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Fabrication of multilayered titanium aluminide sheets by self-propagating high-temperature synthesis reaction using hot rolling and heat treatment

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This study is concerned with the fabrication of multilayered and bulk Ti aluminide sheets by self-propagating high-temperature synthesis (SHS) reaction using hot rolling and heat treatment. A multilayered Ti/Al sheet was prepared by stacking thin Ti and Al sheets alternatively. When this sheet was hot-rolled and heat-treated at 1000°C, a multilayered sheet composed of Ti_3Al and TiAl was made through the process of formation and growth of intermetallic phases at Ti/Al interfaces and porosity reduction. A bulk Ti aluminide sheet having a lamellar structure of TiAl and Ti_3Al was also fabricated successfully by heat treatment at 1400°C.

1. Introduction

Ti aluminide sheets formed in honeycomb or sandwich shapes have been applied to structural components of airplanes, and thus have received growing attentions because of their significant effects on weight and cost reduction [1, 2]. These applications usually require thin sheets under 1 mm in thickness, but have serious problems such as difficult plastic deformation caused by poor room-temperature ductility and fracture toughness, high cost, and oxidation [1–3]. To solve the plastic deformation problem of Ti aluminides, studies have been recently undertaken to fabricate near-net shaped sheets by inducing self-propagating high-temperature synthesis (SHS) reaction after two metals of Ti and Al are mixed or stacked [4, 5]. Fabrication of Ti aluminides by SHS enables to make near-net shaped sheets, and has advantages of simplified fabrication process and reduced cost. However, this method faces restrictions in material size and in continuous production since high-temperature compression is required to minimize pores formed during SHS [4–6]. Thus, the present study suggests a new way to continuously fabricate multilayered Ti aluminide sheets by SHS reaction after stacking thin Ti and Al sheets alternatively. The hot rolling and heat treatment processes, in which the sheet size is less restricted and continuous production is possible, were selected. Also, phases formed at Ti/Al interfaces or inside layers were analyzed, and their formation and growth behaviors were investigated.

2. Experimental procedures

Ti/Al multilayers were prepared by alternatively stacking thin Ti (purity; 99.8%) and Al (purity; 99.999%) sheets of 0.1 mm in thickness as shown in Fig. 1a. These multilayers were canned by a stainless steel foil of 1 mm in thickness to prevent rapid cooling by rolls, oxidation, and leakage of Al melt at temperatures higher than Al melting point (660°C) during hot rolling. To enhance Ti/Al interfacial bonding, the canned Ti/Al multilayers were pre-rolled after holding at 500°C for 3 min (Fig. 1b). 1000°C was chosen as the optimal hot-rolling temperature, considering that many $TiAl_3$ phases having poor ductility are formed when Ti is reacted with Al above the Al melting point. The Ti/Al multilayered sheet was held at 1000°C for 10 min, and then rolled. This process was repeated three times. Initial and final thicknesses of the sheet were 7.3 mm and 1.9 mm, respectively (total reduction ratio; 74%), and total heating time at 1000°C was 30 min. After rolling, the sheet was heat-treated at 1000°C for 1–10 h in a quartz tube filled with argon gas, and was water-cooled.

After the multilayered sheet was etched by a Kroll solution (H_2O 98.5 ml, HF 0.5 ml, and HNO_3 1 ml), it

