Fabrication of TiB$_2$–Fe–Al Cermet Alloys Synthesized by Pulsed Current Process

Keizo Kobayashi and Kimihiro Ozaki

Materials Research Institute for Sustainable Development, National Institute of Advanced Industrial Science and Technology, 2266-98 Anagahora, Shimoshidami, Moriyama, Nagoya 463-8560, Japan

Please note that the full text of the article is provided and no further action is required for a plain text representation.
3. Experimental Results and Discussion

3.1 Characterization of TiB$_2$–Fe–Al powders

Respective microstructures of TiB$_2$ powder and Fe powder, which were starting materials for MA, are shown in Figs. 1(a) and (b). The Fe powder that was fabricated by atomization processing has about five times the particle size of TiB$_2$ powder. These powders change into fine powders by milling for 180 ks, as shown in Fig. 1(c). The MA powder that was collected by milling for 180 ks has 95% or more of the starting materials’ weight. In the obtained MA powder, contamination from the vessel and the balls during MA was not detected by fluorescent X-ray analysis. The XRD pattern of the mechanically alloyed TiB$_2$–17.7 mass%Fe powder is shown in Fig. 2. The MA powder consists only of TiB$_2$ phase and Fe phase; neither Fe nor TiB$_2$ reacted during milling. Because the peaks of Fe are broadened during milling, it is expected that the Fe powder became fine by crushing in the MA.

Using a mortar, TiB$_2$–20 mass%Fe$_3$Al and TiB$_2$–24 mass%FeAl powder were prepared respectively by mixing of the obtained MA powder and Al powder. The DTA curves of these mixed powders are shown in Fig. 3. They reveal an exothermic reaction at 800 K (it was described in Fig. 3 as ‘A’). This exothermic reaction was observed even by heating only TiB$_2$ phase and Fe phase; neither Fe nor TiB$_2$ reacted during milling. Because the peaks of Fe are broadened during milling, it is expected that the Fe powder became fine by crushing in the MA. Using a mortar, TiB$_2$–20 mass%Fe$_3$Al and TiB$_2$–24 mass%FeAl powder were prepared respectively by mixing of the obtained MA powder and Al powder. The DTA curves of these mixed powders are shown in Fig. 3. They reveal an exothermic reaction at 800 K (it was described in Fig. 3 as ‘A’). This exothermic reaction was observed even by heating only TiB$_2$ powder; it accompanied an increase in the powder weight. Therefore, the reaction is expected to be an oxidation reaction of TiB$_2$. The endothermic reaction of Al melt at 933 K is visible in the DTA curve of TiB$_2$–24 mass%FeAl (shown in Fig. 3 as ‘B’). The self-propagating high temperature synthesis between Fe and Al in TiB$_2$–20 mass%FeAl would occur after Al melting. In TiB$_2$–20 mass%Fe$_3$Al powder, because only a little Al powder was added, the endothermic reaction of Al melt and self-propagating high-temperature synthesis of Fe–Al intermetallic compound were observed only slightly.

3.2 Characterization of TiB$_2$–Fe–Al sintered by pulsed current sintering

Under 70 MPa pressure, TiB$_2$–20 mass%Fe$_3$Al and TiB$_2$–24 mass%FeAl were consolidated using PCS. Figure 4 shows the relation of the amount of shrinkage to the die temperature. After it had expanded gradually to 1200 K, shrinkage was observed in TiB$_2$–20 mass%Fe$_3$Al. On the other hand, considerable shrinkage was observed at ca. 900 K in TiB$_2$–24 mass%FeAl. It is expected that densification by generating molten Al promotes shrinkage. In addition, densification in
1200 K or more is advanced because the compacting pressure is increased. The XRD pattern of TiB$_2$–20 mass%Fe$_3$Al sintered at 1423 K is shown in Fig. 5(a). This sintered body mainly comprises TiB$_2$ and various Fe–Al intermetallic compounds, such as Fe$_2$Al$_5$, Fe$_3$Al. In short-term sintering processes like PCS, it is probably difficult to produce a binder phase Fe$_2$Al single phase while sintering. Figure 5(b) shows the XRD pattern of TiB$_2$–24 mass%FeAl sintered at 1423 K. The sintered body consists of TiB$_2$ and FeAl mainly. Because the reaction Fe + Al = FeAl is exothermic ($\Delta H = -51$ kJ/mol), the combustion reaction between Fe powder and molten Al occurs continuously in the sintered body. Therefore, the FeAl particle size in the sintered body depends on the Fe powder particle size. A homogeneous TiB$_2$–24 mass%FeAl compact is probably obtained as a result of a homogeneous reaction under a pressure of 70 MPa in the PCS process. The density of the sintered TiB$_2$–24 mass%FeAl was 88% of the theoretical density. It is necessary for densification of the TiB$_2$–24 mass%FeAl to sinter it at a high temperature under high pressure using pulsed-current sintering.

