Annealing Effect on Mechanical Properties of Ti−Al Alloy/Pure Ti Harmonic-Structured Composite by MM/SPS Process

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Abstract. The Ti−Al alloy/pure Ti harmonic-structured composite was produced by mechanical milling and spark plasma sintering process for improvement of low ductility at room temperature of Ti−Al alloy. The harmonic-structured composite with the dispersed area having coarse grained titanium and the network area having fine-grained Ti−48mol%Al alloy demonstrates high strength and high ductility at room temperature. The annealing effect of the microstructure on the mechanical properties in the Ti−Al alloy/pure Ti harmonic-structured composite are investigated. The microstructure of the Ti−Al alloy/pure Ti harmonic-structured composite annealed at 873K, 973 K and 1073 K are maintained the Ti−Al network structure and pure Ti dispersed regions, the average grain size of pure Ti dispersed region is only coarsen by annealing. The harmonic-structured composite annealed at 873K, 973K and 1073 K are maintained the high hardness. The tensile results reveal that the Ti−Al alloy/pure Ti harmonic-structured composite annealed at 873K exhibits high strength and especially high ductility.

1. Introduction

  Ti−Al alloys demonstrate high specific strength in high temperature environment. Thus they are expected as high temperature materials[1], and already applied to a turbine blade of turbocharger. However, Ti−Al alloys demonstrate limited ductility at room temperature because they are composed by intermetallic compounds. Therefore, in order to practical use of Ti−Al alloy as a structural material, it is necessary to ensure enough ductility at room temperature. Previously, improvements in ductility at room temperature environment by addition of a third element has been studied[2-4], but may cause problems in high temperature strength and toughness[5, 6]. As the other method, to improve the mechanical properties of Ti−Al alloy at room temperature without adding a third element, there is the grain refining process. In this method, when the grain size achieves nano order, the strength is improved but the large ductility can not be obtained because the early plastic instability is caused after yielding[7, 8].

  Harmonic-structured materials demonstrate not only superior strength but also enough strain at the same time[9-12]. The harmonic structure consists of fine-grained network and dispersed coarse grain region. The network region with fine grains is atributed to the strength and the dispersed region with coarse grains is atributed to the ductility. In previous study, the harmonic-structured composite with the dispersed area having coarse grained titanium and the network area having fine grained Ti−Al alloy demonstrates high strength and high ductility at room temperature[13]. Furthermore, there is necessary to reveal thermal effects for the mechanical properties of this composite because titanium aluminide alloy is expected as high temperature materials. Therefore, the anealing effect of the microstructure on the mechanical properties in the Ti−Al alloy/pure Ti harmonic-structured composite produced by mechanical milling (MM) and spark plasma sintering (SPS) process are investigated in detail.

2. Experimental Procedure

Powders of titanium alloyed with 48 mol% Al (Ti−48Al) and commercially pure titanium (Ti; 99.9mass% purity) were used in this study. The initial particle sizes of the Ti-48Al and the pure Ti were about 260 and 200 μm, respectively. These powders were mechanically milled using planetary ball mill equipment (P-6; Fritch Co., Ltd.) with an SKD11 vial and SUJ2 steel balls in Ar atmosphere at room temperature. First, to obtain a fine Ti-48Al powder, the MM was performed with a ball-to-powder weight ratio of 3 : 1 at 200 rpm for 54 ks. Next, a mixture of pure Ti powder and Ti−48Al (16.5 vol%) MM powder was mechanically milled with a ball-to-powder weight ratio of 2 : 1 at 200rpm for 36 ks. The composite MM powder containing Ti−48Al and pure Ti was sintered at 1073 K and 50 MPa for 1.8 ks using SPS apparatus (SPS-510L; Sumitomo Coal Mining co., Ltd.). The SPS compacts were annealed in a vacuum (under 10^{-2} Pa) using heat treating equipment (MILA5000UHV ; ULVAC, Inc.) at 873K, 973K and 1073K for 3.6ks. Scanning electron microscopy (SEM)/ energy dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD) were employed to obtain the microstructure of the SPS compact. The mechanical properties of the SPS compacts were evaluated by the Vickers hardness test (VMT-7; Matsuzawa Co., Ltd.) and tensile test (AGS-10kND; Shimadzu Corporation) with a gauge length of 3 mm and an initial strain rate of 5.5 × 10^{-4} s^{-1}.

