



Effect of Cr and Nb on the phase transformation and pore formation of Ti–Al base alloys



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ARTICLE INFO

Article history:

Received 5 July 2016

Received in revised form

9 January 2017

Accepted 1 March 2017

Available online 2 March 2017

Keywords:

Titanium aluminides

Elemental powder metallurgy

Kirkendall effect

Diffusion

ABSTRACT

The phase transformation and its relation to the porosity of Ti–Al base alloys are investigated in order to understand the effect of composition on the pore formation during sintering at low and high temperatures. Ti–Al base alloys with nominal compositions of $Ti_{52}Al_{48}$, $Ti_{50}Al_{48}Cr_2$, $Ti_{50}Al_{48}Nb_2$ and $Ti_{48}Al_{48}Cr_2Nb_2$ are prepared by the conventional elemental powder metallurgy route. The sintering of mixed powders was carried out at 600° C (low-temperature sintering below the Al melting point) and 1200° C (high-temperature sintering). Al-rich intermetallic compound $TiAl_3$ was primarily formed during low-temperature sintering while Ti-rich intermetallic compound Ti_3Al was formed during high-temperature sintering. The phase transformations increase the overall porosity and pore size of alloys to a similar extent regardless of the alloy composition during the low-temperature sintering. However, large differences in the pore size and pore volume distribution occur depending on the composition of Ti–Al alloys during high-temperature sintering. Chromium and niobium do not affect the formation of Kirkendall voids and thus show a similar void formation behavior at 600° C, but affect the void agglomeration behavior to a different extent due to the different diffusion activities at 1200° C.

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1. Introduction

Porous metals have attracted strong industrial and research interests because of their high specific stiffness and specific strength, excellent energy absorption, and damping capacities. However, the practical application of conventional metals and alloys for high-temperature and corrosive environments is restricted due to limited oxidation and corrosion resistance. Porous intermetallics with metallic and covalent bonding can be good candidate materials for high-temperature applications. Ti–Al alloys are well-known to form intermetallic phases with excellent mechanical properties and good oxidation resistance up to 700° C [1]. Titanium aluminides have long been investigated for high temperature structural applications, and in the last decade, porous titanium aluminides have also been studied for filtration applications.

Ti–Al porous alloys can be made by the conventional elemental powder metallurgy (EPM) process based on the Kirkendall effect. The Kirkendall voids, caused by substantial discrepancies in

diffusion-rate of elements consisting of the diffusion couple, can lead to a porous structure [2]. Recent studies have claimed indeed that the Kirkendall effect is the most important phenomenon for pore formation in Ti–Al base alloys [3–5]. In Ti–Al alloys, the diffusion rate of Al atoms is much higher than that of Ti atoms during solid state diffusion at low temperatures below the melting point of Al [6]. The Kirkendall pores formed at low temperatures are normally much smaller in size than original powders [4]. However, the porosity level reaching up to 20 to 40 vol% is often reported for EPM-processed Ti–Al base alloys, which cannot be rationalized by the Kirkendall effect alone.

Several researches have been carried out with Ti–Al alloys [4,5,7–9], to study the pore formation behavior and the effect of sintering temperature on pore size and its distribution. It is obvious that atomic diffusion in intermetallic compounds is quite complex due to the restrictions imposed by the long range ordering characteristics of the intermetallic compounds. He et al. [10] fabricated TiAl porous alloys through the Kirkendall effect via EPM, and the same group investigated effects of Al composition on pore morphology in TiAl alloys [6]. Wang et al. [7] studied the effect of Nb on pore structure and tensile properties of Ti–48Al alloy. The pore structure and size were studied using microstructural techniques, but the effect of Nb content on mean pore size was not discussed in detail. Liang et al. [5] proposed a reaction mechanism

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in Nb-containing TiAl alloys with an increase in temperature, but the local or overall effect of alloying addition was not discussed.

In spite of the fact that several researches have been reported on porous TiAl alloys, the pore formation mechanisms in EPM-processed TiAl alloys are poorly understood and await clarifications. In addition, previous literature dealing with the phase transformation and pore formation mechanism at different temperatures has not paid enough attention to any local or overall effect of alloying additions. Both Nb and Cr are known as important alloying elements in bulk TiAl alloys, but their detailed effects have been focused mainly on bulk TiAl alloys, not on EPM-processed TiAl [10].

The aim of the present research is to investigate the effect of Nb and Cr additions on the pore formation in PM-processed TiAl alloys. The chemical composition is ranged from binary TiAl alloys to quaternary alloys containing Nb and Cr up to 2 at.%, and the heating rate is controlled to be 10° C/min. The evolution of microstructure and pores during a sintering process is investigated and compared with previous results. Differential scanning calorimetry (DSC), X-ray diffraction, optical and SEM metallography, mercury intrusion tests are utilized for the identification of phases and pores.

2. Experimental

The nominal compositions of Ti₅₂Al₄₈, Ti₅₀Al₄₈Cr₂, Ti₅₀Al₄₈Nb₂ and Ti₄₈Al₄₈Cr₂Nb₂ in atomic percentage are employed for this study. Elemental hydride-dehydride (HDH) Ti powder with a particle size of –200 mesh is used as a raw material. Al powders have a particle size of –600 mesh and Cr and Nb powders have a particle size of –325 mesh. Ti, Nb, and Cr powders were obtained from Sejong materials Ltd., South Korea and Al powders were obtained from ALCO engineering, UK. All powders used in the present study have a purity >99%. The powders are weighed and filled in mixing jars in an argon gas environment to minimize the surface contamination. Powders are dry-mixed in a tumbler ball mill for 2 h at a rotating speed of 200 rpm. The mixed powders were then compressed in a steel die under a pressure of 370 MPa to manufacture green compacts of about 25.5 mm in diameter and 5.60 mm in height. Green compacts were heated at a heating rate of 10° C/min to 600° C or 1200° C and sintered for 2 h under a vacuum of 5 × 10^{–6} bar.

