The Effect of Al₂O₃ Addition on the **Milling Behavior of NiAl Powder**

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The milling behavior of nickel aluminide, NiAl, powder in the presence of a fine Al₂O₃ powder was inves**tigated in the present study. The milling was carried out in an attrition mill. The size and shape of NiAl** particles were not changed after milling while only NiAl powder was milled. When fine Al₂O₃ powder was added to the NiAl powder, the Al₂O₃ particles attached to the surface of NiAl particles during milling. As **a consequence, the size of NiAl particles was reduced after milling. The shape of NiAl particles also** changed. The presence of fine $A1_2O_3$ particles enhanced the milling efficiency. The $A1_2O_3$ particles on the **surface of NiAl powder can be removed by washing repeatedly in an ultrasonic bath.**

Keywords Al2O3, milling, NiAl, particle size distribution

1. Introduction

Nickel aluminide, NiAl, is a potential high-temperature structural material. The melting point of NiAl is 1640 °C, which is approximately 300 °C higher than that of nickel-base superalloys. The thermal conductivity is three to eight times that of nickel-base superalloys (Ref 1, 2). Furthermore, the density of NiAl is lower than that of superalloys. The oxidation resistance and corrosion resistance are also better than those of superalloys. Nevertheless, the toughness of NiAl at room temperature and the tensile strength at high temperature are low. The applications of NiAl for structural components are thus limited. This limitation can be overcome by adding ceramic strengthening agents (Ref 3, 4). For example, the tensile strength of NiAl between 700 and 900 °C is increased by adding TiB₂ and Al_2O_3 particulates (Ref 3). A recent study also suggested that both strength and toughness of NiAl are enhanced by adding partially stabilized zirconia (PSZ) particles (Ref 4).

To prepare a composite with superb mechanical properties, the components of the composite should be thoroughly mixed. However, the size of metallic powder is usually in the range of a few micrometers, while the size of the ceramic particles used to strengthen the matrix are in the submicron range. Furthermore, the physical characteristics of NiAl are very different from those of ceramics. For example, the density of NiAl is higher than that of Al_2O_3 . Thus, to achieve uniform mixing is a difficult task. However, studies on the milling behavior of ceramic/metal powder mixtures are limited. A recent study showed that the size of ceramic particles has a dramatic effect on the milling efficiency of metallic particles (Ref 5). The study suggested that the size of aluminum particles $(20 \text{ to } 200 \mu \text{m in})$ diameter) was not changed by adding fine Al_2O_3 particles (1) µm). However, the size of aluminum particles was significantly

reduced by adding coarse Al_2O_3 particles (13 µm). Dense Al_2O_3/Al composites can thus be prepared by pressureless sintering the fine powders. In the present study, the milling behavior of $\text{Al}_2\text{O}_3/\text{NiAl}$ powder mixtures was investigated.

2. Experimental Procedure

A nickel aluminide (NiAl, Xform Inc., New York, NY) powder and 0 to 100 vol% alumina (TM-DR, Taimei Chem. Co. Ltd., Tokyo, Japan) were combined and attrition milled in ethyl alcohol. The rotation speed of the shaft was 300 rpm, the milling time was 1 h, the grinding media used was zirconia balls, and the diameter of the zirconia balls was 5 mm. Table 1 shows the composition of NiAl reported by the manufacturer. The particle size and particle distribution were determined using a laser particle size analyzer (LS 230, Coulter Co.). A dispersant (Darvan C, R.T. Vanderbilt Co., Inc.) was used to disperse the powders in water. The morphology of the powders was observed via scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The true morphology of the NiAl particles in the powder mixtures was revealed by removing the Al_2O_3 powder by washing it repeatedly in an ultrasonic bath.

3. Results and Discussion

Figures 1(a) and (b) show the morphology of the as-received Al_2O_3 and NiAl powders. The shape of the as-received NiAl particles is angular. The aspect ratio of the particles is roughly unity. Figures 2(a) and (b) show the particle size distribution of the as-received Al_2O_3 and NiAl powders. The alumina powder shows a bimodal size distribution. The larger particles are made

Table 1 The composition of the NiAl powder

	Ni, Al, Fe, Cu, Cr, Si, Ti, C, S, N, O, H, $wt\%$ wt% wt% wt% wt% wt% wt% ppm ppm ppm ppm ppm ppm										
	68.23 31.54 0.05 < 0.01 0.01 < 0.01 < 0.01 249 25 28 1327 29										
Values are reported by the manufacturer.											

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up of aggregates of smaller particles, which are unable to be dispersed in water. The median size of $A₁O₃$ and NiAl powders is 0.2 and 5.9 µm, respectively. Figure 3 shows the particle size distribution of the NiAl powder after milling. The particle size distribution of the powders is not altered as NiAl powder is milled alone. Figure 4 shows the morphology of the NiAl particles after milling. The shape of the NiAl particles is also unchanged.

Figure 5 shows the particle size distribution of the $Al_2O_3/NiAl$ powder mixtures. Because the size of NiAl powder is roughly one order of magnitude larger than that of the Al_2O_3 powder, the peak above 1 µm in Fig. 5 can be attributed mainly to NiAl powder. The peak below 1 µm is attributed mainly to Al_2O_3 powder. As shown in Fig. 3, the particle size distribution of NiAl powder does not change after milling. However, the

100 nm

Fig. 1 Morphology of the as-received (a) NiAl and (b) Al_2O_3 powders

NiAl particles larger than 10 μ m disappeared when 10 vol% of Al_2O_3 particles were added (Fig. 5). The fraction of the particles smaller than 1 µm increased as a result. The size of NiAl particles was reduced further by adding 20 vol% Al_2O_3 . In SEM observation, NiAl particles were covered with a layer of fine Al_2O_3 particles after attrition milling for one hour (Fig. 6).

