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Fabrication of TiAl by blended elemental powder semisolid forming

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Semisolid forming from blended elemental powders is investigated on Ti-Al alloy. The effects of the forming pressure, temperature, and pressure holding time on the density of the green billets are discussed. The forming method is outlined as follows: the mixture of the blended elemental powders are filled in a metal mold, and heated to approximately the melting point of aluminum. Thereafter, the forming pressure is loaded and held for the prescribed time. The pore-free green compacts are obtained under almost all the present forming conditions. However, pores are observed after alloying heat treatment. The volume fraction of the pores depends on the microstructures of the green billets. Experiments have also been carried out on the effects of the alloying heat treatment conditions on the mechanical properties of the compacts. © *2001 Kluwer Academic Publishers*

1. Introduction

The semisolid forming processing is an innovative technique for the near-net-shape processing of alloys and composites with attendant integrity of microstructure and mechanical properties. It can offer not only the complex shaped components forming capabilities of casting method, but also achieves the refinement of the microstructure of forging method simultaneously [1–4]. In the semisolid forming method, there are two kinds of distinctive processes. One of them was proposed by Spencer *et al.* [5] in the early 1970's, in which the alloy slurry is compulsively agitated between the solidus and the liquidus. The alloy may therefore be injected into a die in a slurry state with broken primary crystallite as the crystallizing process is disturbed by agitating. The other is the so-called "elemental powders semisolid forming" method proposed by Young *et al.* [6] in 1980s. After mixing the powders with the high and low melting points, the blended mixture is then heated up to a slurry state because of the melting of the powders which has the lower melting point, and thereafter formed in a mold.

The elemental powders semisolid forming method is capable of forming in a slurry state at low temperature. For instance, even for the high activity alloy system such as Ti-Al, it can be formed at the temperature just higher than the melting point of aluminum, i.e., 933 K, and the normal conditions of high vacuum or inert gas atmosphere becomes unnecessary, meanwhile, the liquid ratio can be freely controlled according to the aluminum content of the alloy. Recently, the elemental powders semisolid forming method applied to Ti-6%Al based alloys has been successfully carried out [7, 8].

Titanium aluminide TiAl exhibits low densities, high strength, excellent creep resistance and oxidation resistance at elevated temperatures. Its potential uses in structural and aeronautical applications have generated considerable interest in the past decades. It is also considered to be a promising material, which will support the future engineering [9–13]. However, the machining of the titanium aluminide (such as cutting and forging) is difficult and its manufacturing cost is extremely high due to its low thermal conductivity, low specific heat and high toughness. Meanwhile, casting of titanium aluminide also requires strictly controlled atmosphere and special melting technique, and refractory materials of crucibles and molds are extremely limited because of its high chemical activity.

In the present study, the elemental powders semisolid forming method as applied to titanium aluminide TiAl is investigated in detail. The influence of process parameters, e.g., applied forming pressure, forming temperature, holding time, particle size, and alloying heat treatment on the microstructures, porosity and mechanical properties of the fabricated materials are discussed. Mechanical alloying (MA) process which can effectively decrease the particle's size was also used in order to suppress the pores of the microstructure. Meanwhile, comparisons of the microstructure, porosity and mechanical property between the conventional casting material and the semisolid forming material are also performed.

2. Experimental procedures

2.1. Starting materials

Starting materials were pure spherical atomized powders of titanium (99.9% purity, $\leqq 50\ \mu$m) and aluminum (99.9% purity, $\leqq 10\ \mu$m). They were thoroughly blended in a mortar. Two kinds of feedstock billets were prepared. One was green-compacted from mechanically alloyed (hereafter, MA in brief) mixture and another was from un-MA powder mixture. The MA processes were performed in a planetary ball milling by using a hardened high Cr steel vial (500 ml) and balls (Φ10 mm) under 66 kPa argon gas atmospheres. For each run, 20 grams of the powders were loaded. The weight ratio of ball to powder was 10 : 1. About 0.5 mass% of stearic acid [$CH_3(CH_2)_{16}COOH$] was used as the process control agent. The ball milling was carried out at the rotation rate of 170 r.p.m. and cooled by air conditioner. The time of milling is 36 ks.