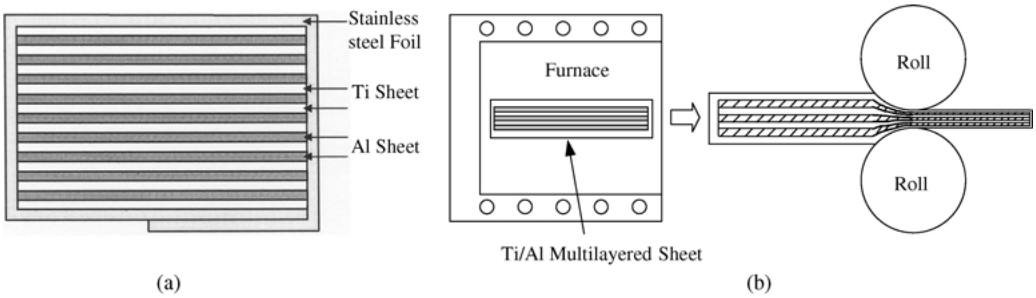

Figure 1 Sketch illustrating (a) a multilayered Ti/Al sheet canned by a stainless steel foil and (b) heat treatment and hot rolling processes of the multilayered Ti/Al sheet.

was observed by an optical microscope and a scanning electron microscope (SEM). Interfacial phases were analyzed by an X-ray diffractometer and a transmission electron microscope (TEM). TEM thin foils were prepared by dimpling and ion-milling of mechanically ground thin discs (about 100 μm in thickness), and interfacial phases were examined by a TEM under an accelerated voltage of 200 keV.

3. Results and discussion

3.1. Microstructural modification of multilayered Ti/Al sheet

Fig. 2a is an optical micrograph of the multistacked Ti/Al sheet pre-rolled at 500°C. Ti/Al interfacial bonding is good, and any interfacial phases are not observed. Micrographs of the sheet held at 1000°C for 5 min and 10 min after pre-rolling are shown in Fig. 2b and c. Spheroidal phases are formed at Ti/Al interfaces and inside Al layers (Fig. 2b). When held at 1000°C for 10 min. Many $TiAl_3$ phases are formed coarsely, thereby eliminating the Al layer (Fig. 2c). Many pores are also observed around them as indicated by arrows. An X-ray diffraction pattern of this sheet is presented in Fig. 3a. As $TiAl_3$, Ti, and Al peaks are observed here, the spheroidal phases of Fig. 2b are identified to be $TiAl_3$.

Fig. 4a is an SEM micrograph of the sheet hot-rolled at 1000°C. The Al layers disappear, leaving layers of Ti and $TiAl_3$; the bright areas for Ti layers and the dark ones for $TiAl_3$ layers. The pores observed in Fig. 2c are greatly reduced according to hot rolling. When the hot-rolled sheet is observed at a higher magnification, three types of phases, indicated as ‘A’, ‘B’, and ‘C’, are revealed (Fig. 4b). Fine pores of several micrometers in size are observed in $TiAl_3$ layers, whereas they are not

