

## A Study on the Characteristics of Amorphous TiAl by P/M Processing

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**Abstract** The P/M processing of titanium aluminide using amorphous TiAl is developed by which it is possible to overcome inherent fabricability problems and to obtain a fine microstructure. A high quality amorphous TiAl powder produced by reaction ball milling shows clear glass transition far below a temperature at the onset of crystallization in differential scanning calorimetry above a heating rate of 0.05 K/s. We obtained a fully dense compact of amorphous TiAl powders, encapsulated in a vacuumed can, via viscous flow by hot isostatic pressing (HIP). Isothermally annealing of HIP'ed amorphous compact under a pressure of 196 MPa shows a progressive growth of  $\gamma$ -TiAl phase with  $\alpha_2$  ( $\text{Ti}_3\text{Al}$ ), which is characterized by increasing sharpness of X-ray peaks with temperature. Fully dense HIP'ed compact of titanium aluminide TiAl shows a high hardness of 505 Hv, suggesting strengthening mechanisms by sub-micron sized grain of  $\gamma$ -TiAl and particle-dispersion by second phase constituent,  $\alpha_2$ .

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**Key words** : P/M processing, Titanium aluminide, Reaction ball milling, Crystallization, particle-dispersion

### 1. Introduction

Intermetallic compound, especially titanium aluminide is a promising structural material in light-weight high-temperature applications [1-5]. However, titanium aluminides have been recognized to be difficult-to-fabricate alloys, due to low ductility at ambient temperature, poor machinability, inhomogeneity and segregation, in conventional ingot metallurgy[6-9]. Powder metallurgy (P/M) technique has the potential to obtain a near-net-shape forming of intermetallic compound and a unique microstructure such as particle dispersion. These processing include a self-propagating high-temperature synthesis of powder elements and a consolidation of rapidly solidified crystalline powder by hot isostatic pressing (HIP), pseudo-HIP and hot pressing and powder forging. However, the uses of a high consolidating temperature needed to get a full density compact and a mixture of element powders lead to a coarsening of desired microstructure and the

formation of unexpected phases.

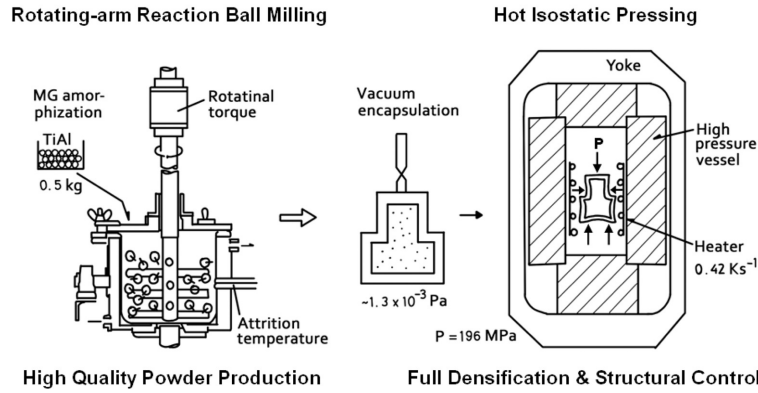
On the other hand, mechanical alloying (MA) makes it possible to produce an amorphous powder and a metallic powder with nanometer-sized grain [10-13], which has not been prepared by melt-quenching. We have recently shown that MA amorphous Ni-Al alloy powder can be easily consolidated to a full density amorphous product via viscous flow by HIP at a relatively low temperature [14]. We here are going to develop a P/M processing of titanium aluminide using amorphous TiAl, to provide a route for an improved strength and a refined structure.

### 2. Experimental Procedures

Figure 1 illustrates the block diagram of a P/M processing developed for the synthesis of intermetallic compounds using solid state amorphized metallic powder. Our processing mainly consists of a high quality powder production of amorphous TiAl

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**Fig. 1.** P/M processing for the synthesis of titanium aluminide using solid state amorphized TiAl powders.

by reaction ball milling, a fully dense compaction via viscous flow and the formation of intermetallic phase by HIP. A more detail description of experimental procedure in this study is described as follow.

