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Spark-Plasma Sintering and Mechanical Property of Mechanically Alloyed NiAl Powder Compact and Ball-Milled (Ni+Al) Mixed Powder Compact

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Summary

Mechanically-alloyed NiAl powder and (Ni+Al) powder mixture prepared by ball-milling were sintered by Spark-Plasma Sintering(SPS) process. Densification behaviour and mechanical property were determined from the experimental results and analysis such as changes in linear shrinkage, shrinkage rate, microstructure, and phase during sintering process, Vicker's hardness and transverse rupture strength tests.

Densification mechanisms for MA-NiAl powder compact and (Ni+Al) powder mixture were different from each other. While the former showed a rapid increase in densification rate only at higher temperature region of 800-900°C, the latter revealed firstly a rapid increase in densification rate even at low temperature of 300°C and a subsequent increase up to 500°C. Densities of both powder compact (MA and mixture) sintered at 1150°C for 5min. were 98 and above 99%, respectively. Sintered bodies were composed mainly of NiAl phase with Ni_3Al as secondary phase for both powders. Sintered body of MA-NiAl powder showed a very fine grain structure. Crystallite size determined by XRD result and the Sherrer's equation was approximately 80nm. Vicker's hardness for the sintered bodies of (Ni+Al) powder mixture and MA-NiAl powder were 410±12 H_v and 555±10 H_v, respectively, whereas TRS values 1097±48 MPa and 1393±75 MPa.

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Keywords:
NiAl, spark-plasma sintering, mechanical alloying, (Ni+Al) powder mixture, Vicker's hardness, TRS

1. Introduction

Recently, many of research works on β-NiAl had been performed with an expectation that it could be used as a high-temperature structural material due to its excellent properties such as high melting temperature, high specific strength, good oxidation and creep resistance, etc [1-5]. In addition to the properties mentioned above, NiAl shows a thermoelastic transformation behavior at relatively higher temperature than usually known shape-memory alloy such as TiNi. However, the poor workability due to high brittleness at room temperature hinders a wider application of NiAl. For this reason many efforts for improving the ductility were already given using microalloying, microstructure control and composite technology, etc. Especially after it was known that the ductility could be remarkably improved by decreasing grain size under a certain critical size, a great deal of works have been made to obtain a nanostructued NiAl through various methods like inert gas condensation, electro-deposition, sputtering, re-crystallization of amorphous materials, and mechanical alloying. Among these the mechanical alloying is known to be one of the simplest methods to produce homogeneous and fine nanostructured alloy powders on relatively large scale.
Many of recent reports on SPS (Spark-Plasma Sintering) show that remarkable improvements in material properties can be made by this processing [6-8]. It is because a consolidation at lower temperature for shorter period is possible in comparison with other conventional sintering methods. SPS is a similar process with a combination of conventional Electric-Current Sintering and Hot-Pressing. But it is different from them in the sense that the electric current is applied to a specimen in a form of pulse and the specimen is heated both by resistance heating of the specimen itself and the conductive die mold (usually graphite). Figure 1 is a schematic configuration of SPS facility. The reported material systems involve most of hard-to-sinter materials such as intermetallic compounds [9] and refractory [10-11], high-performance engineering ceramics and their composites [12-16]. In this study a production of nanograined NiAl powder and its full densification by SPS was aimed. For comparison a powder mixture of (Ni+Al) was also produced by ball-milling and sintered by SPS, too. The densification behavior,

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microstructure and mechanical properties were compared each other.

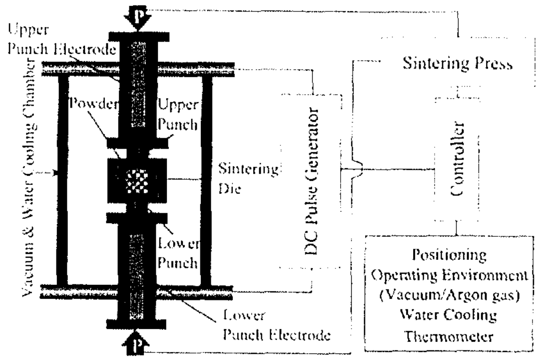

Fig. 1 Schematic configuration of SPS facility.