The milling operation produces both compressive and shear forces on particles (Ref 6). The hardness of Al_2O_3 is much greater than that of NiAl. Fine alumina particles can thus squeeze into NiAl particles by the compressive force. The compressive and shear forces are amplified through the $Al_2O_3/NiAl$ contacts. The NiAl particles can thus be deformed into flakes (Fig. 7). No Al_2O_3 particles were found on the surface of the NiAl flakes. This indicates that the Al_2O_3 particles can be easily removed by washing repeatedly in an ultrasonic bath. The NiAl flakes can further be fractured into smaller particles during milling. Therefore, as fine Al_2O_3 particles are added, not only the shape but also the size of the resultant NiAl milled powder is changed. Therefore, the presence of the fine particles enhances the milling efficiency considerably. However, the ductility of the metallic particles may play an important role on the milling behavior of ceramic/metal powder mixtures. For exam-

Fig. 2 Particle size distribution of the as-received powders

Fig. 3 Particle size distribution of the NiAl powder after attrition milling for 1 h. The particle size distribution of the as-received powder is also shown for comparison.

(b)

ple, the presence of a fine fraction of $A1_2O_3$ powder is not able to reduce the size of aluminum powder (Ref 5). This behavior may be due to the ductility of aluminum being higher than that of NiAl. However, the shape of the metallic particles can still be altered by milling the metal powder with fine ceramic powder (Ref 5, 7).

The milling efficiency of NiAl is increased by adding up to 20 vol% Al_2O_3 (Fig. 5). The efficiency is not improved any further by adding more than 20 vol% Al_2O_3 (Fig. 8). The amount of Al_2O_3 particles adhering to the surface of NiAl particles is limited by the surface area of the NiAl powder. The surface area of a NiAl particle, S_{NiAl} , can be roughly estimated as:

$$
S_{\text{NiAl}} = 4\pi (d_{\text{NiAl}}/2)^2
$$
 (Eq 1)

where d_{NiAl} is the particle size of NiAl. The effective surface area that can be covered by an Al_2O_3 particle, $S_{\text{Al}_2\text{O}_3}$, is:

Fig. 4 Morphology of the NiAl particles after attrition milling for 1 h

Fig. 6 Morphology of the $\text{Al}_2\text{O}_3/\text{NiAl}$ powder mixtures after milling. Al_2O_3 , 50 vol%, was added into the NiAl powder.

$$
S_{\text{Al}_2\text{O}_3} = \pi (d_{\text{Al}_2\text{O}_3}/2)^2
$$
 (Eq 2)

where $d_{\text{Al}_2\text{O}_3}$ is the particle size of Al₂O₃. The values of $d_{\text{Al}_2\text{O}_3}$ and d_{NiAl} are 0.2 and 5.9 µm, respectively. From Eq 1 and 2, 13 vol% Al_2O_3 particles are needed to form a single layer on the surface of the NiAl particles. As a result, 10 vol% Al_2O_3 particles are not enough to cover all the surface of NiAl particles. From the results of this study, 20 vol% Al_2O_3 particles can cover all the surface of the NiAl particles. Therefore, the milling efficiency of the 20% Al₂O₃/80%NiAl powder mixtures is higher than that of 10% Al₂O₃/90%NiAl powder mixtures. As the surface of NiAl particles is fully covered by one layer of fine Al_2O_3 particles, further addition of Al_2O_3 particles can no longer contact the surface of NiAl particles. The milling efficiency is thus not increased any further by adding more than 20 vol% Al_2O_3 .

Fig. 5 Particle size distribution of the $A1_2O_3/NiA1$ powder mixtures after milling. Al_2O_3 , 0, 10, or 20 vol%, was added into the NiAl powder.

Fig. 7 Morphology of NiAl flakes after attrition milling. 80% Al_2O_3 was added into the NiAl powder before milling. The Al_2O_3 particles were removed by repeated washing with water.

Fig. 8 Particle size distribution of the $\text{Al}_2\text{O}_3/\text{NiAl}$ powder mixtures after milling. Al_2O_3 , 20, 40, 60, or 80 vol%, was added into the powder mixtures.

4. Conclusions

In the present study, the milling behavior of NiAl powder in the presence of a fine Al_2O_3 powder is investigated. During milling, the milling media applies compressive and shear forces on the powder. The particle size distribution of NiAl powder is not changed when the NiAl powder is milled with Al_2O_3 . When the NiAl powder is milled with fine Al_2O_3 powder, the Al_2O_3 particles attach to the surface of the NiAl particles during milling. The Al_2O_3 particles act as "nails," which amplify the milling forces at the contact points. Therefore, the NiAl particles are deformed into NiAl flakes. Coarse NiAl flakes can be further fractured into smaller particles. For the present system, roughly 20 vol% Al_2O_3 particles can cover the surface of NiAl particles. Volume fractions of Al_2O_3 greater than 20% do not affect milling efficiency.

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