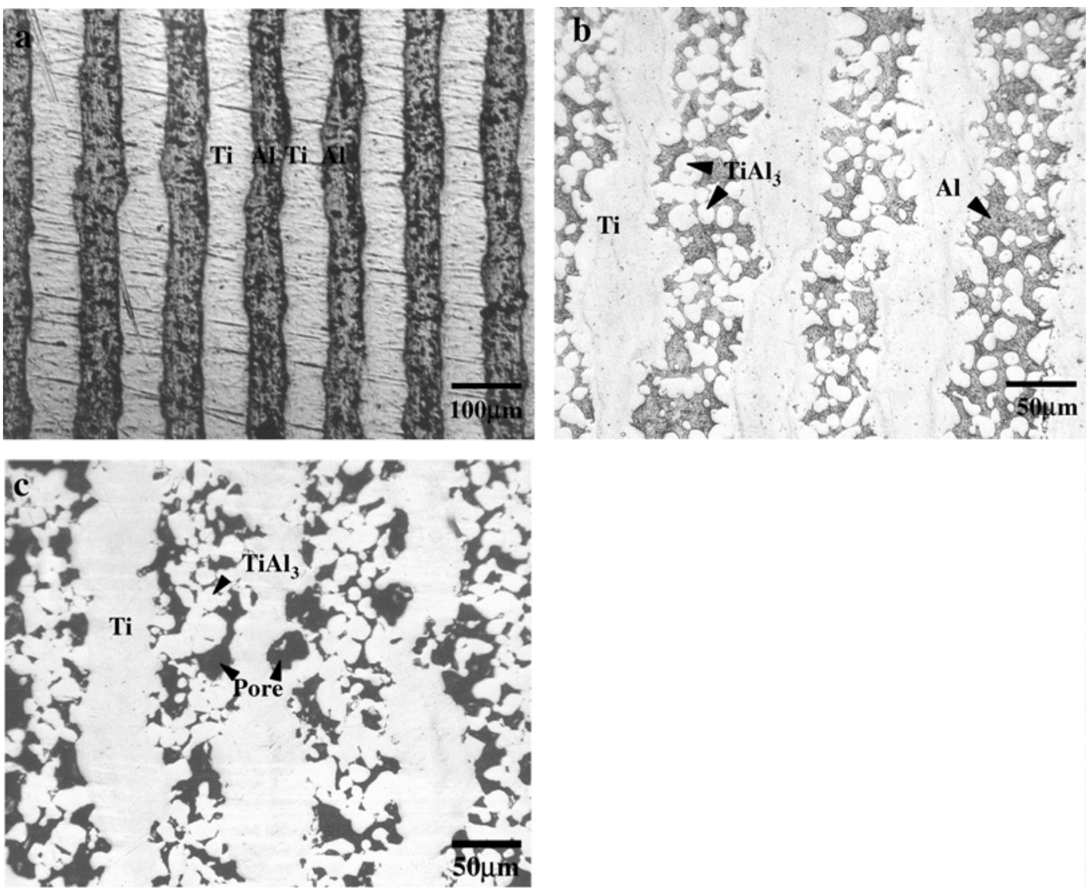

Figure 2 Optical micrographs of (a) the Ti/Al multilayered sheet hot-rolled at 500°C, (b) the multilayered sheet held at 1000°C for 5 min, and (c) the multilayered sheet held at 1000°C for 10 min.

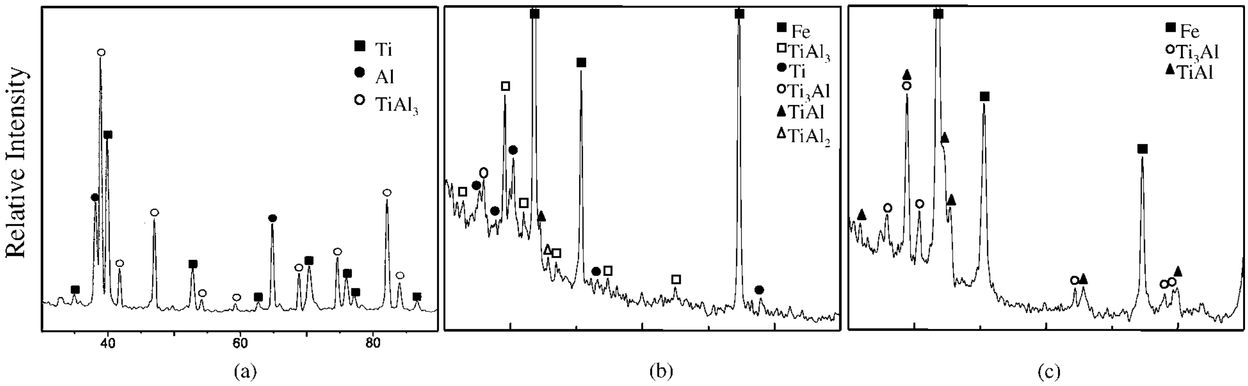

Figure 3 X-ray diffraction patterns of the multilayered sheet (a) held at 1000°C for 5 min, (b) hot-rolled at 1000°C, and (c) heat-treated at 1000°C for 10 h after hot-rolling.