2. Experimental Procedures

Al and Ni powders with a purity of 99.9% were used as starting materials. Average particle sizes were 45 and 5㎛, respectively (Fig. 2). Al and Ni powders were weighed to have a final composition of Ni-36 at.% Al. Mechanical alloying was performed at 600rpm for 20h in a high-energy attrition mill, Simoloyer (CM01-01, Zoz Co., Germany), with stainless steel ball media. The weight ratio of powder to ball media was 1:40. Stearic acid ($C_{17}H_{35}COOH$) was used as process control agent. (Ni+Al) powder mixture was prepared by ball-milling with 120 rpm for 1 and 10h.
Sintering was carried out in a pulse-electric-current-sintering facility under the following conditions: Sintering temperature of 1150℃, heating rate of 10℃/min, holding for 5min at sintering temperature, and applying pressure of 50Mpa. Sintered density was measured by an electronic densimeter (model SD –120, Mirage Co., Japan). Data of shrinkage along the pressure axis, temperature, and electrical power input was automatically stored through a data-acquisition system into PC. Stored data was further processed in a form of densification rate (% relative density/sec.) vs. temperature. Crystallite size of starting powders and sintered bodies were measured by X-ray line broadening method.

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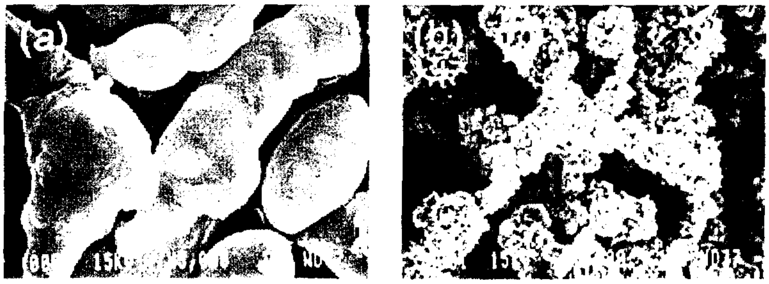

Fig. 2. SEM images of (a) Al and (b) Ni starting powders used in this study.

3. Results and Discussion

Fig. 3 is the summary of XRD results for prepared powder. Whereas powder mixture of (Ni+Al) showed that Ni and Ti exist unchanged irrespective of mixing time, NiAl and Ni3Al phases are formed in mechanically alloyed powder.

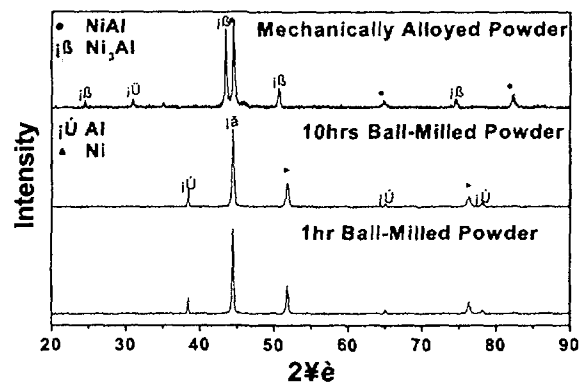

Fig. 3. XRD result for (Ni+Al) powder mixture and mechanically-alloyed NiAl powder.

Fig. 4 shows the curve of change in relative density during a whole sintering process for mechanically alloyed NiAl powder and (Ni+Al) powder mixture. It can be seen in figure that MA-NiAl powder compact has a higher green density and starts the shrinkage at higher temperature compared to (Ni+Al)

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powder mixture. Sintered density was 97% and 99% for MA-NiAl and (Ni+Al) powder mixture, respectively.

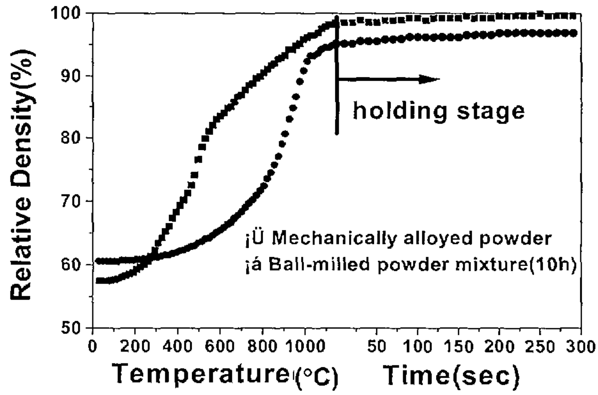

Fig. 4. Change in relative density of MA-NiAl powder and (Ni+Al) powder mixture during SPS process.

Fig. 5 shows the curve of change in densification rate during sintering process for the specimens given in Fig. 4. It is evident that both two powder compacts have different densification behaviour. The densification rate of mechanically alloyed NiAl powder compact increased gradually up to 800°C and drastically to 900°C, forming a single maximum on curve. (Ni+Al) powder mixture showed a drastic increase in densification rate even at low temperature and had two maxima on the curve near 300 and 500°C, respectively. This different behaviour seems to be caused by occurring of self-propagating high-temperature synthesis (SHS) reaction between Ni and Al in (Ni+Al) powder mixture. In fact, we could confirm an exothermic reaction near 300°C from DSC analysis. Additionally to this reaction, a melting can be occurred particularly on Al particles. It seems that a formation of heat and liquid phase could enhance densification, especially in the early stage of sintering.

