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Synthesis of γ-TiAl Based Alloy by Mechanical Alloying and Reactive Hot Isostatic Pressing

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Elemental Ti-48Al-2Cr-2Nb powders were mechanically alloyed (MA) for 8 h and 45 h. The MA powders were then consolidated by reactive hot isostatic pressing (HIP). The microstructure of the HIPed materials consisted of equiaxed γ-TiAl and α_2-Ti_3Al phases. During the high-temperature annealing of the HIPed materials, the α_2-Ti_3-Al phase transformed into a lamellar structure consisting of alternating laths of α_2-Ti_3Al and γ-TiAl. It is suggested that a high content of interstitial elements together with the microalloying elements of niobium and/or chromium in MA powders raises $\alpha/(\alpha + \gamma)$ transus to a higher temperature.

Keywords hot isostatic pressing, intermetallic compounds, TiAl

1. Introduction

In γ-based TiAl alloys, four different types of equilibrium microstructures including a dual structure (equiaxed γ and α_2 grains), a duplex structure (equiaxed γ grains + lamellar γ/α_2 grains), and near and fully lamellar $(\gamma + \alpha_2)$ structures have been generated. The duplex structure is beneficial for ductility but possesses a low fracture toughness as compared with the near and fully lamellar structures (Ref 1, 2). Of all microstructures, the fully lamellar structure has the best fracture toughness (Ref 3-5). This is because of the reinforcement effect produced by the lamellae (Ref 6). The dual structure has a good deformability and transforms easily into duplex or (near) fully lamellar structure during subsequent heat treatments (Ref 8). The aim of the present study is to synthesize γ-based TiAl alloy via mechanical alloying and reactive hot isostatic pressing (HIP) and to characterize the microstructure and annealing behavior of MA and HIP material.

2. Experimental Details

One hundred grams of elemental powder mixture with the nominal composition of Ti-48Al-2Cr-2Nb (at.%) was mechanically alloyed under an argon protective atmosphere for 8 or 45 h in a Fritsch Pulverisette 5-type planetary ball mill. Stearic acid, 1 wt%, was used as a process control agent. The actual ratio of titanium to aluminum in mechanically alloyed (MA) powders is approximately 1.07 to 1.08. Mechanically alloyed powders were then consolidated by reactive HIP under the pressure of 100 MPa at a temperature of ~1150 to 1200 °C for 3 h. Annealing of HIP materials was carried out for 1 to 3 h in a vacuum Instron furnace at temperatures of ~1350 to 1400 °C and ~1300 to 1350 °C. Back-scattering scanning electron microscopy (SEM) mode in a Philips XL 30 SEM and an Edwards energy dispersive x-ray spectrometer (EDS) attached to the microscope as well as a JEOL JEM-2010 analytical transmission microscope (JEOL, USA, Peabody, MA) (ATEM) operated at an acceleration voltage of 200 kV were used for the microstructural study of HIPed materials before and after annealing.

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3. Results

Eight hours of milling resulted in a macroscopically homogeneous lamellar Ti-Al composite microstructure in powder particles together with a small volume fraction of solid Ti(Al) solution. During HIP of the powder milled for 8 h, a microstructure consisting of γ-TiAl matrix and α_2-Ti_3Al agglomerates was formed (Fig. 1). Conversely, the milling for 45 h resulted in a good homogeneity of the material. Fine powder particles with amorphous microstructure were obtained. Only a very limited volume fraction of small α_2 agglomerates was observed in the material milled for 45 h and HIPed, as shown in Fig. 2.