2.2. Forming process

The mixture was filled into a metal mold, and heated up to a temperature in the range of 943–973 K. Then, the metal mold with billet in it was moved to another pre-heated furnace at the temperature of 673 K and the pressure of 300–400 MPa was loaded for 1 minute. The obtained coin-shaped sample was then alloying heat treated for the step-I at 923 K for 2–5 hours under a pressure of 200 MPa. Thereafter, sample was alloying heat treated for step-II at 1473 K for 5–48 hours under the vacuum of 1.3×10^{-3} Pa. Fig. 1 schematically shows the forming process of the experiments.

2.3. Characterization

The fabricated compacts are characterized by density measurements by using Archimedes method, microscopic observations, X-ray diffraction analyses, and Vickers hardness. For comparison, a TiAl intermetallic compound of the same compositions was prepared by casting and characterized in the same way.

3. Results and analyses

3.1. Microstructure

Microstructures of the samples semisolid formed at 943 K under a pressure of 400 MPa are shown in Fig. 2b, where the starting powders are just blended mixture, and Fig. 2a, where the starting powders are

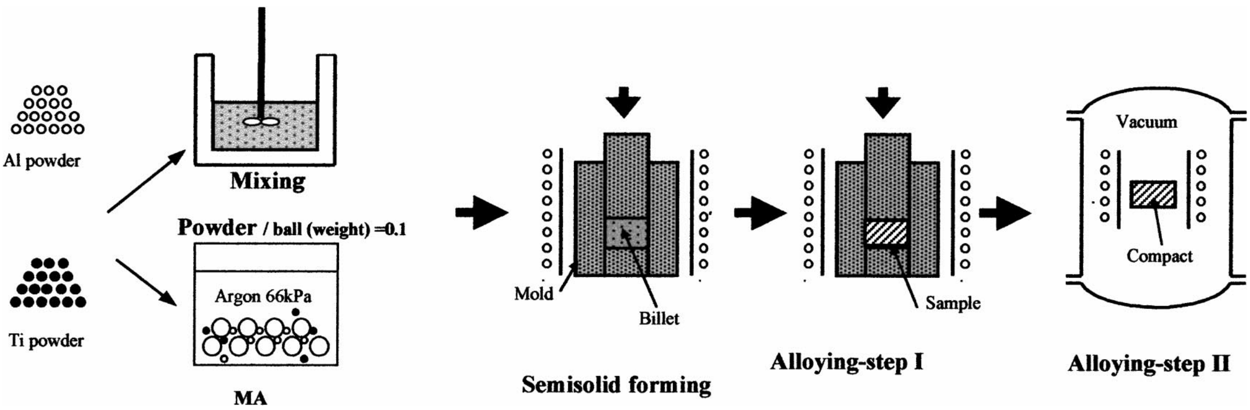

Figure 1 Schematic illustration of TiAl alloy semisolid processing.

