 h, and discontinuous α_2 lamellae (d) and parallel decomposition of α_2 lamellae (e) after aging for 100 h in TiAl alloy

$$\frac{a}{6}[11\bar{2}]+\frac{a}{3}[111]\rightarrow\frac{a}{2}[110] \tag{1}$$

As aging time increases up to 100 h, α_2 lamella is found to be discontinuous (Fig. 4(d)). When the α_2 lath between two γ laths of the same variant completely disappears, the two γ laths coalesce to form a wider one. As indicated in Fig. 3(a), the content of α_2 phase decreases dramatically with aging time prolonging, suggesting that α_2 phase has undergone the transformation. In comparison with Fig. 4(c), the dislocation density decreases. This might be because prolonged aging leads to annihilation of matrix dislocations or their arrangement into low energy configurations, thus reducing the net mobile density. Further observation shows that the α_2 lamella is decomposed by parallel decomposition and fine γ and α_2 lamellae are formed (Fig. 4(e)).

It is well known that during aging α_2 phase will transform into γ phase due to the non-equilibrium phase constitution, which provides one of the driving forces to reach equilibrium conditions, thus results in structural changes upon exposure at elevated temperatures [10,23]. As revealed in this work, the α_2 lamella is decomposed by the phase transformation of $\alpha_2 \rightarrow \gamma$ during aging, and two modes are involved. One is characterized by parallel decomposition of coarse α_2 lamellae into fine γ and α_2 lamellae, which leads to the microstructure degradation such as thinning of α_2 lamellae and coarsening of neighboring lamellae. The other is characterized by breakdown of α_2 lamella occurred by nucleation of all possible orientations of γ (e.g., twinned or rotated by 120°) within the α_2 phase and by growth of the original lamellae from either side of the α_2 phase to produce regions where no α_2 phase remains [24]. Along with the decomposition processes in TiAl alloys, α_2 phase is dissolved and thus results in disappearance of α_2 lamella and coarsening of the γ lamella. The coarsening of the lamellar structure will degrade the creep properties by increasing the creep strain rates. In addition, the density of α_2/γ lamellar boundaries is reduced due to the dissolution of α_2 lamella, which leads to the reduction in the channeling stress and the increase in the mean free path of mobile dislocations, then associated increase in creep rates [12].

In the Ti_2AlN/TiAl composite, slight parallel decomposition of α_2 lamella can be observed after aging for 50 h, as shown in Fig. 5(a). Thin α_2 laths are separated by ultrathin γ laths. The white rod-like precipitates with an average size of 0.32 μm in length and 0.14 μm in width are assumed to be Ti_2AlN phase. These precipitates tend to stay at α_2/γ interfaces and their long-axis is parallel to the parent α_2 lamella, suggesting that the precipitates may nucleate at α_2/γ interfaces. Although parallel decomposition and precipitation occur, the lamellar interface is basically kept in a straight way. The aged composite exhibits finer lamellar structure with an average lamellar spacing of 0.16 μm than the aged TiAl alloy with 0.84 μm. For longer aging time of 100 h (Fig. 5(b)), the parallel decomposition of α_2 into fine α_2 and γ lamellae becomes more common, whereas more α_2 laths maintain a relative stability. It is interesting to note that the short rod-like Ti_2AlN particle precipitated at the α_2/γ interface is developed into a slender one with a size of 1.37 μm in length and 0.06 μm in width and its growth direction is parallel to the lamellar interface. Also, many nano-sized precipitates are found at the interface.

Compared with the counterpart of TiAl alloy, the composite possesses better lamellar structure stability during aging at 900 °C for different time. No breakdown of α_2 lamellae has been found except for some parallel decomposition and precipitation of Ti_2AlN. The Ti_2AlN particles are generally adjacent to α_2 lamellae, indicating that the decomposition of α_2 phase and the nucleation of the Ti_2AlN phase are related. The TEM observations indicate that secondary fine Ti_2AlN particles with nano-size dispersively precipitate at primary α_2/γ interface in the aged Ti_2AlN/TiAl composite. The process can be illustrated as $\alpha_2 \rightarrow \gamma + Ti_2AlN$. Thus, two types of phase transformation get involved in the process of α_2 decomposition, $\alpha_2 \rightarrow \gamma$ and $\alpha_2 \rightarrow \gamma + Ti_2AlN$.