As a first step, a rotating-arm reaction ball mill (Fritsch GmbH, model Pulverisette 5), that has peripheral equipments to measure the attrition temperature inside the tank and the applied torque, was used to prepare amorphous powder ( $27\ \mu\text{m}$ ) of TiAl in an equi-atomic composition by mechanical grinding (MG) pre-alloyed powders (approximately 500 g) mainly consisting of  $\gamma$ -phase. Next, amorphous TiAl powders were encapsulated in a copper tube with a diameter of  $10\ \text{mm}\phi$  or a stainless can with a diameter,  $27\ \text{mm}\phi$  in vacuum. Then, these encapsulated amorphous TiAl powders were heated up to the temperatures of 853 K and 873 K respectively using a heating rate of  $0.42\ \text{K/s}$ , and then consolidated for 1800 s by a laboratory hot isostatic press with a pressure ( $P$ ) of 196 MPa. Finally, annealing of an amorphous TiAl compact was performed under a pressure of 196 MPa at the temperature ranging from 948-1273 K for 1800 s for the formation of intermetallic compound phase.

For attritted TiAl amorphous powders and HIP'ed compacts, the structure was characterized by conventional X-ray diffraction (XRD) using a radiation

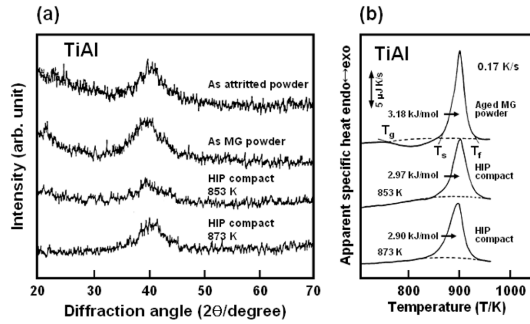
of  $\text{CuK}\alpha$ , and thermal properties were examined by differential scanning calorimetry (DSC) with a data acquisition system. The optical microscopy was utilized to observe the surface of a HIP'ed amorphous bulk. Vickers hardness of a HIP'ed powder compact of TiAl was measured under loads of 1 kg and 5 kg.

### 3. Results & Discussion

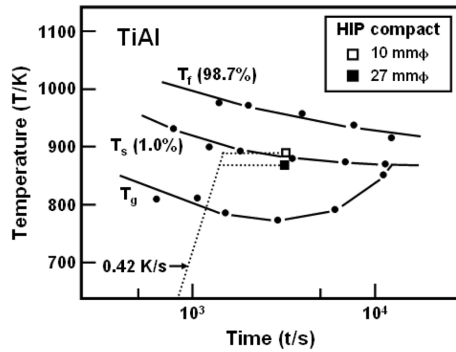
#### 3.1 High quality production and consolidation of amorphous powder

Figure 2(a) shows X-ray diffraction patterns for as attritted (MG) amorphous TiAl powders and aged MG powders. The aged MG TiAl powder shows a broader X-ray peaks relative to as attritted powder. The ageing also leads to a sharp DSC peak of crystallization and a clear endothermic peak of glass transition far below crystallization as shown in Fig. 2(b). An apparent glass temperature ( $T_g$ ), and the temperature at the onset ( $T_o$ ) and the completion ( $T_c$ ) of crystallization can be fairly well determined as depicted in Fig. 2(b). Note that ageing makes it possible to bring a more uniform amorphous structure by releasing strain-induced inhomogeneity in as attritted (MG) TiAl powder.

Figure 3 shows HIP conditions of temperature and pressure as a function of time for consolidating aged amorphous TiAl powders encapsulated in a stainless



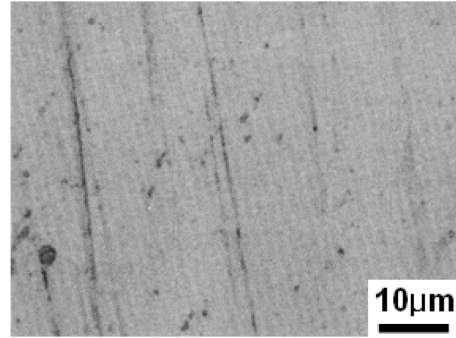
**Fig. 2.** X-ray diffraction patterns (a) and DSC traces (b) for mechanically ground amorphous TiAl powder, aged at ambient temperature, comparing those of compacts, HIP'ed at 853 K and 873 K respectively. (a) includes a XRD pattern of as attrited powders.



**Fig. 3.** HIP conditions of temperature and pressure as a function of time for consolidating high quality amorphous TiAl powders. This figure also describes  $T_s$ ,  $T_f$  and  $T_g$  at various heating rates.

can, 27 mmφ in diameter and in a copper tube, 10 mmφ.

