Microstructure and Mechanical Properties of $\text{TiAl}_3/\text{Al}_2\text{O}_3$ *in situ* Composite by Combustion Process

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The microstructure and mechanical properties of $\text{TiAl}_3/\text{Al}_2\text{O}_3$ *in situ* composite by combustion process are investigated in details. Pure Al powder and TiO_2 powder were mechanically milled with a molar ratio of 13 : 3 at 300 rpm for 18 ks. This milling powder's green compact cold-pressed at 200 MPa for 0.3 ks sintered at 923 to 1173 K for 1.8 ks under Ar atmosphere. The microstructure of the sintered compact was identified by XRD, SEM and TEM/EDS. Mechanical properties were evaluated by Vickers hardness and indentation fracture toughness tests. The compacts at less than 973 K consist of a TiAl_3 phase and remained phases of Al and TiO_2 , while the compact at more than 1023 K consist of TiAl_3 matrix, dispersed Al_2O_3 particles and some plate like Ti_3Al phase. The compacts at more than 1023 K have the higher hardness around 800 Hv compared to TiAl_3 single phase compact with 450 Hv. The fracture toughness of $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composite is about two times TiAl_3 single phase compact. This improvement of hardness and toughness is attributed to the effect of the microstructure formation of dispersed Al_2O_3 particles and Ti_3Al plate like phase in the TiAl_3 matrix. [doi:10.2320/matertrans.Y-M2014823]

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1. Introduction

Titanium aluminide alloys are expected as light high temperature materials because of the high specific strength in high temperature environment. In the Ti–Al binary system, there are 5 kinds of intermetallic such as Ti_3Al , TiAl , δ phase, TiAl_2 and TiAl_3 .¹⁾ Especially, Ti_3Al , TiAl and TiAl_3 are expected as high temperature materials. Ti_3Al and TiAl are predominant candidate for the practical use. On the other hand, the mechanical properties of TiAl_3 are not enough for the practical use because of poor toughness and ductility compared to Ti_3Al and TiAl .²⁾ However TiAl_3 is expected as light high temperature materials due to the superior oxidation resistance. It is reported that the plasticity of TiAl_3 is improved by the addition of other elements.^{3,4)} Then, due to improving toughness of TiAl_3 , TiAl_3 composite with fine Al_2O_3 particle is fabricated by the combustion process and its microstructure and mechanical properties of the $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composite are evaluated in detail.

2. Experimental Procedure

Pure Al powder (99.7% purity) and TiO_2 (Anatase) powder (99% purity) were used in this study. The initial particle sizes of the pure Al and TiO_2 powders were approximately 60 and 0.2 μm , respectively. At first, pure Al powder and TiO_2 powder were mechanically milled with a molar ratio of 13 : 3 at 300 rpm for 18 ks to fabricate a composite powder. Mechanical milling (MM) was used a planetary ball mill (Yangzhou Nuoya Machinery Co., Ltd., NQM-4) at room temperature under argon atmosphere. Next, the composite powder was formed by cold compression at 200 MPa for 0.3 ks. These green compacts were sintered at 923, 973, 1023 and 1173 K for 1.8 ks under Ar atmosphere and the $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composite compacts were fabricated. The microstructure was identified by XRD (Rigaku Corporation, Smart

Lab), SEM (JEOL Ltd., JSM7001FD) and TEM/EDS (JEOL Ltd., JEM2100F). The mechanical properties were evaluated by Vickers hardness test (Shimadzu Corporation, HMV-1) at 245.2 mN for 15 s and indentation fracture toughness test at 4.905 N for 15 s.

3. Results and Discussions

Figure 1 shows XRD results for the compact sintered at 923 to 1173 K. The compact at 923 and 973 K consists of a TiAl_3 phase and remained phases of Al and TiO_2 , while the compact at 1023 and 1173 K consists of TiAl_3 and Al_2O_3 .

Fig. 1 XRD results for the compacts sintered at 923 to 1173 K.

Fig. 2 SEM back scattered images of the compacts sintered at 923 K (a), 973 K (b), 1023 K (c) and 1173 K (d).

These TiAl_3 and Al_2O_3 phase are formed by the combustion process.^{5,6} The sintering at more than 1023 K is required for the fabrication of the $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composite.

Figure 2 shows SEM back scattered images of the compact sintered at 923 (a), 973 K (b), 1023 K (c) and 1173 K (d). Figures 2(a) and 2(b) show the remained white TiO_2 particle

Fig. 3 (a) TEM image of the compact sintered at 1023 K. (b) Selected area diffraction pattern from an elongated grain shown in (a).

with a size of about less than $1\ \mu\text{m}$ in the matrix consisting of TiAl_3 with the light gray region and Al with the dark gray region. On the other hand, Figs. 2(c) and 2(d) show the TiAl_3 matrix with the dispersed fine Al_2O_3 particles. Note that plate like phase with a white contrast is also observed in the $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composites. Figure 3 shows a TEM micrograph (a) and a selected area diffraction pattern (SADP) (b) of this plate like phase. This plate like phase lies in TiAl_3 matrix and impinges on an Al_2O_3 particle as shown in Figs. 2(c) and 2(d) and Fig. 3(a). The SADP of Fig. 3(b) reveals that a crystal structure of this phase corresponds to D0_{19} structure. The EDS results of TEM reveal that the average composition of this phase demonstrates Ti : Al of 75.1 : 24.9. Therefore, this plate like phase corresponds to Ti_3Al phase. The formation mechanism of Ti_3Al is not clarified yet and a further study will make clear this formation. The TiAl_3 composites with fine dispersed Al_2O_3 particles and the plate like Ti_3Al phase are fabricated by the sintering at more than 1023 K.