The annealing of the material milled for 8 h at a temperature of ~1350 to 1400 °C for 1 h results in the transformation of most prior α_2 agglomerates into fully lamellar structures. As shown in Fig. 3(a), no significant coarsening of prior α_2 agglomerates was observed but the spheroidization of coarse α_2 agglomerates did occur. Figure 3(b) shows a closer study of a fully transformed $\alpha_2$agglomerate that consists of four lamellar grains except for several small coalescent α_2 particles located at the edge and for some quite finely scattered α_2 particles around the lamellar agglomerate. Very fine lamellar spacings were commonly observed although they varied in each lamellar grain and from grain to grain. The BEI image of area I in Fig. 3(b) at higher magnification (Fig. 3c) showed nanoscale interspacings between γ laths (as depicted by dark contrast) and α_2 laths (depicted by light contrast). Also, an unfinished γ phase precipitation process in an individual α_2 lath was revealed, as indicated by the arrow in Fig. 3(c). The results show that the precipitation of γ lath starts from one point. Then the lath grows up along the length direction instead of forming the whole length of γ lath across prior α_2 grain by solute partitioning out of an α_2 matrix at the same time. From the point of view of the free energy, the former formation mechanism should be more reasonable. The lamellar structures consisting of alternating layers of γ and α_2 laths have the crystallographic orientation relationship of $(11\bar{1})_\gamma//(0001)_{\alpha_2}$ and $[011]_\alpha//[2\bar{1}\bar{1}0]_{\alpha_2}$, as shown in Fig. 4. This suggests that the close-packed $\{111\}_\gamma$ planes are nucleating and grow from the $(0001)_{\alpha_2}$ basal planes. The γ laths in lamellar structure are generally related to the formation of $\{111\}_\gamma$ twins.

As shown in Fig. 5, the microstructure of the material milled for 45 h consisted of equiaxed submicron grains of γ-TiAl. After annealing for 3 h at a temperature of ~1300 to 1350 °C, some grain growth can be seen in Fig. 6. No lamellar reactions occurred during annealing.

4. Discussion

As shown in Fig. 3, the small volume fraction of γ/α_2 lamellar structure in the HIPed materials after annealing at the used high temperature definitely excludes the formation of a fully (or near) lamellar structure because the lamellar structure seems to form only in prior α_2 agglomerates. However, the formation of a fully lamellar structure was reported during HIP of powder mixture with the nominal composition of Ti-48Al-2Mn-2Nb and subsequent annealing for 1 h at 1400 °C (Ref 9). Further, according to Murray's titanium-aluminum phase diagram (Ref 10) a significant part of γ matrix should transform into α phase when the annealing temperature is raised to that high value. In other words, lamellar structures originating from the formation of γ laths from the dominant α (or α_2) phase during cooling should, after annealing at the used temperature, constitute a major part of the microstructure of HIPed materials instead of equiaxed γ grains. A chemical analysis revealed 0.032 N, 0.50 O, 0.62 C, and 0.24 Fe in the powder milled for 8 h and 0.038 N, 0.57 O, 0.70 C, and 0.20 Fe (wt%) in the powder milled for 45 h, respectively. It is therefore suggested that the high content of interstitial elements in the studied materials significantly influences the mechanism of lamellae formation. According to Ref 11, the nucleation of γ laths from an α phase occurs via the movement of $(a/6)[1\bar{1}00]$ Shockley partial dislocations, which introduces the stacking faults at the lamellar interface. Similarly, the nucleation of α_2 laths originates from the stacking faults in γ, which have the same stacking sequence as α_2. The growth of α_2 laths is occurring on $\{111\}_\gamma$ slip planes by the movement of $\frac{1}{6}$ [112] partial dislocations and compositional adjustment (Ref 12). Therefore, the nucleation of either α_2 or γ laths always involves the movement of Shockley partial dislocations. It has been reported in Ref 13 that γ phase has a very limited solubility of interstitial elements. The maximum solubility of oxygen in γ is approximately 250 at.% ppm for the temperatures ranging from 1000 to 1300 °C (Ref 13). However, there are no difficulties for the materials that have a higher content of interstitial elements, for example, 550 wt% ppm of oxygen (Ref 1), to obtain fully lamellar structure upon annealing at high temperature. In other words, a very limited content of interstitial elements in γ solution cannot effectively prevent the movement of dislocations and thus the transformation of γ into α at high temperature during the heating leg of annealing. Con-

