



# Phase evolution and alloying mechanism of titanium aluminide nanoparticles



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## ABSTRACT

The evolution of phase composition of titanium aluminide nanoparticles synthesized by the flow-levitation method was systematically investigated by adjustment of the evaporating temperature of the mixed metallic droplet and the X-ray diffraction spectrum. Their alloying mechanism was analyzed according to the variation of phase composition.  $\epsilon$ (h, l)-TiAl<sub>3</sub>,  $\gamma$ -TiAl and  $\alpha_2$ -Ti<sub>3</sub>Al phases are gradually formed in TiAl alloy nanoparticles with the increasing of evaporating temperature of the mixed droplet. The alloying reaction is possible to perform between the small clusters of Ti and Al during the cooling process with high cooling rate. And the alloying mechanism can be explained based on the Gibbs free energy of alloying reaction of Ti and Al small clusters.

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## 1. Introduction

Nanocrystalline structures are known as the effective way to increase both strength and ductility of metal/alloy materials [1,2]. So it is a promising strategy to refine the coarse grains to ultrafined or nanostructured grains, in order to improve the low ductility at room temperature and to balance the comprehensive mechanical properties of high-temperature structural TiAl alloys for aerospace and automotive applications due to their high specific strength and stiffness, high strength retention and high creep resistance at elevated temperatures [3,4].

Many efforts are being performed by powder metallurgies of TiAl based alloy nanocrystalline powders which are prepared by various mechanical millings [3–11]. Moon et al. [5–7] produced nanocrystalline TiAl alloys with TiAl<sub>3</sub> phase by the reactive ball milling of elemental powders of Al and Ti in a hydrogen atmosphere and following hot extrusion or plasma activated sintering. During the initial milling the formation of TiH<sub>2</sub> was considered to not only reduce the particle size but also impede the grain growth during the heat treatment. Senkov et al. [8] prepared a dense nanocrystalline TiAl based alloy with a fully equiaxed gamma structure by the high-energy ball milling and following hot isostatic pressing of pre-alloyed gas-atomized powders.

Bhattacharya et al. [10] synthesized nanocrystalline powders by the high-energy milling. And the milling yield and contamination were investigated by various milling parameters including the addition of process control agents. Forouzanmehr et al. [11] synthesized the nanocrystalline TiAl intermetallic compounds with a grain size of about 50 nm by the mechanical alloying of Ti–50 at.%Al powder mixture and subsequent heat treatment. And a series of phase transformations including an amorphous phase with milling times were investigated. Yu et al. [3] synthesized an ultrafine grained TiAl based alloy with the grain size less than 400 nm by a combination of subzero temperature milling at temperatures less than –5 °C and following hot isostatic pressing at maximum temperature of 1000 °C. During the process of subzero temperature milling the excessive cold welding was suppressed and the effectiveness of mechanical milling was enhanced.

With the preparation of TiAl based alloy nanopowders, it is a better choice for ultrafined or nanocrystalline TiAl based alloys to densify directly nanopowders by powder metallurgies such as Spark Plasma Sintering (SPS). Liu et al. [12] synthesized TiAl alloy nanoparticles (NPs) with the average particle size less than 100 nm by the hydrogen plasma-metal reaction. There were various phase compositions in TiAl alloy NPs with the different Ti content. And the Ti content in these NPs was always less than that in the master alloys because of the higher evaporation rate of Al than Ti. Luo et al. [13] synthesized the TiAl binary alloy NPs with specific phase composition by the flow-levitation (F–L) method [14,15], including pure  $\alpha_2$ -Ti<sub>3</sub>Al phase. It was discovered that the formation

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appearance of phases during the preparation of NPs was different with that of micropowders of TiAl alloys [16,17]. So in this study, the evolution of phase composition and alloying mechanism of TiAl alloy NPs are systematically investigated by adjustment of technical parameters such as the evaporating temperature of the mixed metallic droplet in the F–L method.