This figure also describes the temperatures at the onset and the completion of crystallization, and the apparent glass temperature at various heating rates, derived from DSC traces at ambient pressure of Fig. 2(b). It can be seen that a broad temperature range,  $T_g < T < T_s$  where viscous flow of amorphous TiAl oughts to occur without precipitation, when using a heating rate above 0.05 K/s (3 K min<sup>-1</sup>). Both amorphous TiAl powders, HIP'ed for 1800 s at 853 K and 873 K between  $T_g$  and  $T_s$  were consolidated to high density compacts retaining amorphous state, characterized by broad X-ray peaks and crystallization

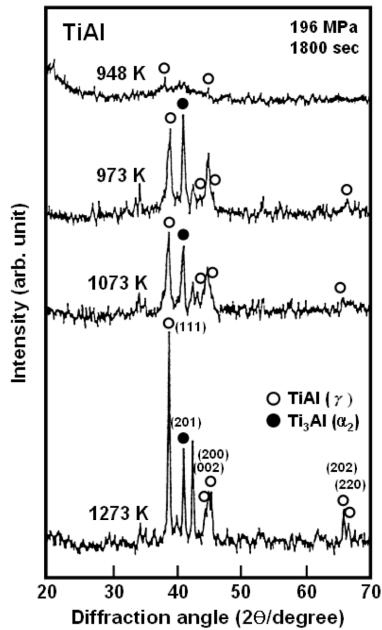


**Fig. 4.** Surface of amorphous TiAl compact, HIP'ed at 873 K for 1800 s by optical microscopy.

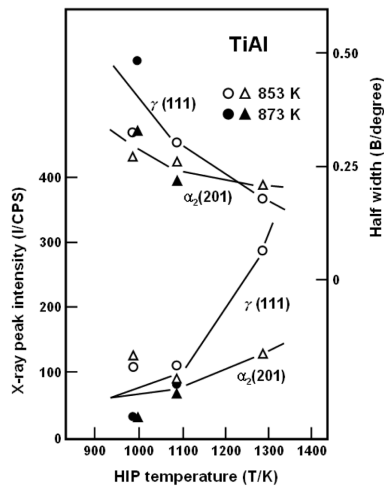
DSC peaks almost identical to those of aged MG powder as shown in Fig. 2(a) and (b). Especially, the amorphous TiAl compact HIP'ed at a higher temperature of 873 K was fully dense below a porosity of 0.1% as seen in Fig. 4. So, the predicted  $T_s$ ,  $T_g$ -time curves for amorphous TiAl without pressure can be used to optimize conditions for consolidation via viscous flow and precipitation.

### 3.2 Formation of titanium aluminide from amorphous phase

Figure 5 shows a change of XRD patterns for the fully dense amorphous TiAl compact, HIP'ed at 873 K with increasing annealing temperature under  $P=196$  MPa using  $\text{CuK}_\alpha$  radiation. Relatively broad X-ray peaks of  $\gamma$ -TiAl and  $\alpha_2$  ( $\text{Ti}_3\text{Al}$ ) appear among an amorphous halo at annealing temperature ( $T_a$ ), 948 K. A complete amorphous-crystalline transformation under  $P=196$  MPa occurs at 973 K, which is higher than  $T_f$  as in Fig. 3. With increasing  $T_a$ , intensity of X-ray peaks of  $\gamma$ -TiAl (111) and  $\alpha_2$  (201) greatly increase, concomitant with decreases in half width of both X-ray peaks as shown in Fig. 6. These dependence strongly suggest a grain coarsening of intermetallic phases of  $\gamma$ -TiAl and  $\alpha_2$  with increasing  $T_a$ . Note that an ordering process in  $\text{L1}_0$  TiAl from amorphous phase (supercooled liquid) can progressively take place by isothermal annealing [15, 16].



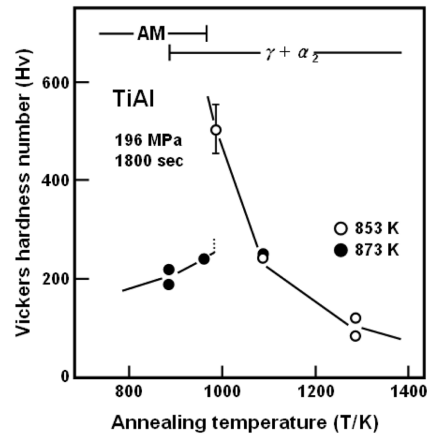
**Fig. 5.** Amorphous-crystalline transformation in XRD patterns of fully dense TiAl compact, HIP'ed at 873 K with increasing annealing temperature from 948-1273 K under a pressure of 196 MPa.



**Fig. 6.** Relationship between HIP temperature and intensity and half width of X-ray peak of  $\gamma$  (111) and  $\alpha_2$  (201) as shown in Fig. 5.