3. Result and Discussion

3.1 Microstructure of Ti−48Al/Pure Ti Harmonic-Structured Composite

The Ti−48Al powder was refined to about 80.5 μm by MM for 54ks. The fine Ti−48Al powder and the pure Ti powder were mixed with Ti−48Al (16.5 vol%), and mechanically milled at 200 rpm for 36ks to fabricate the Ti−48Al/Ti composite powder. Figs. 1 (a), (b) and (c) show the cross-sectional image of the Ti−48Al/Ti composite MM powder and EDS results for the Al and Ti concentrations, respectively. The composite MM powder shown in Fig. 1(a) exhibits a different contrast between the surface and middle regions, and Fig. 1(b) demonstrates that the surface region has high Al concentration. While, Fig. 1(c) demonstrates that the middle region has high Ti concentration and the surface region has low Ti concentration. Therefore, the surface region in Fig. 1(b) corresponds to Ti−48Al and the middle region in Fig. 1(c) corresponds to pure Ti.
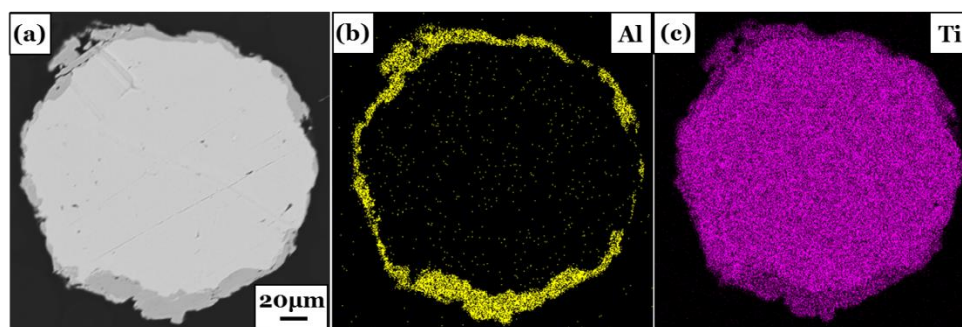

Fig. 1 SEM micrograph and EDS analysis results. (a) shows cross section of the Ti−48Al/Ti composite MM powder, (b) and (c) show elements distribution of Al and Ti, respectively.

The composite MM powder as shown in Fig. 1 was sintered using an SPS apparatus. Fig. 2(a) shows the microstructure of the SPS compact of Ti−48Al/pure Ti composite sintered at 1073 K for 1.8ks. As shown in Fig. 2(a), this composite has a network structure and dispersed region surrounding the network. Thus, the microstructure with a network and dispersed regions is referred to as "harmonic structure". Figs. 2(b) and (c) show EDS results for the Al and Ti concentrations, respectively. Fig. 2(b) demonstrates that the network structure has high Al concentration. While, Fig .2(c) demonstrates that the dispersed region has high Ti

concentration and the network structure has low Ti concentration. Fig. 3(a) shows the XRD result of this composite. This composite consists of Ti, TiAl and Ti_3Al phases. Therefore, the network structure corresponds to TiAl and Ti_3Al, dispersed regions correspond to pure Ti. Figs. 4(a) and (b) show enlargement micrographs of the network and dispersed regions of the harmonic-structured composite, respectively. The network Ti−48Al region has the average grain size of 840 nm as shown in Fig. 4(a), and the dispersed pure Ti region has the average grain size of 21 μm as shown in Fig. 4(b). Thus, the Ti−48Al/Ti harmonic-structured composite consists of a nano grain network region and a coarse grain dispersed region.