## 2. Experimental details

Titanium aluminide NPs with the average diameter of less than 100 nm were synthesized by the F–L method after adding another same metal wire feeding unit [13]. Titanium and aluminum wires with the purity of 99.99 wt% were melted into the metallic liquid droplet in the high-intensity high-frequency electromagnetic field. Due to the continuous heating, mixed Ti and Al atom vapors were escaped from the surface of the droplet, which was levitated in this electromagnetic field because of the vortex action. The mixed atom vapors were quickly cooled due to their collision with the converse inert gas (Ar, et al.), and then formed monometallic/intermetallic clusters till to intermetallic NPs because of the further cooling of the inert gas. The evaporating temperature of the mixed metallic droplet was automatically adjusted only by the wire feeding rate under the other same technical parameters, at the same time the ratio of Ti and Al wires was fixed to  $Ti_{48}Al_{52}$  in atomic percent. Generally the evaporating temperature decreases with the decreasing of wire feeding rate after the dynamic equilibrium between the melting and evaporation of droplet. The phase composition of titanium aluminide NPs was adjusted by the varied evaporating temperatures.

Titanium aluminide NPs were annealed at 900–1000 °C for 5 h in the vacuum furnace with the base pressure of 0.5 MPa. The X-ray diffraction investigation of all NPs was performed on a DX2000 diffractometer with the Cu K $\alpha$  radiation at the angle range of 20–90°.

## 3. Results

The XRD spectra of titanium aluminide NPs synthesized at different evaporating temperatures are shown in Fig. 1a and b. Fig. 1a shows that when the evaporating temperature ranges from 2250 °C to 2400 °C, there are two phases ( $\gamma$ -TiAl and  $\alpha_2$ -Ti $_3$ Al) in the titanium aluminide NPs. And the relative phase composition of  $\gamma$ -TiAl and  $\alpha_2$ -Ti $_3$ Al is depending on the evaporating temperatures according to the relative intensity comparison of the strongest peak of two phases. At 2360 °C and 2400 °C, all diffraction peaks of  $\gamma$ -TiAl phase cannot be found from their XRD spectra, indicating forming pure  $\alpha_2$ -Ti $_3$ Al phase in the two kinds of titanium aluminide NPs. When the evaporating temperature decreases to 2330 °C,  $\gamma$ -TiAl phase is observed. With decreasing the evaporating temperature (2300 °C and 2250 °C), more  $\gamma$ -TiAl phase and less  $\alpha_2$ -Ti $_3$ Al phase have been formed in the NPs.

Fig. 1b shows that when the evaporating temperature further decreases from 2180 °C to 2120 °C, complex phases including  $\alpha_2$ -Ti $_3$ Al,  $\gamma$ -TiAl,  $\epsilon$ (h, l)-TiAl $_3$ , TiO $_2$  and Al $_2$ O $_3$  coexist in the titanium aluminide NPs.  $\alpha_2$ -Ti $_3$ Al phase (diffraction peak: 41.17°) and  $\gamma$ -TiAl phase (diffraction peaks: 45.55° and 65.49°) can still be detected at relatively higher temperature (2180 °C for  $\alpha_2$ -Ti $_3$ Al phase; 2180 °C and 2150 °C for  $\gamma$ -TiAl phase). Moreover, the relative phase composition of  $\gamma$ -TiAl phase slightly decreases with the decreasing evaporating temperature according to the tiny displacement of the overlapped strongest diffraction peak and the relative intensity of other peaks between  $\gamma$ -TiAl phase (65.49°) and  $\epsilon$ (h)-TiAl $_3$  phase (64.98°). It is worth noting that  $\gamma$ -TiAl phase cannot be observed at 2120 °C. Three strong diffraction peaks of  $\epsilon$ (l)-TiAl $_3$  phase (39.12°, 47.13° and 64.98°) and  $\epsilon$ (h)-TiAl $_3$  phase (39.12°, 46.81° and 84.02°) can be detected, suggesting that they form simultaneously within this evaporating temperature range. Moreover, at lower evaporating temperature, there are more  $\epsilon$ (h)-TiAl $_3$  phase with higher crystalline fraction according to the quantity and shape of diffraction peaks. The TiO $_2$  phase and Al $_2$ O $_3$  phase appear at 2120 °C, which maybe result from the oxidation of NPs with high surface activity during the sampling and measurement.