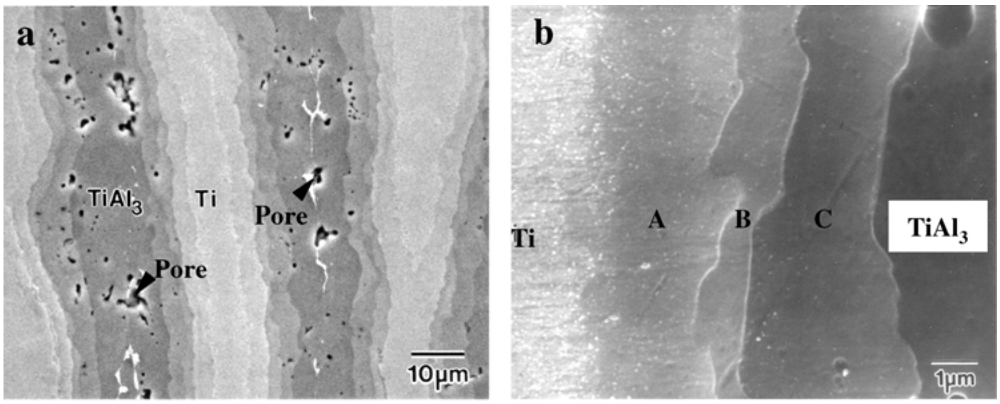

Figure 4 (a) and (b) SEM micrographs of the multilayered sheet hot-rolled at 1000°C. (b) is a higher magnification micrograph showing a Ti/$TiAl_3$ interfacial area.

found in Ti layers. Fig. 3b presents an X-ray diffraction pattern of the hot-rolled sheet. Besides peaks of Fe, which is the canning material, peaks of Ti_3Al, TiAl, and $TiAl_2$ as well as peaks of Ti and $TiAl_3$ are observed. This indicates that the hot-rolled sheet consists of a multilayered structure in which $TiAl_3$, $TiAl_2$, TiAl, Ti_3Al, and Ti phases are aligned in order.

Fig. 5a and b are SEM micrographs of the multilayered sheet hot-rolled and subsequently heat-treated at 1000°C for 1 h and 10 h, respectively. The thickness of $TiAl_3$ and Ti decreases, whereas that of $TiAl_2$, TiAl, and Ti_3Al increases (Fig. 5a). After the 10-h heat treatment, only two phases exist as shown in Fig. 5b. An energy dispersive spectroscopy (EDS) line profile in Fig. 5b indicates more detection of Al in dark gray layers, whereas more of Ti in light gray layers. According to the X-ray diffraction analysis data of this sheet, Ti_3Al and TiAl peaks are observed in addition to Fe peaks (Fig. 3c). In the 10-h heat-treated sheet, thus, Ti, $TiAl_2$, and $TiAl_3$ have mostly disappeared, leaving multilayers composed of only Ti_3Al (dark gray layer in Fig. 5b) and TiAl layers (light gray layer in Fig. 5b). TEM bright-field image of this sheet is shown in Fig. 6a, accompanied with selected area diffraction patterns obtained from Ti_3Al and TiAl (Fig. 6b and c). These patterns confirm that the two phases present in the 10-h heat-treated sheet are TiAl and Ti_3Al.