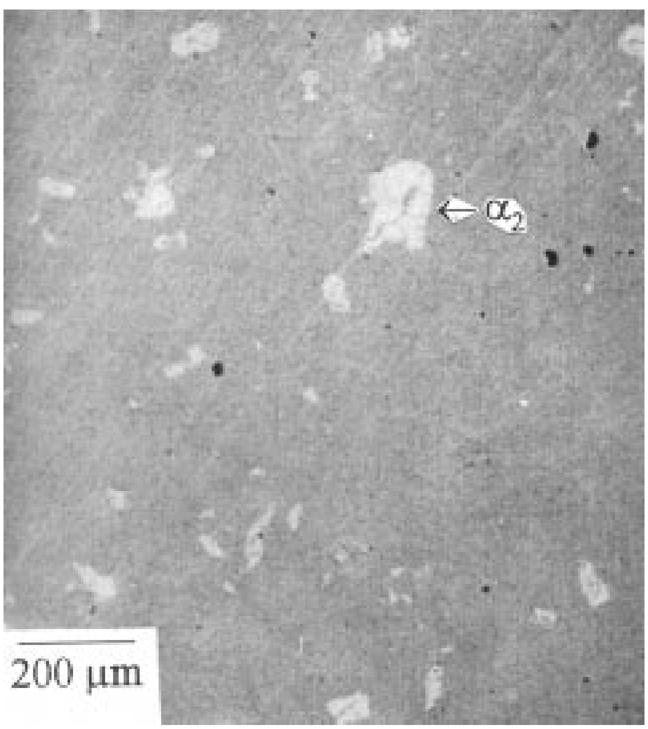

Fig. 1 Microstructure of the material milled for 8 h and hot isostatically pressed (scanning electron microscopy BEI image): γ-TiAl matrix and α_2-Ti_3Al agglomerates

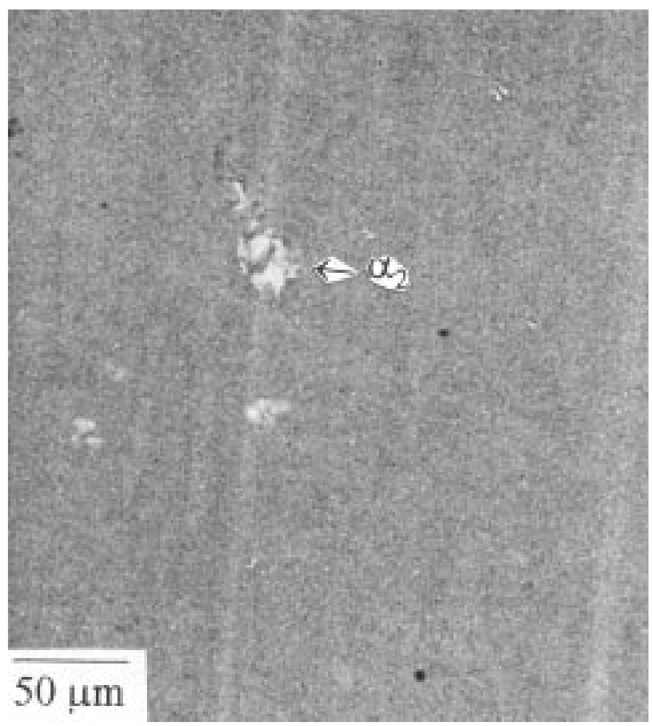

Fig. 2 Microstructure of the material milled for 45 h and hot isostatically pressed (scanning electron microscopy BEI image): γ-TiAl matrix and a very limited volume fraction of α_2-Ti_3Al agglomerates

versely, the high density of the fine carbonitrides dispersed in γ matrix in the HIPed materials in the present case (Ref 14) could stabilize the γ phase. As a result, the γ matrix in HIPed materials did not transform sufficiently into α phase during the annealing and thus could not form lamellar structure during the subsequent cooling. Conversely, a high content of interstitial elements instead of the carbonitrides in α phase possibly stabilizes the α phase and therefore prevents the formation of γ laths. The unfinished transformation of α agglomerates shown in Fig. 3d shows this lack of formation.