A differential scanning analysis was also performed to understand the reaction mechanism with a heating rate of 10° C/min using SDT Q6000. Phase analyses were performed for all nominal compositions by X-ray diffraction (XRD: Rigaku D/max 2500). Density and porosity of the alloys were calculated using the Archimedes principle, and pore parameters and size distribution were measured by a mercury intrusion testing machine (Micromeritics, Autopore IV 9510). The evacuation pressure was 50 mm/hg and hold time for the mercury intrusion of the sample was 10 s. Pore structure was observed by using a scanning electron microscope (SEM, Jeol JSM 6610LV).

3. Results and discussions

3.1. Reaction mechanism during processing of porous Ti–Al alloys

Fig. 1 shows the heat flow and weight loss curve for Ti₅₂Al₄₈ alloy. The heat flow indicates the presence of a distinctive exothermic reaction peak. The reaction starting at 560° C and ending at around the Al melting point corresponds to a solid-state phase transformation. The phase transformation appears to be related to the reaction, 3Ti + 3Al → 2Ti + TiAl₃. As the temperature reaches close to the Al melting point, the Al diffusion rate increases

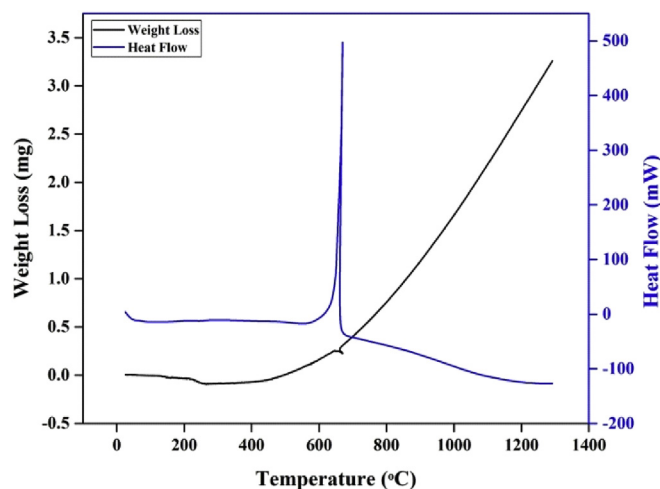


Fig. 1. Heat flow and weight loss curves as function of temperature for Ti₅₂Al₄₈ alloy.

rapidly and TiAl₃ phases form at interfaces between Ti and Al powders. Assuming that all of the aluminum is consumed during the solid-solution reaction, the reaction enthalpy calculated from the DSC curve is 115 kJ/mol. That is consistent with the previously obtained values by Wang et al. [11] (149 kJ/mol) and Sina et al. [12] (114 kJ/mol) for TiAl₃ formation. Gupta et al. [13] have proposed that the diffusion of Al has the prime role in the TiAl₃ forming reaction, based on the consideration of the activation energies. A large amount of heat, caused by the exothermic reaction, increases local temperature and eventually induces local melting of Al. The melting of Al is an endothermic reaction. However, Fig. 1 does not show the presence of any endothermic reaction, indicating that the remnant Al is too little to show any indication of melting and the reaction is overshadowed by the high TiAl₃ forming exothermic peak.

With increasing temperature further above the Al melting point, the DSC curve moves down slowly. Similar results have also been reported previously [14]. Once all Al atoms are consumed to form TiAl₃, additional phase transformations, TiAl₃+2Ti → 3TiAl and TiAl+2Ti → Ti₃Al, occur by the diffusion of Ti. After the TiAl₃-forming exothermic reaction is finished, Ti atoms become a major diffusing element for the formation of TiAl and Ti₃Al. The activation energy of self-diffusion for Ti in the intermetallic structure is much higher than that of Al [14] and heat is progressively absorbed for the formation of Ti-rich intermetallic phases [13], which causes a continuous decrease in the heat flow with temperature.

A significant weight loss is observed with increasing temperature above 500° C, which can be related to the release of hydrogen gas during the reaction process [5,7,11,12,15]. As HDH Ti powders are used in the present study, the entrapped gas can be released during heating under vacuum. In addition, the intermetallic phases, TiAl₃, TiAl, and Ti₃Al, have a very low solubility of H₂, and thus further enhances the gas release [5,16]. Simultaneously, Kirkendall voids are formed mainly by Al diffusion towards Ti-rich areas; these small voids unite together and help the gas to escape. The present rationale explains why the weight loss curve becomes inclined at temperatures around 560° C (near exothermic reaction starting temperature).

Fig. 2 shows the BSE image of Ti₅₂Al₄₈ alloy after sintering at 600° C and 1200° C for 2 h. It can be seen in Fig. 2a that a gray layer is formed around the bright Ti particle region, indicating the formation of the second phase (TiAl₃) between Al and Ti interfaces. The EDS point analysis of Fig. 2 is given in Table 1 from which it can

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