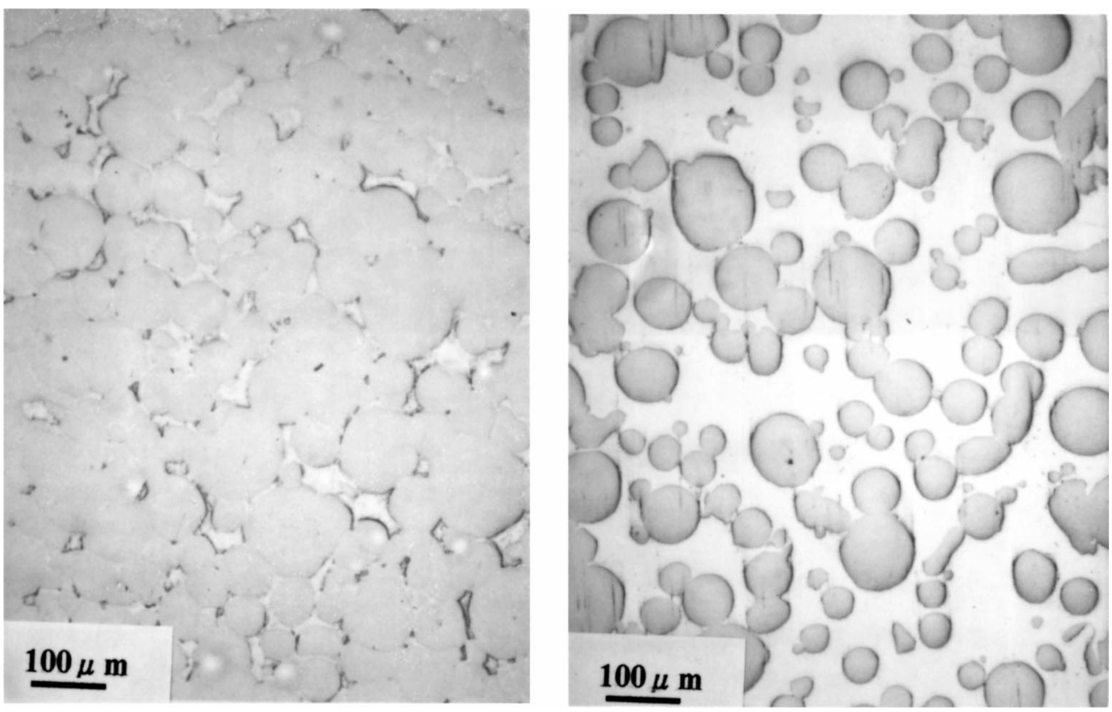

Figure 2 Micrographs of Ti-Al green compacts semisolid formed (a) By MA powders, and (b) By un-MA powders.

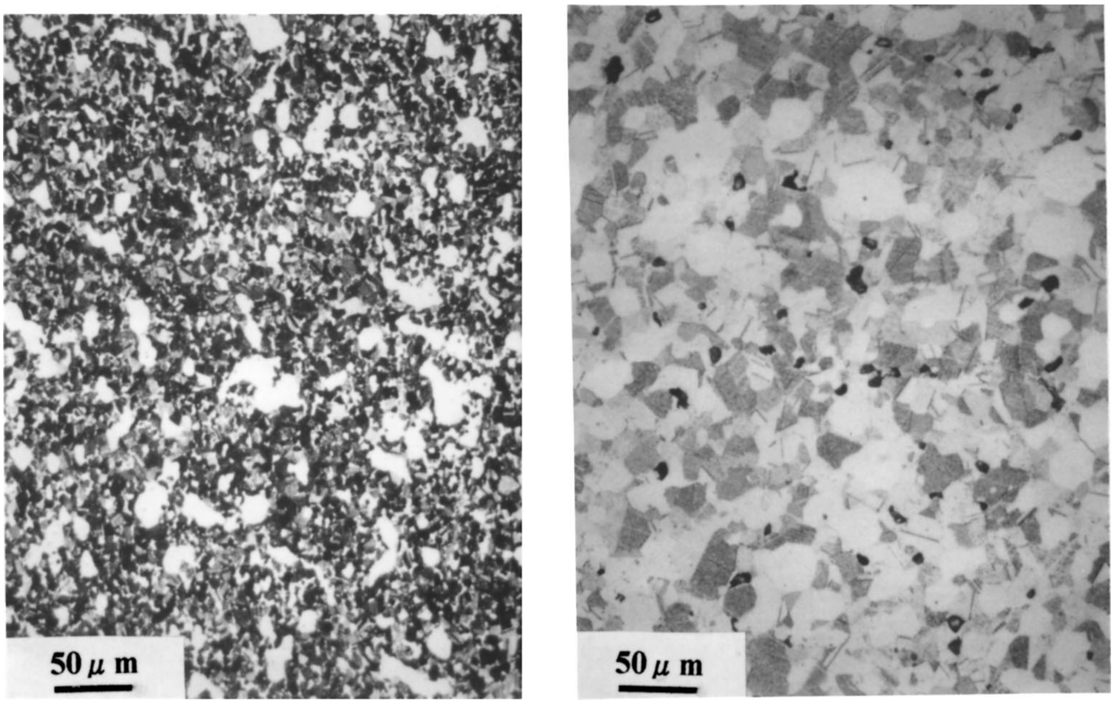

Figure 3 Micrographs of TiAl compacts after alloying heat treatments (a) By MA powders, and (b) By un-MA powders.