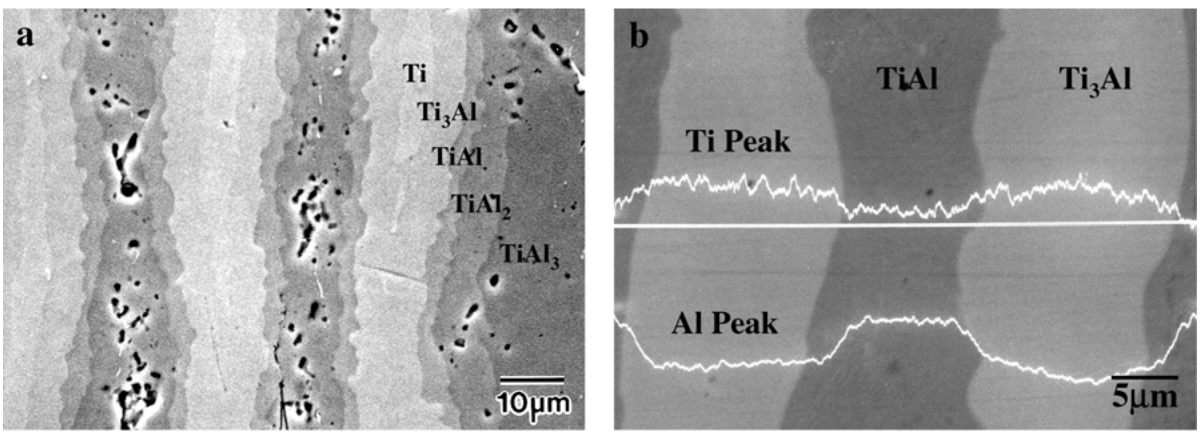

Figure 5 SEM micrographs of the multilayered sheet heat-treated at 1000°C for (a) 1 h and (b) 10 h after hot-rolling at 1000°C.

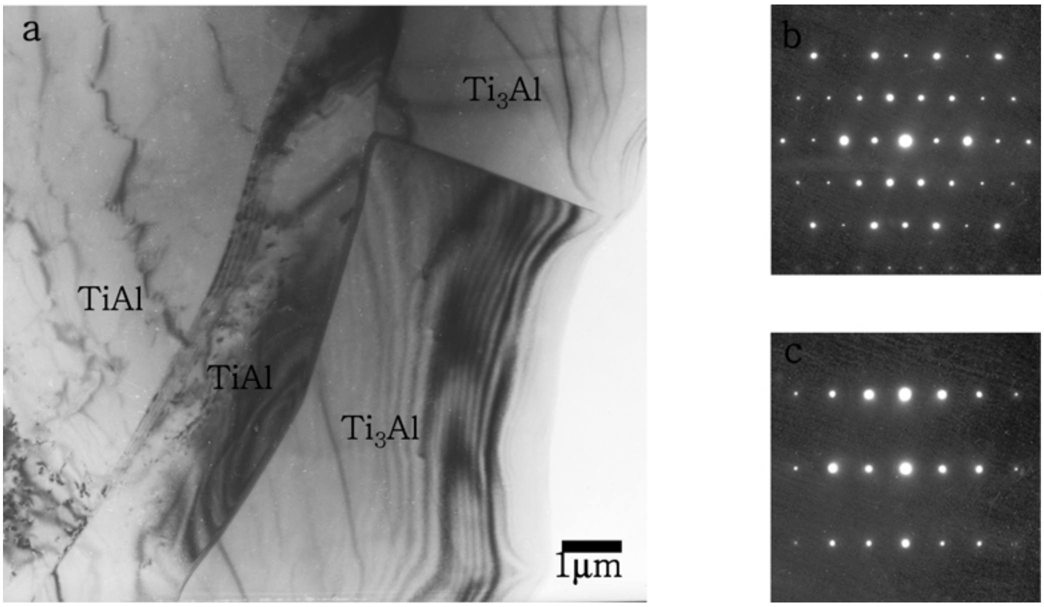

Figure 6 (a) TEM bright-field image of the multilayered sheet heat-treated at 1000°C for 10 h and (b) and (c) selected area diffraction patterns of TiAl and Ti_3Al, respectively.