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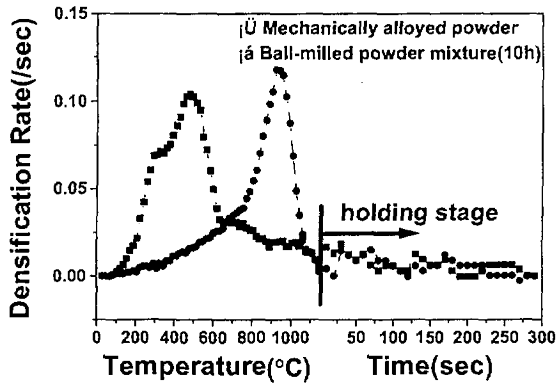

Fig. 5. Change in densification rate of MA-NiAl powder and (Ni+Al) powder mixture during SPS process.

XRD analysis for sintered bodies of MA-NiAl and (Ni+Al) powder mixture revealed that NiAl exists as major phase with Ni_3Al as secondary phase (Fig. 6). Phase change of (Ni+Al) powder mixture during sintering process is given in Fig. 7. It can be seen that Ni and Al remain unchanged up to 500°C, form NiAl, Ni_2Al and Ni_3Al between 500 and 600°C, and above 900°C NiAl and Ni_3Al coexist.

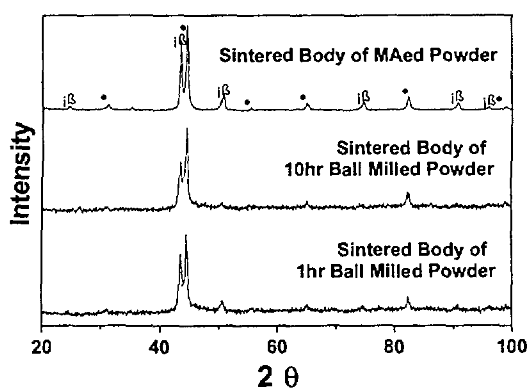

Fig. 6. XRD analysis for sintered bodies of MA-NiAl and (Ni+Al) powder mixture.

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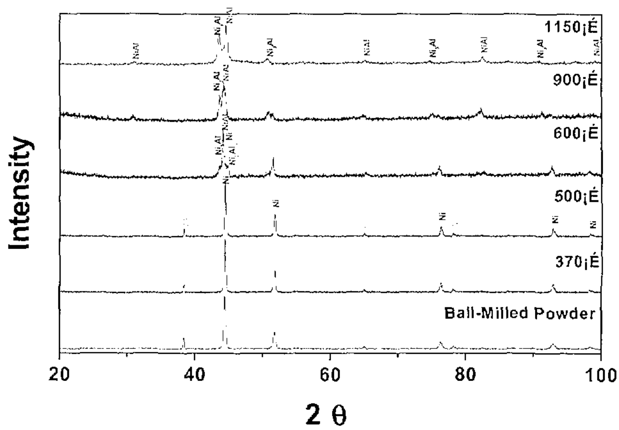

Fig. 7. Phase change of (Ni+Al) powder mixture during SPS process.

SEM observation on fracture surface of sintered body of MA-NiAl powder compact (Fig. 8) showed that agglomerates formed during MA became fine and densified to very fine grain-sized microstructure. The grain size determined by X-ray line broadening method was approximately 80 nm.

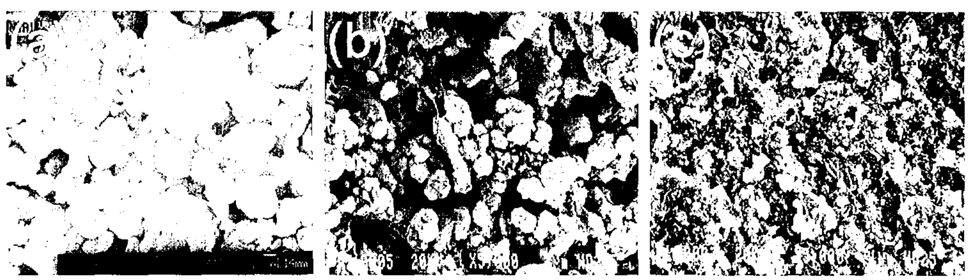

Fig. 8. SEM images of fracture surface of sintered body of MA-NiAl powder compact : (a) Mechanically alloyed powder, (b) sintered at 900°C for 0min. and (c) at 1150°C for 5min.

Fig. 9 shows a microstructure change of (Ni+Al) powder mixture during SPS process. Ni particles with sharp needlike surface cannot be found already at 500°C. A significant grain growth is observed at 600°C. Further increase of temperature to 900°C led to a rapid grain growth.