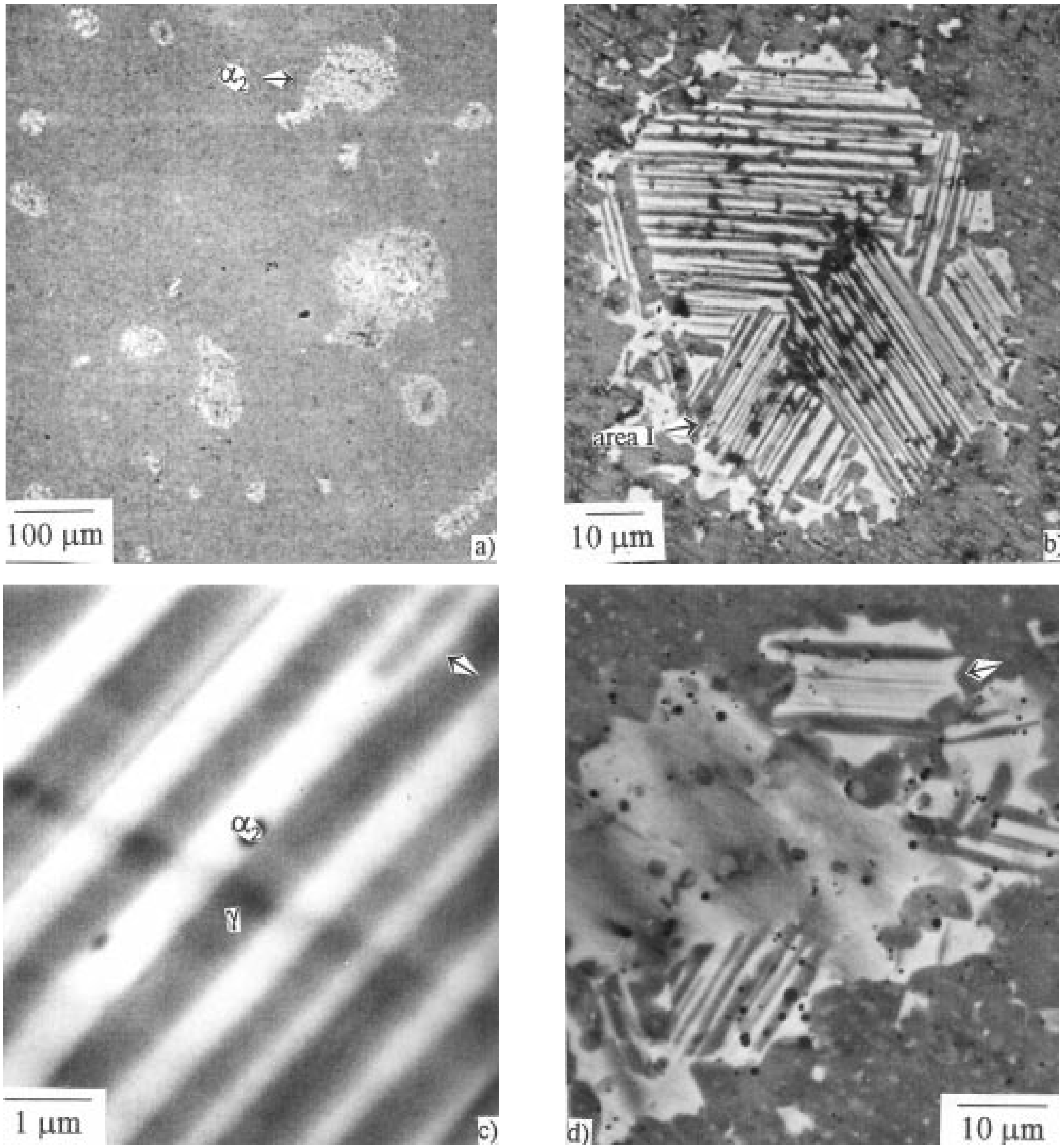

Fig. 3 Scanning electronmicroscopy BEI micrographs of the structure of the material milled for 8 h after heat treatment for 1 h at the temperature of ~1350 to 1400 °C. (a) Spheroidization of α_2 agglomerates. (b) A lamellar region formed from a previous α_2 agglomerate during the heat treatment. A typical lamellar structure in the prior α_2 agglomerate consists of four lamellae grains. (c) Nanoscale interspacings of γ laths (dark contrast) and α_2 laths (light contrast) in area I of Fig. 3(b). A continuing process of γ lath precipitation from α_2 phase is clearly revealed as indicated by the arrow. (d) A partially transformed α_2 agglomerate