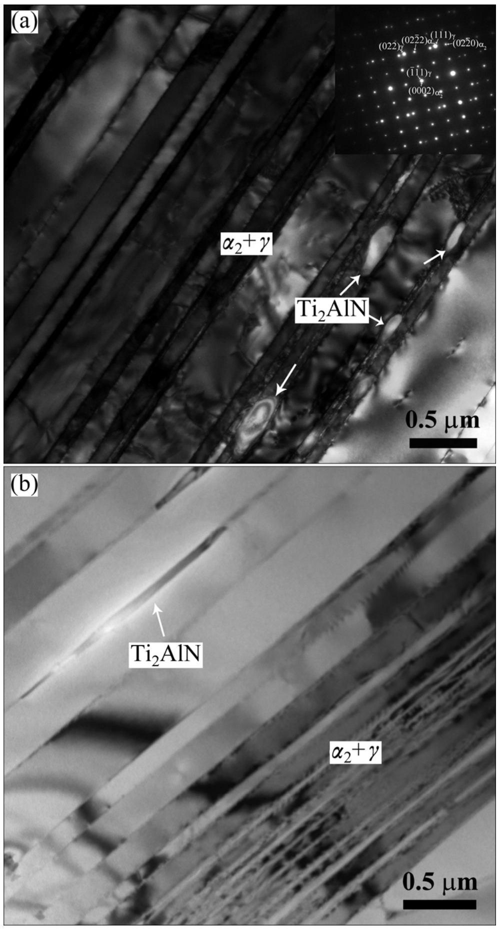

Fig. 5 TEM images showing lamellar microstructures of Ti_2AlN/TiAl composite aged at 900 °C for 50 h (a) and 100 h (b)

3.3 Precipitation in aged composite

According to TEM examination, many precipitates with different morphologies are revealed in the aged Ti_2AlN/TiAl composite, as shown in Fig. 6. The rod-like precipitate forms at the α_2/γ interface after aging at 900 °C for 50 h (Fig. 6(a)). According to the electron diffraction analysis, the interfacial precipitate with an ordered hexagonal structure is identified as Ti_2AlN phase, which lies parallel to the lamellae. In addition, another nano-sized precipitate is found to distribute continuously along lamellar interfaces in the composite (Fig. 6(b)). The corresponding selected area electron diffraction

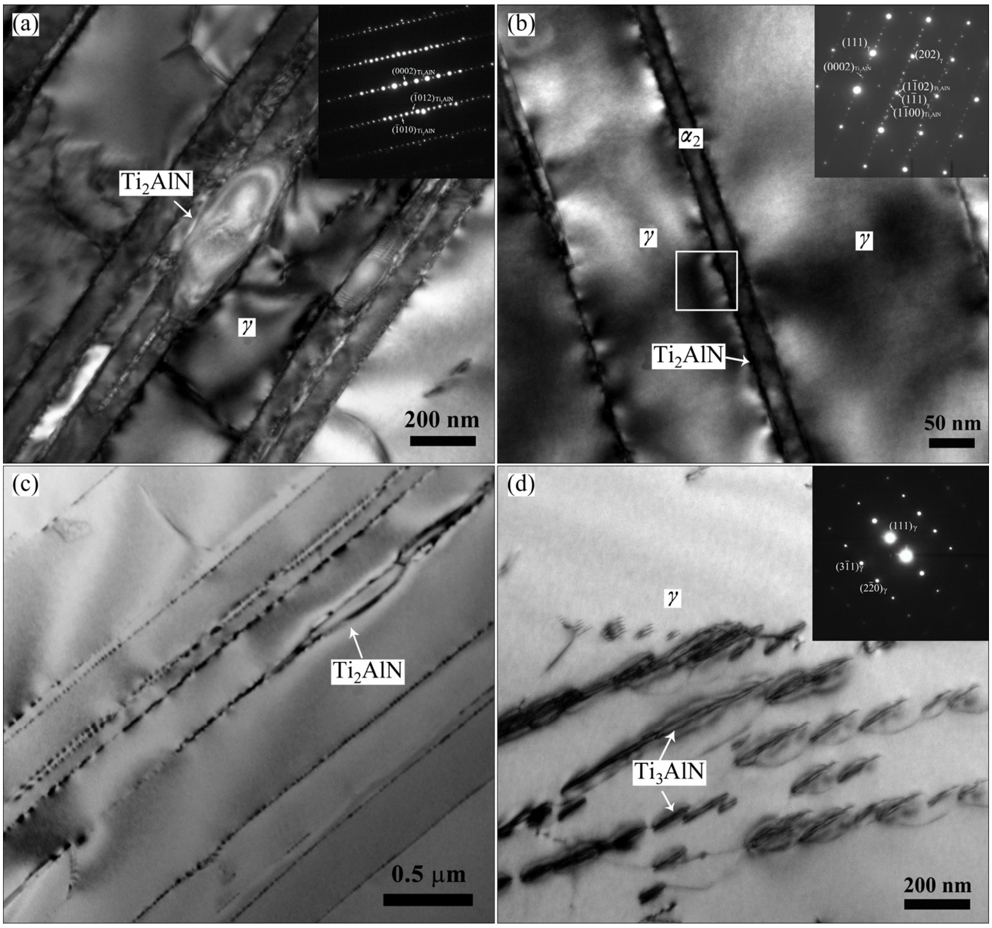

Fig. 6 TEM images showing Ti_2AlN precipitates at α_2/γ interfaces (a, b, c) and Ti_3AlN precipitates in γ matrix (d)

pattern clearly illustrates that the nano-sized precipitate is Ti_2AlN phase with crystallographic orientation relationship of $(111)_\gamma//(0001)_{Ti_2AlN}$ and $[10\bar{1}]_\gamma//[11\bar{2}0]_{Ti_2AlN}$. As the aging time increases to 100 h, the rod-like precipitate becomes elongated and its growth orientation keeps the same relationship with the lamellae, and the nano-sized precipitate tends to arrange in line (Fig. 6(c)). The other needle-like precipitate with low content is found to arrange along a particular direction in equiaxed γ matrix (Fig. 6(d)). This kind of precipitate prefers forming at the dislocations. By electron diffraction analysis, it is identified as Ti_3AlN phase. The growing axis of the needle-like precipitates is parallel to [001] direction of the matrix.