MA mixture. It can be seen that both samples are pore-free. In the present study, conditions for obtaining the pore-free green compacts are found to be at the range of 943–983 K and a pressure at the range of 300–400 MPa. Beyond these conditions, low temperatures and/or low pressures may result pores, while high temperatures and/or high pressures may result pores and intermetallic compounds such as Al_3Ti phase and Ti_3Al phases in the microstructures as inspected by X-ray diffraction.

Microstructures of the compacts after alloying heat treatments step-I at 923 K for 5 hours and step-II at 1473 K for 24 hours in vacuum are shown in Fig. 3b, where the starting powders are just blended mixture, and Fig. 3a, where the starting powders are MA mixture. It can be seen that pores are found in the microstructure of the compact made from un-MA powders as shown in Fig. 3b; However, no pore was found in the microstructure of the compact made from the MA powders as shown in Fig. 3a. In addition, inspection of the microstructures after alloying heat treatments reveals that the grain size in Fig. 3a is finer than Fig. 3b. Observations and X-ray diffraction reveal that fine and homogeneous microstructures consisting of γ-TiAl and α_2-Ti_3Al phases can be obtained after alloying heat treatment step-I at 923 K for longer than 5 hours and step-II at 1473 K for a time at the range of 24–48 hours. Pores and heterogeneous microstructures are found in the compacts when alloying heat treatment temperature is lower than the above, and/or the time shorter than above.

This suggests that the pore-free compact with a fine and homogeneous microstructure consisting of γ-TiAl and α_2-Ti_3Al can be obtained by semisolid forming process and the further alloying heat treatments step-I and step-II.

3.2. Density

The influence of the time of alloying heat treatment step-I on the densities of the compacts is shown in Fig. 4. It can be seen that, in general, densities of the compacts made from the mechanically alloyed powders are higher than that of the compacts made from un-MA powders. And it shows the same tendency in both kinds of starting powders that the density increases with the time of the alloying heat treatment step-I. For compacts made from the mechanically alloyed powders, the density reaches the maximum, i.e., the theoretical density value of TiAl alloy when the time of the alloying heat treatment step-I is longer than 5 hours.

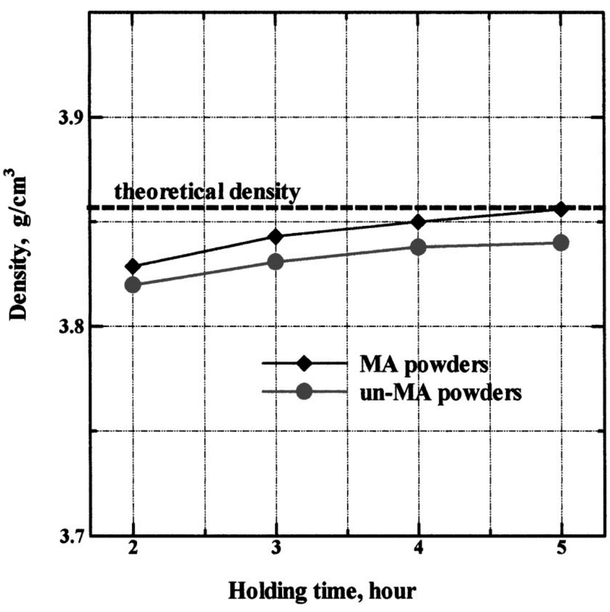

Figure 4 Effect of the alloying heat treatment step-I on the densities of TiAl compacts fabricated from different starting powders.