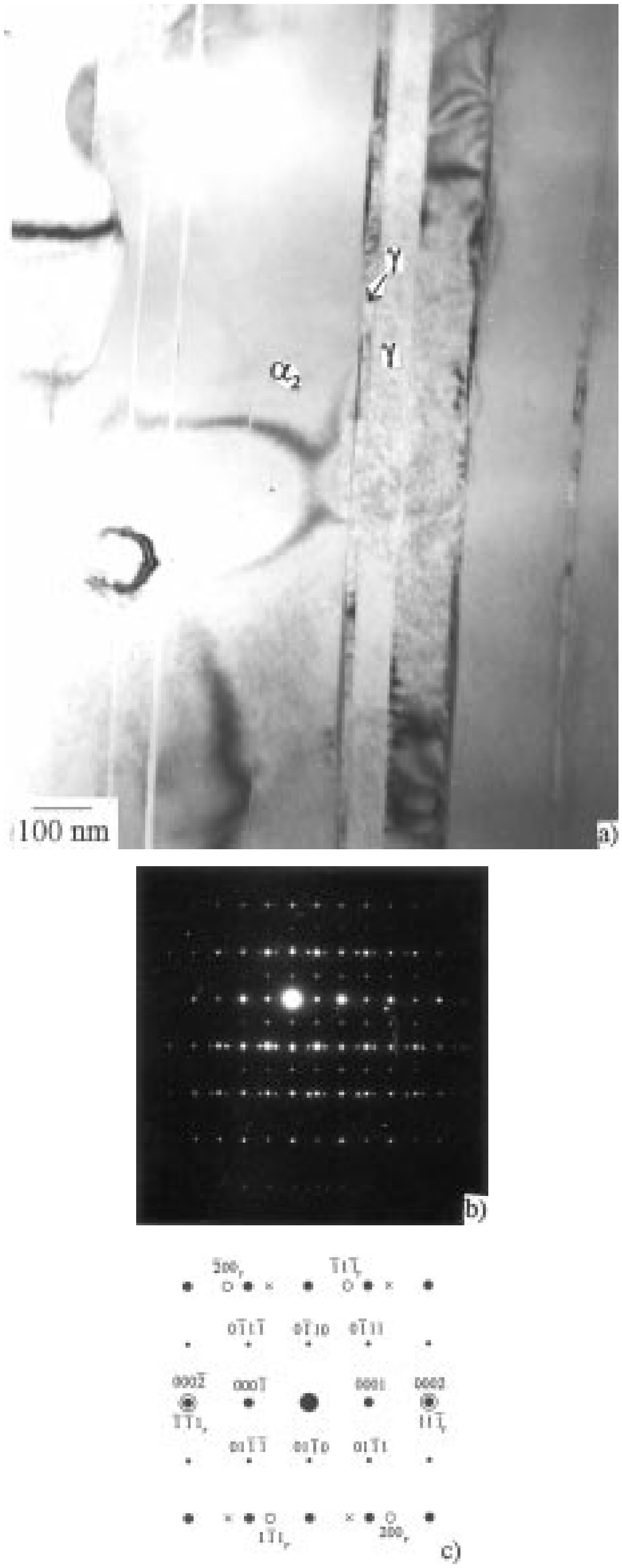

Fig. 4 A transmission electron microscopy micrograph of the lamellar structure of the material milled for 8 h after annealing for 1 h at the temperature of ~1350 to 1400 °C. (a) Selected-area diffraction pattern taken from the lamellar structure. (b) Schematic representation. (c) Crystallographic orientation relationships between twin-related γ and α_2 laths: $(1\bar{1}\bar{1})_\gamma//(0001)_{\alpha 2}$ and $[011]_\gamma//[2\bar{1}\bar{1}0]_{\alpha 2}$