 Figure 2(d) shows the microstructure of the Ti−48Al/pure Ti harmonic-structured composite annealed at 873 K for 3.6ks. The harmonic structure has maintained after the heat treatment at 873 K as shown Fig. 2(d). Figs. 2(e) and (f) show EDS results for the Al and Ti concentrations, respectively. As shown in Figs. 2(e) and (f), Al is hardly diffused from network regions to dispersed regions. Fig. 3(b) shows the XRD result of the compact annealed at 873 K. This result indicates that the phase composition is the same as Fig. 3(a), Ti, TiAl and Ti_3Al phases. Therefore, the harmonic-structured composite annealed at 873K consists of the network structure of TiAl and Ti_3Al, and dispersed regions of pure Ti. In addition, the microstructure of 973K and 1073 K compact are similar with the compact annealed at 873 K. Figs. 4(c) and (d) show enlargement micrographs of the network and dispersed regions of the harmonic-structured composite annealed at 873 K, respectively. The network Ti−48Al region has the average grain size of 1 μm as shown in Fig. 4(c), and the dispersed pure Ti region has the average grain size of 31 μm as shown in Fig. 4(d). The average grain size of the dispersed region increases with increasing the annealing temperature. However, the average grain size of network region is hardly changed. This result is attributed to the different grain growth rate between Ti−48Al and pure Ti. Therefore, the harmonic structure is maintained in spite of the annealing at 873 K.
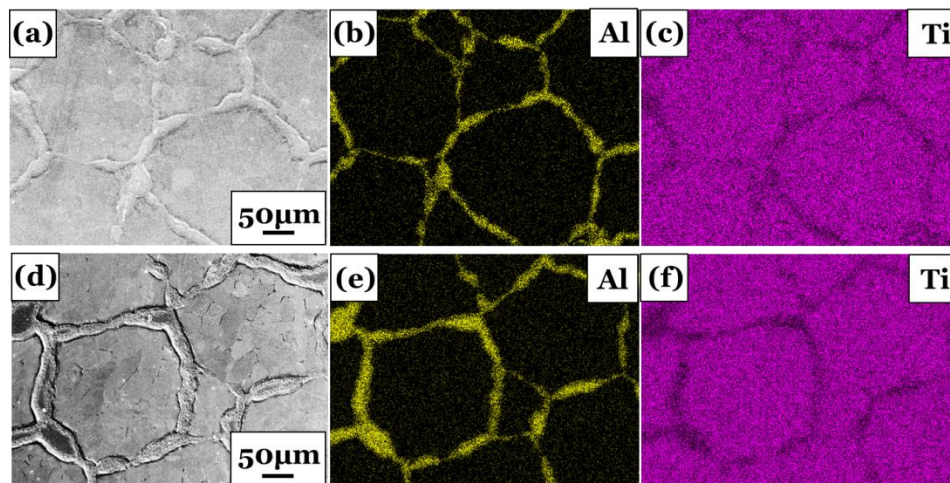