3.2. Formation and growth process of multilayered Ti aluminide sheet

According to Sujata *et al.* [7], when a composite material fabricated by inserting an Al bar of 8 mm in diameter into the center of a Ti cylinder of 20 mm in diameter was heat-treated at a temperature higher than the Al melting point, a $TiAl_3$ layer was formed at Ti/Al interfaces by SHS reaction ($Ti(s) + 3Al(l) \rightarrow TiAl_3$). In this study, spheroidal $TiAl_3$ phases are also observed at Ti/Al interfaces (Fig. 2b) when the multistacked Ti/Al sheet was held at 1000°C for 5 min. These $TiAl_3$ phases are continuously formed at Ti/Al interfaces by SHS reaction until Al is completely consumed. Besides the $TiAl_3$ formation at Ti/Al interfaces, continuous SHS reaction also allows $TiAl_3$ phases to intrude even into Al layers because of high exothermic heat ($Ti + 3Al \rightarrow TiAl_3 + \Delta H$) and the convection of the Al melt [7]. When $TiAl_3$ phases are formed by reaction of Ti with Al, the temperature at the reactive interface rises over the melting point of $TiAl_3$ (1392°C) by high exothermic heat [8] to locally melt $TiAl_3$ phases formed at Ti/Al interfaces. As the reactive interfaces move toward Ti with decreasing temperature [9], $TiAl_3$ phases are precipitated again, and exist as islands near the reactive interfaces.

Pores formed during holding at 1000°C are refined by hot rolling, and their number also decreases (Fig. 4a). In addition, interfacial phases such as $TiAl_2$, TiAl, and Ti_3Al are formed at Ti/$TiAl_3$ interfaces due to reaction between Ti and $TiAl_3$ (Fig. 4b). When the hot-rolled sheet is heat-treated, the thickness of Ti and $TiAl_3$ decreases, while interfacial phases of Ti_3Al, TiAl, and $TiAl_2$ grow up (Fig. 5a). In the next heat-treatment stage, Ti and $TiAl_3$ are completely extinguished, and a multilayered sheet composed of Ti_3Al, TiAl, and $TiAl_2$ is formed. With longer heat-treatment time, TiAl grows as $TiAl_2$ is consumed, and eventually a multilayered sheet of Ti_3Al and TiAl is achieved (Fig. 5b). Fig. 7a through f are schematic diagrams illustrating the formation process of the multilayered Ti aluminide sheet when the multistacked Ti/Al sheet was hot-rolled and heat-treated at 1000°C.

In addition, a bulk Ti aluminide sheet having a lamellar structure of TiAl and Ti_3Al can be obtained when the

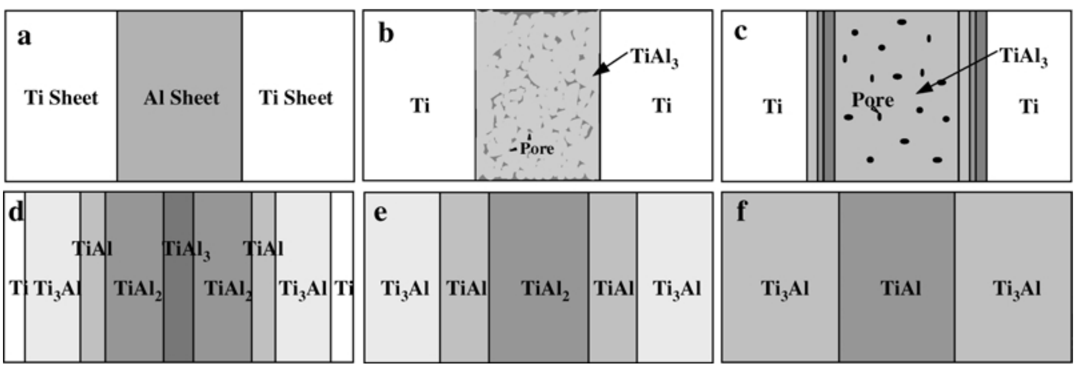

Figure 7 Schematic diagrams illustrating the formation process of a multilayered Ti aluminide sheet by hot rolling and heat treatment: (a) stacking of thin Ti and Al sheets, (b) formation of $TiAl_3$ phases at Ti/Al interfaces and Al layers, (c) densification of the $TiAl_3$ layer and the formation of interfacial phases between Ti and $TiAl_3$ layers after hot rolling, (d) growth of Ti_3Al, TiAl, and $TiAl_2$ and reduction of Ti and $TiAl_3$, (e) further growth of Ti_3Al, TiAl, and $TiAl_2$, and (f) eventual formation of a multilayered sheet composed of Ti_3Al and TiAl.