Figure 4 shows the result of the Vickers hardness test of the compact sintered at 923, 973, 1023 and 1173 K. The compacts at less than 973 K show the low hardness, while the compact at more than 1023 K is performed the higher hardness around 800 Hv. This hardness value is very high compared to the hardness (450 Hv) of TiAl_3 single phase as shown in Fig. 4. This improvement of hardness is attributed to the effect of fine dispersed Al_2O_3 in the TiAl_3 matrix.

Fig. 4 Vickers hardness results for the compacts sintered at 923 K (a), 973 K (b), 1023 K (c) and 1173 K (d).

Fig. 5 Fracture toughness results for TiAl_3 single phase and $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composite.

Figure 5 shows the results of the indentation fracture toughness for $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composite and TiAl_3 single phase. $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composite demonstrates approximately twice toughness compared to TiAl_3 single phase. Figure 6 shows SEM micrograph of a corner of the indentation in $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composite. The white dotted line shows a trace of the Vickers indentation. The crack produced by the Vickers indentation is obstructed by the fine Al_2O_3 particle and it is clear that the Al_2O_3 particle has a role of the obstacle for the propagation of the crack. Furthermore, the Ti_3Al plate like phase is deformed by the Vickers indentation as shown by arrows in Fig. 6. Therefore, the formation of Ti_3Al phase can be expected to improve the ductility of TiAl_3 matrix.

F. Wagner *et al.* reported that $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composite produced by gas-pressure infiltration and squeeze casting demonstrated the fracture toughness of more than $8.0 \text{ MPa}\cdot\text{m}^{1/2}$.⁷⁾ However, this composite consists of nearly 60 vol% Al_2O_3 , 35 vol% TiAl_3 and 5 vol% Al and has Al_2O_3 matrix with high toughness. The present studies composite consists of TiAl_3 matrix and dispersed fine particle Al_2O_3 and plate like Ti_3Al phase as shown in Figs. 2(c) and 2(d). Therefore, the $\text{TiAl}_3/\text{Al}_2\text{O}_3$ matrix composite demonstrates higher toughness than the present studies composite. On the other hand, C. W. Hsu and C. G. Chao reported that TiAl_3 matrix/ Al_2O_3 particle composite produced by squeeze casting demonstrated the Vickers hardness of approximately 1000 HV.⁸⁾ The present studies composite produced by powder metallurgy process show a relative density of 87%. The low hardness compared to the composite by squeeze casting is attributed to low relative density. This suggests that

Fig. 6 SEM micrograph of a corner of the Vickers indentation in $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composite.

the mechanical properties can be improved by changing the sintering process. The present studies composite consists of TiAl_3 matrix and dispersed fine particle Al_2O_3 and plate like Ti_3Al phase as shown in Figs. 2(c) and 2(d) so that this composite is greatly expected on the improvement of mechanical properties at the elevated temperature.

4. Conclusion

TiAl_3 composites containing fine dispersed Al_2O_3 particle were fabricated via mechanical milling (MM) followed by conventional sintering. The compacts at less than 973 K consist of a TiAl_3 phase and remained phases of Al and TiO_2 , while the compacts at more than 1023 K consist of TiAl_3 and Al_2O_3 . The microstructure of the $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composite sintered at more than 1023 K consists of the TiAl_3 matrix, the dispersed fine Al_2O_3 particles and some Ti_3Al phase. The hardness of the $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composite sintered at more than 1023 K is very high compared to TiAl_3 single phase material. The fracture toughness of the $\text{TiAl}_3/\text{Al}_2\text{O}_3$ composite sintered at more than 1023 K is about twice compared to TiAl_3 single phase material.

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REFERENCES

- 1) J. C. Schuster and M. Palm: *JPEDAV* **27** (2006) 255–277.
- 2) Y. V. Milman, D. B. Miracle, S. I. Chugunova, I. V. Voskoboinik, N. P. Korzhova, T. N. Legkaya and Y. N. Podrezov: *Intermetallics* **9** (2001) 839–845.
- 3) S. Zhang, D. E. Mikkola and W. M. Miligan: *Scr. Met.* **27** (1992) 1073–1077.
- 4) H. Mabuchi, K. Hirukawa and Y. Nakayama: *Scr. Met.* **23** (1989) 1761–1765.
- 5) N. J. Welham: *Intermetallics* **6** (1998) 363–368.
- 6) D. Y. Ying, D. L. Zhang and M. Newby: *Met. Mater. Trans. A* **35** (2004) 2115–2125.
- 7) F. Wagner, D. E. Garcia, A. Krupp and N. Claussen: *J. Eur. Ceram. Soc.* **19** (1999) 2449–2453.
- 8) C. W. Hsu and C. G. Chao: *Met. Mater. Trans. B* **33** (2002) 31–40.