Fig. 2 SEM micrograph and EDS analysis results of the Ti−48Al/Ti harmonic-structured composite. (a) shows microstructure of the as sintered compact, (b) and (c) show elements distribution of Al and Ti, respectively. (d) shows microstructure of the 873 K annealing compact, (e) and (f) show elements distribution of Al and Ti, respectively.
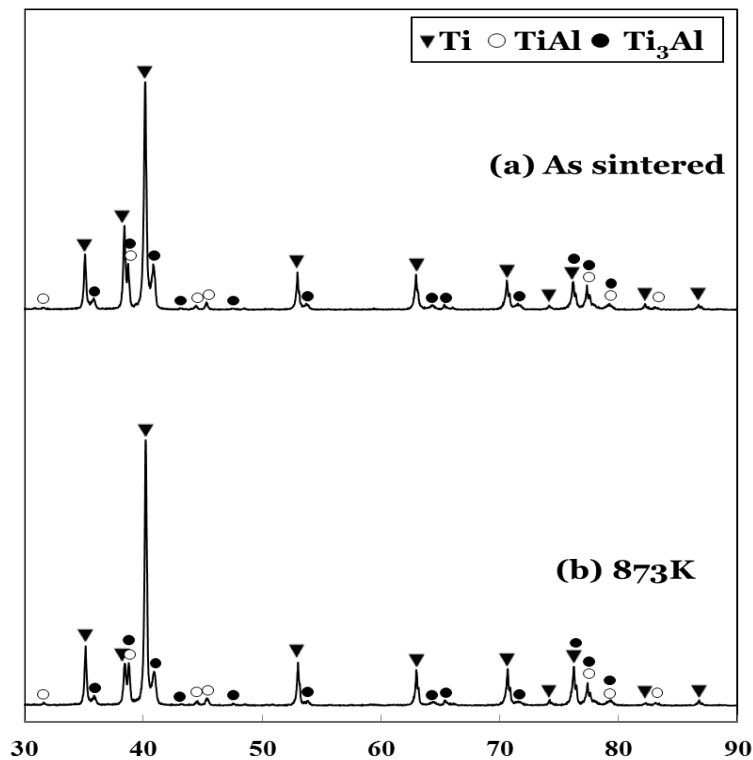
Fig. 3 XRD results of the Ti−48Al/Ti harmonic-structured composite. (a) shows as sintered compact and (b) shows 873 annealing compact, respectively.
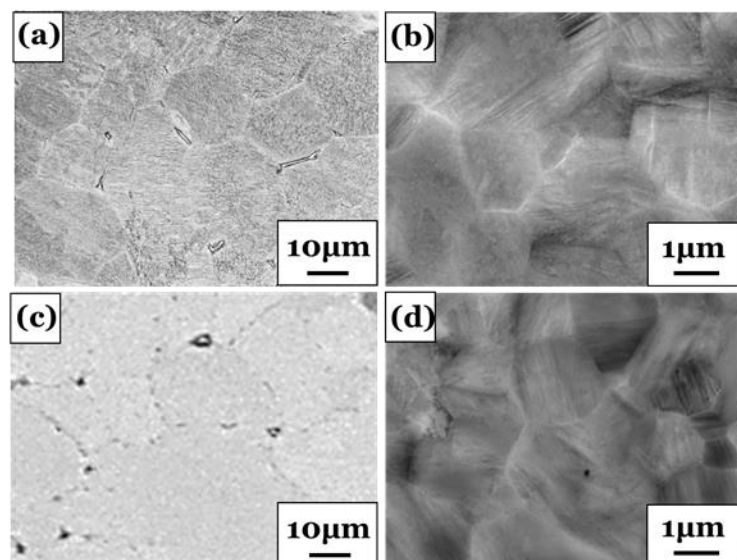

Fig. 4 SEM micrographs of the dispersed regions and the network region. (a) and (b) show the as sintered compact, (c) and (d) show the 873 annealing compact, respectively.

3.2 Mechanical Properties of Ti−48Al/Pure Ti Harmoni-Structured Composite

Figure 5 shows the Vickers hardness test results of the Ti−48Al compact, the pure Ti compact and Ti−48Al/pure Ti harmonic-structured composite before and after annealing. In this case, the temperature before annealing is expressed as 293 K. As the temperature increases, the hardness decrease of the pure Ti compact corresponds to about 60 Hv. Then,

the hardness decrease of the Ti−48Al compact and the harmonic-structured composite correspond to about 14 and 23 Hv, respectively. Especially, the all of hardness of the harmonic-structured composite annealed at 873K, 973K and 1073 K are about 230 Hv. These values are hardly changed. This high hardness is due to the effect of the maintained network structure and its hardly changed grain size. Because the network structure keeps the microstructure from plastically deforming by the indenter, the high hardness is maintained.
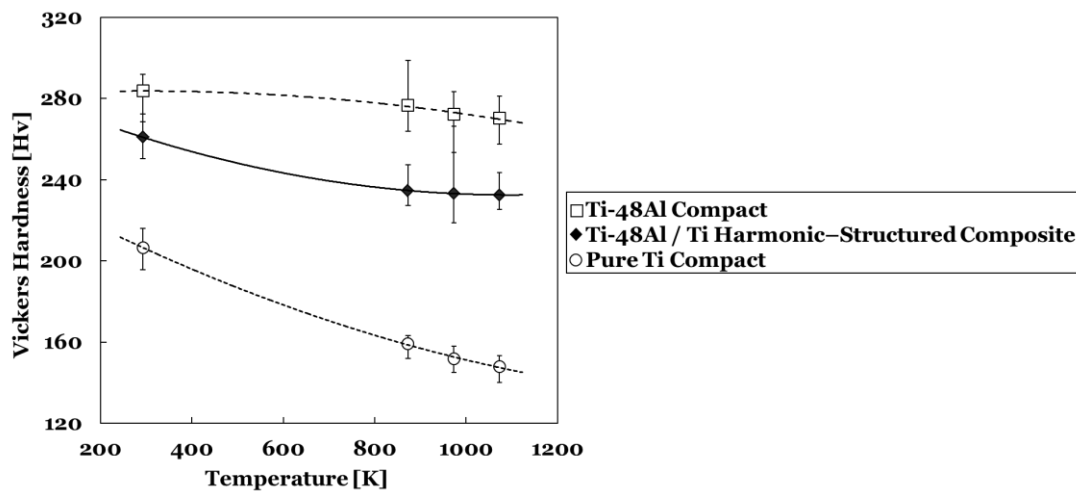