The formation and morphology of different types of precipitates are considered to be closely related to crystal structure, misfit, structure of the interphase boundary between precipitate and matrix and stress applied during aging treatment [25,26]. Ti_3AlN precipitate shows preferential orientation in [001] direction of the γ matrix due to minor lattice misfit between them in this direction [27]. Therefore, it often forms in γ matrix. As for Ti_2AlN precipitates, they prefer presenting along α_2/γ lamellar interfaces, rather than in the γ lamella. This may be associated with the decomposition of α_2 phase. During in-situ synthesis of Ti_2AlN/TiAl composite, a trace of nitrogen was inevitably dissolved into the TiAl matrix. Moreover, the in-situ Ti_2AlN reinforcements tended to be decomposed at 1400 °C and additional nitrogen was introduced. The two factors caused excess concentration of the element in the TiAl matrix, especially in α_2 phase. It is known that nitrogen has a higher solubility in α_2 phase than in γ phase [28]. During aging at 900 °C, nitrogen atom is likely to be segregated at α_2/γ interfaces because α_2 phase is supposed to approach an equilibrium condition. Thus, nitride precipitates would nucleate at the α_2/γ interfaces and subsequently grow along the interfaces. Although decomposition of α_2 phase occurs, the composite contains certain amount of α_2 phase and the $\alpha_2+\gamma$ lamellar structure is still remained even after aging for 100 h, suggesting that the dissolution of α_2 phase is retarded by the precipitation processes in the composite. This could be attributed to the fact that nitrogen atoms segregated at α_2/γ interfaces may lead to the decrease of the number of faults in the lamellar structure. The density of faults present in the lamellar structure influences the rate of mass transport associated with a unit volume of the lamellar structure [29].

Meanwhile, nitrides precipitated in lamellar region would retard initial critical processes that lead to instability.

It is known that stabilized microstructures with precipitates decorated on or parallel to the lamellar interfaces are identified to hold the promise for high temperature applications [22]. In the Ti_2AlN/TiAl composite, formation of nitride precipitates at the α_2/γ interfaces is expected to effectively block the emission and motion of dislocations at and along the lamellar interfaces and is beneficial to reducing creep rates.

4 Conclusions

1) Fully lamellar microstructure consisting of γ and α_2 phases was obtained for TiAl alloy and Ti_2AlN/TiAl composite after solution-treated at 1400 °C for 10 min. Aging at 900 °C resulted in noticeable microstructure changes for TiAl alloy. Parallel decomposition and breakdown of α_2 lamellae were introduced. As aging time increased, α_2 lamellae became discontinuous and γ coalesced to form a wider lath. The α_2 lamella was decomposed by the phase transformation from α_2 to γ.

2) The Ti_2AlN/TiAl composite displayed a relatively stable lamellar microstructure during aging process. Parallel decomposition and nitride precipitation were the two major decomposition processes of the α_2 lamellae. The former was characterized by phase transformation of α_2 to γ and became more common with increasing aging time, while the latter was represented by precipitation of rod-like or nano-sized Ti_2AlN particles at α_2/γ interface, which were elongated or arranged in line at the interface for longer aging time. The need-like Ti_3AlN particles mainly formed in the equiaxed γ matrix. The precipitation of fine Ti_2AlN particles at α_2/γ interface would retard the degradation of lamellar structure of Ti_2AlN/TiAl composite and was beneficial to microstructure stability of the composite.

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Ti_2AlN/Ti−48Al−2Cr−2Nb 复合材料在 900 °C 的组织稳定性

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摘 要：采用 XRD、OM 和 TEM 等方法对原位合成 Ti_2AlN/Ti−48Al−2Cr−2Nb 复合材料在 900 °C 时效过程中的组织稳定性进行研究，并与未增强的 Ti−48Al−2Cr−2Nb 合金进行对比分析。研究结果发现，在 TiAl 合金中，α_2 片层变细并发生破碎，且随着时效时间的延长变得不连续。以 α_2 板条平行分解和破碎为特征的 α_2 片层向 γ 的分解导致片层结构退化。而在复合材料中，在 900 °C 时效 100 h，片层结构保持相对稳定。除了片层平行分解和细小的氮化物沉淀外，没有发现 α_2 片层破碎。复合材料较好的组织稳定性主要与 α_2/γ 界面上的 Ti_2AlN 颗粒沉淀有关。Ti_2AlN 相的析出对延缓复合材料基体片层组织粗化具有重要作用。

关键词：TiAl 复合材料；Ti_2AlN；组织稳定性；片层结构；沉淀

(Edited by Xiang-qun LI)