In order to check the characteristic of  $\gamma$ -TiAl and  $\alpha_2$ -Ti $_3$ Al phases, titanium aluminide NPs prepared at 2400 °C (pure phase – labeled PP) and 2330 °C (mixed phase – labeled MP) were annealed at 900 °C and 1000 °C respectively for 5 h. The XRD spectra in Fig. 2 shows that  $\alpha_2$ -Ti $_3$ Al phase transforms partly into  $\gamma$ -TiAl phase after annealing for both NPs, and the ratio of phase transformation increases with the elevating annealing temperature. The phase transformation probably occurs inside the nanoparticles

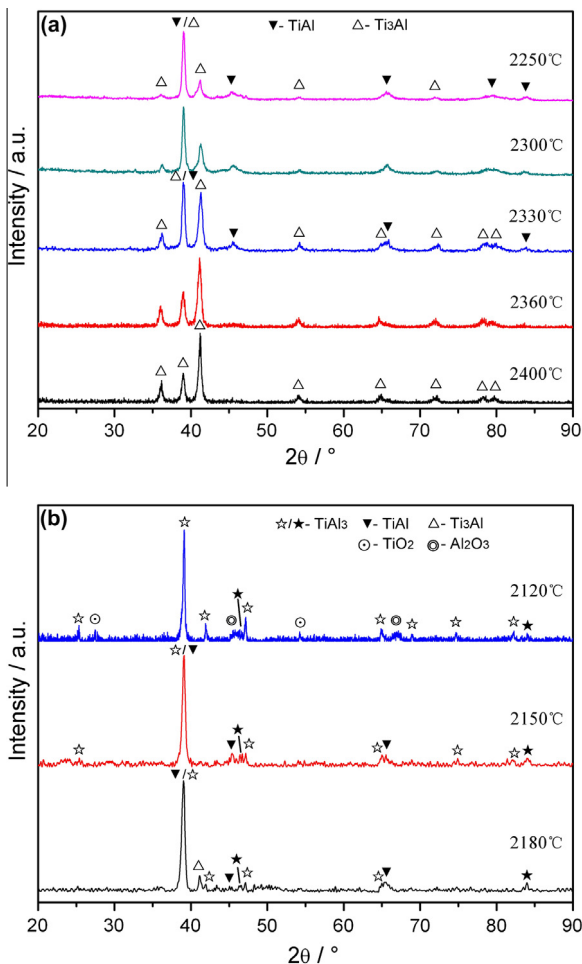


Fig. 1. XRD spectra of titanium aluminide NPs synthesized at various evaporating temperatures.

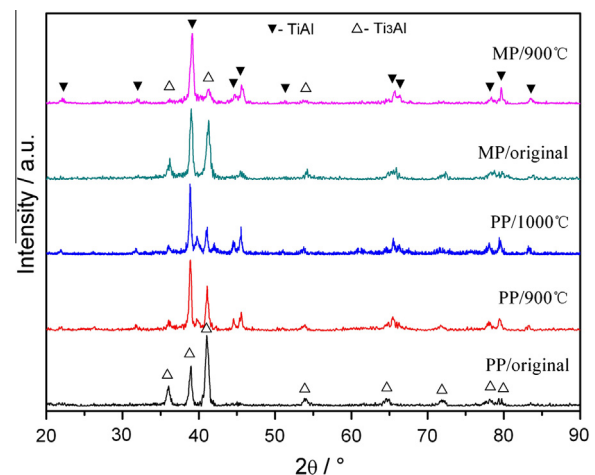


Fig. 2. XRD spectra of titanium aluminide NPs after annealing.

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