Microstructures of sintered TiB$_2$–20 mass%Fe$_3$Al and TiB$_2$–24 mass%FeAl after Vickers hardness test are shown in Fig. 6. Pores remained in each sintered compact. The respective Vickers hardness values measured using the load of 9.8 N were 1930 and 1430 Hv. In TiB$_2$–20 mass%Fe$_3$Al, cracks were apparent surrounding the Vickers indentation. This probably occurs because the binder phase contains brittle Fe–Al intermetallic compounds aside from Fe$_3$Al. In TiB$_2$–24 mass%FeAl, the amount of FeAl binder phase is large; cracks were not observed around the Vickers indentation.

The TiB$_2$–20 mass%Fe$_3$Al and TiB$_2$–24 mass%FeAl sintered bodies were heated in air; then their weight changes were measured with TG. For comparison, TiB$_2$–17.7 mass%Fe synthesized by MA was sintered by PCS and measured. The obtained result is shown in Fig. 7. As for TiB$_2$–17.7 mass%Fe, the weight was increased rapidly by oxidation at 800 K or higher temperatures. However, the weight has changed only slightly at 1200 K for composite materials of which the binder phase is a Fe–Al intermetallic compound. The reason for this slight change is that excellent binder phase at oxidation resistance envelops the TiB$_2$ particle. The effect of the amount of Al in the binder phase on the oxidation resistance was not observed. These TiB$_2$–20 mass%Fe$_3$Al and TiB$_2$–24 mass%FeAl are appropriate materials for cutting tools and dies used in air at high temperatures.

4. Conclusion

Mechanical alloying for 180 ks of TiB$_2$ and Fe powders synthesized TiB$_2$–17.7 mass%Fe powder. That MA powder and Al powder were mixed to produce TiB$_2$–20 mass%Fe$_3$Al and TiB$_2$–24 mass%FeAl. These mixture powders were consolidated with pulsed-current sintering for a short time. The reaction path during the pulsed current sintering and the microstructures of TiB$_2$–20 mass%Fe$_3$Al and TiB$_2$–24 mass%FeAl were investigated. Results indicate the following:

(1) The TiB$_2$–17.7 mass%Fe mixture of TiB$_2$ powder and fine Fe powder is synthesized by mechanically alloying for 180 ks. Apparently, no reaction occurs between TiB$_2$ and Fe during mechanically alloying.
When $\text{TiB}_2$–20 mass%$\text{Fe}_3\text{Al}$, a mixture of mechanically alloyed $\text{TiB}_2$–17.7 mass%$\text{Fe}$ powder and $\text{Al}$ powder, is consolidated using pulsed-current sintering at 1473 K under 70 MPa pressure, it consists of $\text{TiB}_2$ and some $\text{Fe}$–$\text{Al}$ intermetallic compounds. Therefore, the obtained sintered compact is brittle, but it shows high hardness.

When $\text{TiB}_2$–24 mass%$\text{FeAl}$, which mixes mechanically alloyed $\text{TiB}_2$–17.7 mass%$\text{Fe}$ powder and $\text{Al}$ powder, is consolidated using pulsed-current sintering, a self-propagating high temperature synthesis is observed between $\text{Fe}$ and molten $\text{Al}$. The obtained sintered compact mainly consists of $\text{TiB}_2$ phase and $\text{FeAl}$ phase. It is a composite materials with hardness of 1430 Hv with toughness. In a sintering process for a short time such as pulsed current sintering, it is easy to fabricate $\text{TiB}_2$ based cermet alloy with an $\text{FeAl}$ binder phase.

The oxidation resistance of the sintered $\text{TiB}_2$–20 mass%$\text{Fe}_3\text{Al}$ and $\text{TiB}_2$–24 mass%$\text{FeAl}$ in 700 K or more is far superior to that of sintered $\text{TiB}_2$–17.7 mass%$\text{Fe}$. The $\text{TiB}_2$–$\text{Fe}$–$\text{Al}$ cermet alloy is probably useful for high-temperature applications to prevent oxidation in air.

REFERENCES