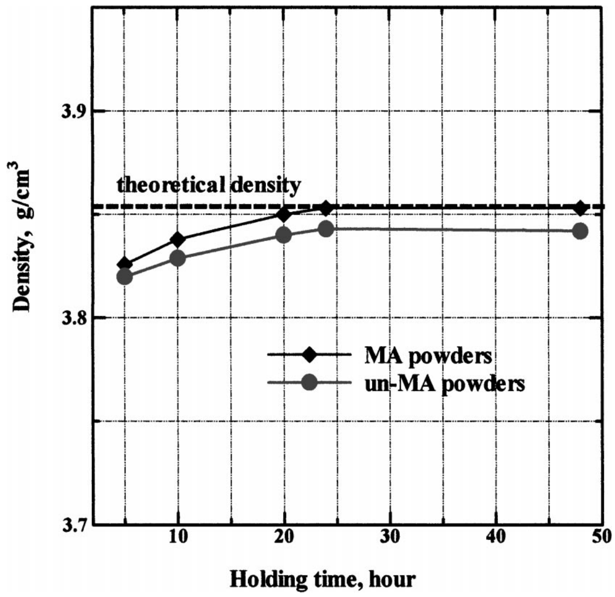

Figure 5 Effect of alloying heat treatment step-II on the densities of TiAl compacts (alloying temperature: 1473 K).

Fig. 5 shows the effect of the time of alloying heat treatment step-II on the densities of the compacts. Densities of the compacts made from the mechanically alloyed powders are higher than that of the compacts made from un-MA powders. The density increases with the time of alloying heat treatment step-II and reaches the theoretical density value when the time longer than 24 hours. Microstructure observations reveal that fine and homogeneous phases of γ-TiAl and α_2-Ti_3Al are obtained when samples alloying heat treated at 1475 K for 24 hours. X-ray diffraction confirms the results of the optical microstructure inspections.

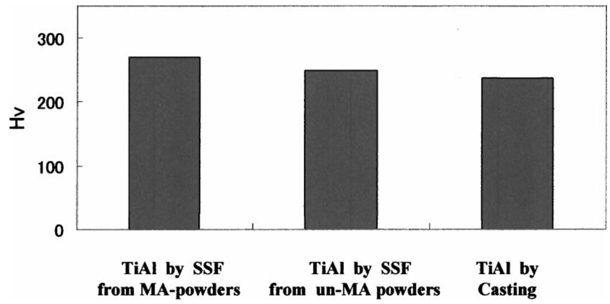

Figure 6 Vickers hardness of various TiAl samples.

3.3. Vickers hardness

Fig. 6 shows the Vickers hardness of the samples made by different methods. For comparing, value of cast sample is also listed. It can be seen that the hardness of the compact made by mechanical alloying powders is the highest, then, the compact made by un-MA powders. The cast sample has the lowest Vickers hardness. It can be deduced that the pore-free compact with a fine and homogeneous microstructure consisting of γ-TiAl and α_2-Ti_3Al has the highest strength.

3.4. Discussion

It is conclusively demonstrated from the experimental results that the pore-free Ti-Al green compact can be obtained if the conditions of the semisolid forming process are controlled properly, as shown in Fig. 2. However, the distribution of the aluminum among the titanium particles is substantially different for the two starting powders. The aluminum penetrates uniformly into the titanium particles and forms a network around them in the sample fabricated from MA powders (Fig. 2a) and the aluminum distributes inhomogeneously among