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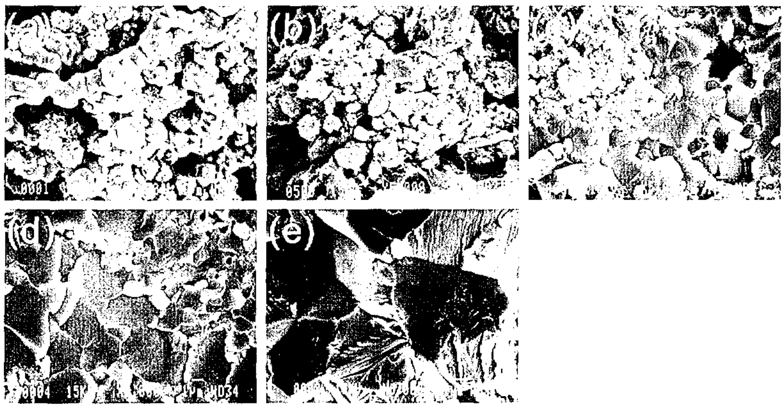

Fig. 9. SEM images of fracture surface of sintered body of (Ni+Al) powder mixture : (a) As-ball-milled powder, sintered (b) at 500°C for 0min., (c) at 600°C for 0min., (d) at 900°C for 0min., and (e) at 1150°C for 5min.

Fine grain size of mechanically alloyed NiAl powder compact resulted in an higher mechanical properties than (Ni+Al) powder mixture. Fig. 10 is the summarized result of Vicker's hardness and transverse rupture strength. Mechanically alloyed NiAl powder compact showed an average Hv value of 555 and TRS of 1393MPa. Fracture behaviour was also different from each other. As can be seen in Fig. 11 the mechanically alloyed NiAl specimens were broken with multi-fracture paths, while the (Ni+Al) powder specimen showed a typical brittle fracture behavior.

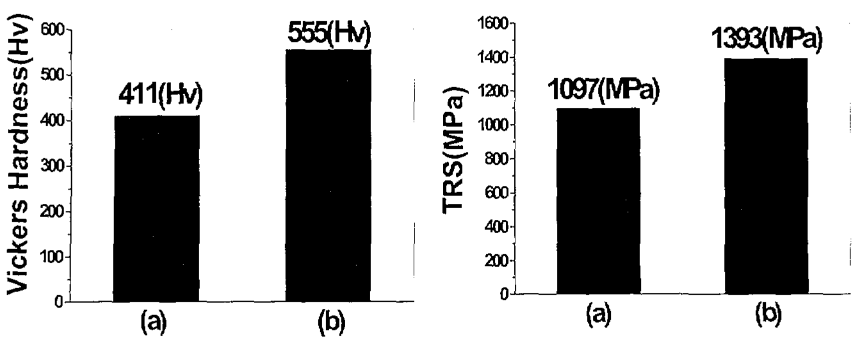

Fig. 10. Comparison of average Vicker's hardness and TRS (Tranverse Rupture Strength) value of sintered body of (a) (Ni+Al) powder mixture and (b) mechanically alloyed NiAl powder.

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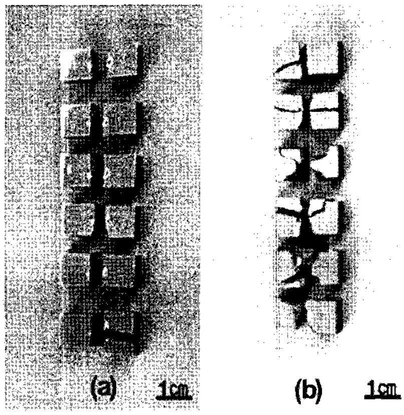

Fig. 11. Fractured specimens of (a) (Ni+Al) powder mixture and (b) mechanically alloyed NiAl

4. Conclusion

From the above results of spark-plasma sintering experiments on mechanically alloyed NiAl powder and (Ni+Al) powder mixture it could be concluded as follows:

(1) Mechanically alloyed NiAl powder compacts showed a somewhat lower sintered density (97%) compared to (Ni+Al) powder mixture (99%). But the grain size of the former one was extremely finer than the latter one (80nm : several tens μm).

(2) The different behavior of densification between both two powder compacts seems from the fact that a self-propagating high-temperature synthesis reaction occurs in case of (Ni+Al) powder mixture during sintering process.

(3) Sintered body of mechanically alloyed NiAl powder compact showed higher Vicker's hardness and TRS than (Ni+Al) powder mixture: (555 H_v:411 H_v) and (1397MPa : 1093MPa).

Acknowledgements

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