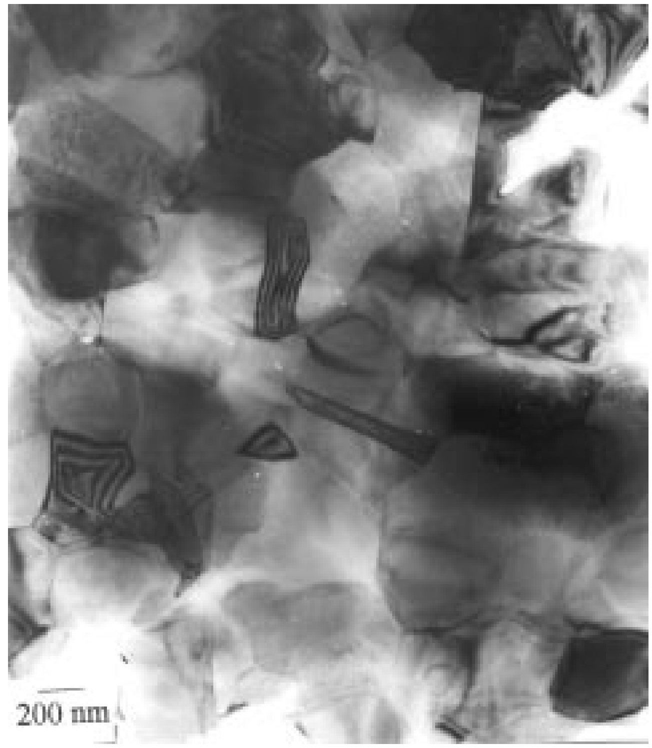

Fig. 5 Transmission electron microscopy micrograph of the material milled for 45 h and hot isostatically pressed. The microstructure consists of an equiaxed submicron microstructure of γ-TiAl phase.

Fig. 6 Transmission electron microscopy micrograph of the material milled for 45 h and hot isostatically pressed after annealing for 3 h at a temperature of ~1300 to 1350 °C. Slight grain growth of the γ phase occurred during annealing.

A question now arises whether the γ laths nucleate and grow from α phase before ordering. It was reported in Ref 9 that the driving force for lamellar structure formation is the supersaturation of aluminum in α phase. For Ti-50Al alloy, the formation of γ laths from α phase occurs above 1370 °C and the ratio (aluminum/titanium) in α phase is larger than 47 to 53 (Ref 1). In this case, the saturation degree of aluminum in α phase at the annealing temperature (aluminum to titanium = 45 to 48) is sufficient for the formation of γ laths from the α phase. In other words, type I lamellae formation (Ref 1) could occur during the cooling of supersaturated α phase into ($\alpha + \gamma$) field. It should be pointed out that the process of type I or II lamellae formation (Ref 1) becomes more complicated when taking into account the effect of microalloying additions because the microalloying additions could shift the $\alpha/(\alpha + \gamma)/\gamma$ transus lines. It has been reported that a 2% Nb addition into Ti-48Al alloy shifts the $(\alpha + \gamma)/\gamma$ transus line toward the aluminum-lean direction and the $\alpha/(\alpha + \gamma)$ transus line toward the aluminum rich side (Ref 15).

5. Conclusions

During mechanical alloying and subsequent HIP of Ti-48Al-2Cr-2Nb powder mixture, a microstructure consisting of equiaxed γ-TiAl and α_2-Ti_3Al phases was formed in the material milled for 8 h. Conversely, an almost single γ-TiAl phase was formed in the material milled for 45 h. The high-temperature annealing of the HIPed material milled for 8 h resulted in the final duplex microstructure consisting of γ-TiAl matrix and lamellar ($\gamma + \alpha_2$) grains. The γ matrix was rather stable and did not join the lamellar reaction. It is suggested that the high density of fine and stable carbonitrides (which resulted from the high content of interstitial elements in the MA powders and was homogeneously distributed in the γ matrix) together with the microalloying elements of niobium and/or chromium stabilized the γ matrix.

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