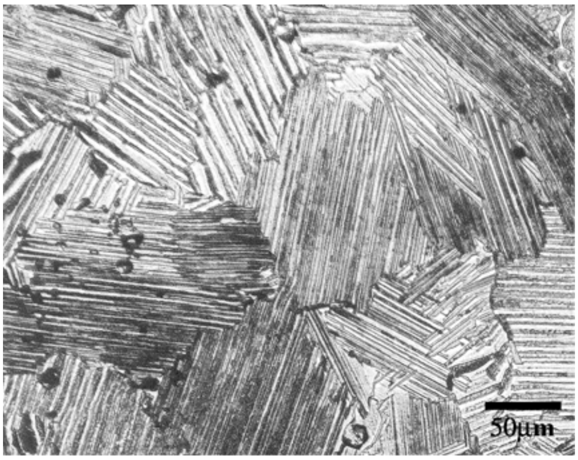

Figure 8 Optical micrograph of a bulk Ti aluminide sheet having a lamellar structure composed of thin TiAl and Ti_3Al layers after hot-rolling at 1000°C and heat treatment at 1400°C for 30 min.

hot-rolled sheet is heat-treated at 1400°C for 30 min and then furnace-cooled (Fig. 8). Ti, $TiAl_3$, and other interfacial phases disappear, and the microstructure changes to a lamellar structure composed of thin layers of TiAl (γ) and Ti_3Al (α_2), like in a typical lamellar structure of Ti aluminides fabricated by casting. This fabrication method of the bulk Ti aluminide sheet having a lamellar structure has several advantages, i.e., prevention or minimization of interior cracks or pores, decrease in hot-rolling temperature, and achievement of desired sheet size by controlling the number and thickness of thin Ti and Al sheets stacked, over the methods by hot forging or rolling of conventionally cast Ti aluminides [1–3]. In the latter case, the poor formability of Ti aluminides requires higher hot rolling temperatures of 1200–1400°C and increased number of rolling pass, and interior cracks are easily initiated during hot deformation. It also has merits such as less restriction in material size and continuous fabrication using existing facilities, over the fabrication methods of Ti aluminide sheets by SHS reaction [4–6].

4. Summary

A multilayered Ti aluminide sheet was fabricated by SHS reaction using hot rolling and heat treatment in the present study, and its microstructural modification occurred during fabrication was investigated. A multilayered Ti/Al sheet was prepared by stacking thin Ti and Al sheets alternatively. Microstructural analysis on the hot-rolled Ti/Al sheet revealed that intermetallic phases such as $TiAl_2$, TiAl, and Ti_3Al were formed at Ti/$TiAl_3$ interfaces due to interaction between Ti and $TiAl_3$. When this hot-rolled sheet was heat-treated at 1000°C, interfacial phases such as Ti_3Al, TiAl, and $TiAl_2$ were grown as Ti and $TiAl_3$ were consumed. As the heat treatment proceeded, TiAl grew further, eventually leading to the multilayered sheet composed of Ti_3Al and TiAl. A bulk Ti aluminide sheet having a lamellar structure of TiAl and Ti_3Al, instead of the multilayered sheet, was also fabricated successfully by heat treatment at 1400°C. This fabrication method of the bulk sheet had several advantages such as prevention or minimization of interior cracks or pores and decrease in hot-rolling temperature, over the methods by hot forging or rolling of conventionally cast Ti aluminides. From these findings, an idea to fabricate multilayered or bulk Ti aluminide sheets by hot rolling and heat treatment was suggested as an economical and continuous fabrication method.

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