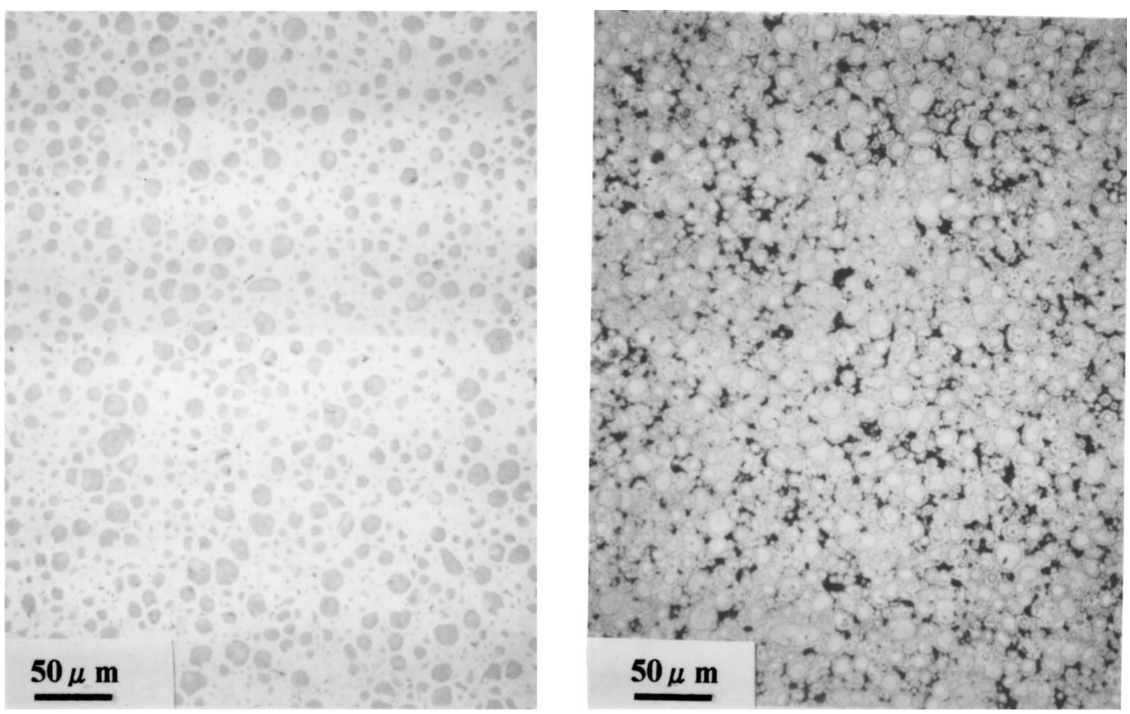

Figure 7 Micrographs of Ti-Al green compact after alloying heat treatment step-I (b) By MA powders, and (b) By un-MA powders.

the titanium particles in the sample fabricated from the un-MA powders (Fig. 2b).

Pores probably occur in the microstructure of the TiAl compact after alloying heat treatments step-I and step-II because of the diffusion of the aluminum and the reaction between the titanium and aluminum during the alloying heat treatment processes. The crucial factor that influences the microstructure and density of the TiAl compacts is the distribution of the aluminum in the green compacts. Microstructure observations reveal that after alloying heat treatment step-I, there are neither pores nor single phase of aluminum left in the microstructure of the sample fabricated from the MA powders, while there are pores around the titanium particles in the microstructure of the sample fabricated from the un-MA powders, as shown in Fig. 7a and b, respectively. During the alloying heat treatment step-I, the aluminum diffuses into the titanium particles and partially reacts with titanium and form Al_3Ti, TiAl, and $TiAl_3$ intermetallics. In the compact with the coarse aluminum matrix, when the aluminum is diffused into the adjacent titanium particles and partially reacted with them, apertures appear between the titanium particles because of the thermal shrinkage of the aluminum. The pores are formed at the location where coarse aluminum exists. These pores grow bigger because of the volume shrinkage due to the further production of the intermetallics during the alloying heat treatment step-II.

4. Summary

The forming conditions of near-net-shaping method by using the non-equilibrium semisolid state of elemental powders mixture were fundamentally discussed regarding to the high active materials which are difficult for shaping by casting as well as by forging method on TiAl alloy. Results are as follows:

1. Completely pore-free TiAl green compacts can be obtained by using semisolid forming method. The forming conditions can be selected as heating to a temperature of 943–973 K, and under a pressure of 400 MPa holding for only one minute at 673 K.
2. Mechanical alloying process is an effective approach for obtaining pore-free TiAl compact.
3. The green compacts can transform into TiAl intermetallic compounds during the following two steps of alloying heat treatments.
4. The temperatures and times of the two steps of alloying heat treatments are critical factors affecting the density of the TiAl compact. Conditions of a temperature of 913 K and 5 hours for step-I, and 1473 K and 24 hours for step-II are necessary for obtaining the pore-free TiAl compact.

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