### 3.3 Hardness of amorphous TiAl and titanium aluminide

Figure 7 shows the relationship between vickers hardness and HIP temperature for both fully dense



**Fig. 7.** Relationship between vickers hardness and HIP temperature for HIP compact of amorphous phase and  $\gamma + \alpha_2$ .

compact (873 K) and porous compact (853 K) of amorphous TiAl as shown in Fig. 5 and Fig. 6.

The HIP'ed porous compact above  $T_a=973$  K leads to fully densification. HIP'ed compact of titanium aluminide at  $T_a=973$  K has a high hardness,  $505 \pm 50$  Hv. This high hardness results from a fine structure of  $\gamma$ -TiAl with  $\alpha_2$  as well as a high hardness (886 Hv) of  $Ti_3Al$ -dispersed TiAl (grain size= $0.1 \mu m$ ) by hot pressing MA powder mixture [17-19]. Assuming that a Hall-Petch mechanism works for a hardness for particle-dispersed  $\gamma$ -TiAl, together with 140 Hv for  $50 \mu m$ , a grain diameter of compact ( $T_a=973$  K) is derived as approximately  $0.3 \mu m$ .

## 4. Conclusions

The P/M processing using amorphous TiAl was developed for the synthesis of titanium aluminide. The high quality amorphous TiAl prepared by ageing MG powder can be consolidated to a fully dense compact via viscous flow above the glass temperature by HIP. The HIP'ed compact of titanium aluminide annealed at a relatively low HIP temperature,  $T_a=973$  K shows a high hardness of  $505 \pm 50$  Hv, suggesting strengthening by a sub-micron sized grain structure.

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### References

1. T. Kawabata, T. Kanai and O. Izumi : *Acta Metall.*, **33** (1985) 1355.
2. W. Liang and D. Yang : *Acta Metall. Sinica*, **34** (1998) 597.
3. M. Zupan and K. J. Hemker : *Mater. Sci. & Eng.*, **319** (2001) 810.
4. C. S. Han, *J. Kor. Soc. Heat Treat.*, **18** (2005) 281.
5. C. S. Han and K. W. Koo : *Kor. J. Mater. Res.*, **18** (2008) 51.
6. T. Khan, P. Caron and S. Naka : *High Temperature Aluminides and Intermetallics*, Ed. by S. H. Whang, C. T. Liu, D. P. Pope and J. O. Stiegler, TMS, Warrendahle, (1990) 219.
7. M. V. Nathal : *Ordered Intermetallics-Physical Metallurgy and Mechanical Behaviour*, Ed. by C. T. Liu, R. W. Cahn and G. Sauthoff, NATO ASI Series E, Kluwer Academic Publ., Dordrecht, **213** (1992) 541.
8. S. H. Kim, M. C. Kim, M. H. Oh and D. M. Wee : *J. Kor. Inst. Met. & Mater.*, **39** (2001) 731.
9. R. T. Zheng, Y. G. Zhang, C. Q. Chen and G. A. Cheng : *Mater. Sci. & Eng., A*, **362** (2003) 192.
10. S. Romankov, W. Sha, S. D. Kaloshkin and K. Kaevitser : *Surf. & Coat. Tech.*, **201** (2006) 3235.
11. C. Suryanarayana : *J. Alloys and Comp.*, **509** (2011) S229.
12. K. Fantao, Y. Hongbao and C. Yuyong : *Rare Met. Mater. & Eng.*, **34** (2005) 446.
13. H. Bahmanpour and S. Heshmati-Manesh : *Inter. J. Mod. Phys., B*, **22** (2008) 2933.
14. C. S. Han and J. Y. Nam : *J. Res. Inst. Eng. & Tech.*, **34** (2015) 21.
15. O. N. Senkov, M. L. Övecoglu, N. Srisukhumbowornchai and F. H. Froes : *Nanostructured Mater.*, **10** (1998) 935.
16. H. Sugimoto, K. Ameyama, T. Inaba and M. Tokizane : *J. Jpn. Inst. Met.*, **53** (1989) 628.
17. D. L. Zhang, H. B. Yu and Y. Y. Chen : *Mater. Sci. forum*, **683** (2011) 149.
18. K. P. Rao, Y. V. Prasad and K. Suresh : *Mater. & Design*, **32** (2011) 4874.
19. A. G. Adams, M. N. Rahaman and R. E. Dutton : *Mater. Sci. & Eng. properties, microstructure and processing. A*, **477** (2008) 137.