Fig. 5 Vickers hardness of Ti−48Al compact, the pure Ti compact and Ti−48Al/pure Ti harmonic-structured composite before and after annealing, respectively.

   Figure 6 shows nominal stress−strain curves from the tensile test of the Ti−48Al/pure Ti harmonic-structured composite before and after annealing at 873 K and 1073 K. The 0.2 % proof stress and the total elongation of the as-sintered compact are 560 MPa and 0.45 %. These values are higher strength and higher elongation compared with the conventional Ti−48Al compact [14]. On the other hands, the 0.2 % proof stress and the total elongation of the compact annealed at 873 K are 490 MPa and 2.2 %. The strength is lower than the as-sintered compact, but this value is higher than the conventional Ti−48Al compact [14]. Furthermore, the ductility is improved greatly compared with the as-sintered compact. The elongation became about 4.8 times after annealing at 873 K. This drastic improvement of the ductility is due to coarse grain by annealing in dispersed regions. The compact annealed at 1073 K shows intermediate properties. The lower elongation at 1073 K is repeatable and its reason is not clarified yet. A further study is underway to reveal this phenomenon.
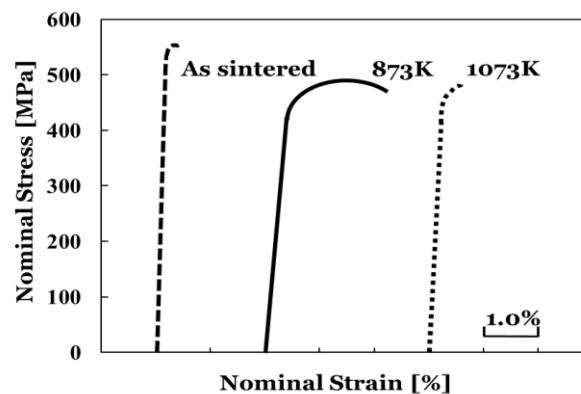

Fig. 6 Nominal stress-strain curves of the Ti−48Al/Ti harmonic-structured composite before and after annealing at 873 K and 1073 K.

4. Conclusion

The anealing effect of the microstructure on the mechanical properties in the Ti–Al alloy/pure Ti harmonic-structured composite fabricated by mechanical milling and spark plasma sintering process are investigated. The Ti–48Al/pure Ti harmonic-structured composite annealed at 873K, 973 K and 1073 K maintain the Ti–Al (TiAl and Ti$_3$Al) network structure and pure Ti dispersed regions. The average grain size of the network region is hardly changed, while the average grain size of pure Ti dispersed region is coarsening by annealing. The hardness of the harmonic-structured composite annealed at 873K, 973K and 1073 K indicates the high hardness of 230 Hv. This high hardness is due to the effect of hardly changed network structure. The 0.2 % proof stress and the total elongation of the Ti–48Al/pure Ti harmonic-structured composite annealed at 873K are 490 MPa and 2.2 %. In comparison with conventional Ti–48Al/pure Ti harmonic-structured composite, the ductility is improved particularly.

Acknowledgement

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