Journal of Korean Powder Metallurgy Institute
Vol. 10, No. 3, 2003

Densification Behavior of Mechanically Alloyed NiAl Powder Compact during Spark-plasma Sintering and its Mechanical Property

Ji-Soon Kim, Soon-Ho Jung, Young-Il Jang and Young-Soon Kwon
School of Materials Science and Engineering, University of Ulsan,
San-29 Mugeo-2 Dong, Namgu, Ulsan 680-749, Korea
(Received 16 May 2003 ; Accepted form 9 June 2003)

Abstract Mechanically-alloyed NiAl powder was sintered by Spark-Plasma Sintering (SPS) process. Densification and behavior mechanical property were determined from the experimental results and analysis such as changes in linear shrinkage, shrinkage rate, microstructure, and phase during sintering process, Vickers hardness, and transverse-rupture-strength (TRS). Above 97% relative density was obtained after sintering at 1150°C for 5 min. Crystallite size determined by the Scherrer method was approximately 80 nm. From the X-ray diffraction analysis it was confirmed that the sintered bodies were composed mainly of NiAl phase together with Ni_3Al phase. Measured Vickers hardness and TRS value were 555±10 H_v and 1393±75 MPa , respectively.

Keywords : NiAl, Spark-plasma sintering, Mechanical alloying, Vickers hardness, Transverse-rupture strength, 4-point bending strength, Fracture toughness, Elastic modulus

1. Introduction

NiAl is being expected as a promising 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]. Additionally, it shows a thermoelastic transformation behavior at relatively higher temperature than usually known shape-memory alloy such as TiNi. But, its poor workability at room temperature limits a wide application of NiAl. Some efforts to improve the ductility have already done by 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. Mechanical alloying is known to be one of the simplest methods to produce homogeneous and fine nanostructured alloy powders on relatively large scale.

Mechanically-alloyed nanostructured powders should be densified without any grain growth during sintering process. SPS (Spark-Plasma Sintering) is recently being known to be very effective for this purpose. 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. Electric current is applied to a specimen in pulse-form and the specimen is heated both by resistance heating of the specimen itself and the conductive die mold (usually graphite). Fig. 1 is a schematic configuration of SPS facility. The reported material systems involve most of hard-to-sinter materials such as intermetallic compounds[6] and refractory[7-8], high-performance engineering ceramics and their composites[9-13].

In this study a production of nanograined NiAl

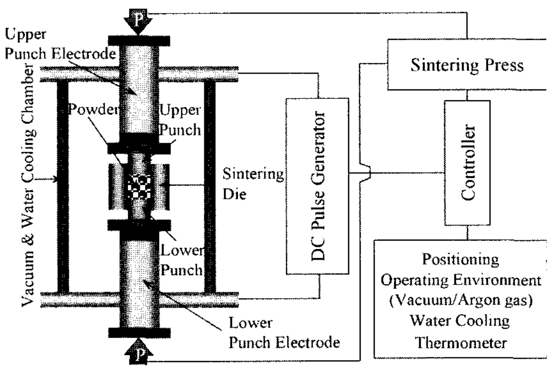

Fig. 1. Schematic configuration of SPS facility.

powder and its full densification by SPS was aimed. Densification behavior, microstructure and mechanical properties were investigated.

2. Experimental Procedure

Al and Ni powders with a purity of 99.9% were used as starting materials. Average particle sizes were 45 and 5 μm, respectively. Al and Ni powders were weighed to have a final composition of Ni-36 at.% Al. Mechanical alloying was performed at 600 rpm for 20 h in a high-energy attrition mill 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.

Sintering was carried out in a SPS facility under the following conditions: Sintering temperature of 1150°C, heating rate of 100°C/min, holding for 5 min at sintering temperature, and applying pressure of 50 MPa. Sintered density was measured by an electronic densimeter. 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.

3. Results and Discussion

Fig. 2 is the summary of XRD results for prepared powder. NiAl and Ni_3Al phases are formed in mechanically alloyed powder.

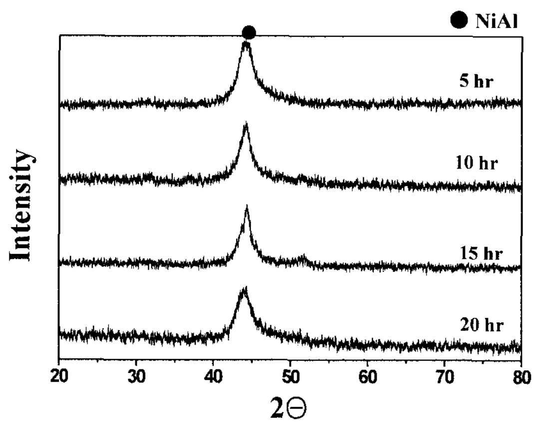

Fig. 2. XRD results of mechanically alloyed powders as a function of milling time.

Fig. 3 shows the curve of change in relative density during a whole sintering process for mechanically alloyed NiAl powder. It can be seen in figure that MA-NiAl powder compact has a green density of 61%. Shrinkage starts near 400°C. Sintered density was 97%.

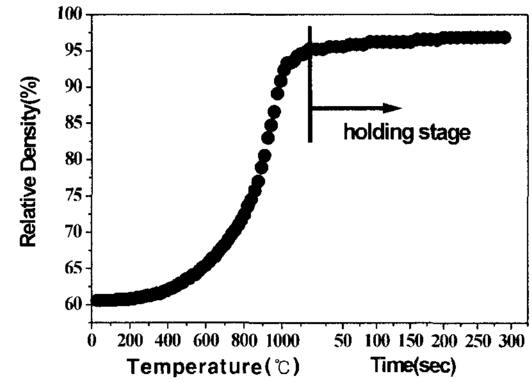

Fig. 3. Change in relative density of MA-NiAl powder during SPS process.

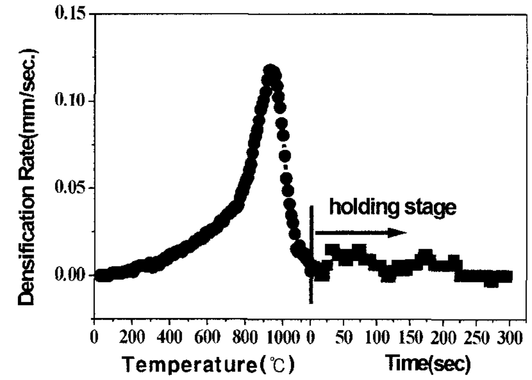

Fig. 4. Change in densification rate of MA-NiAl powder during SPS process.

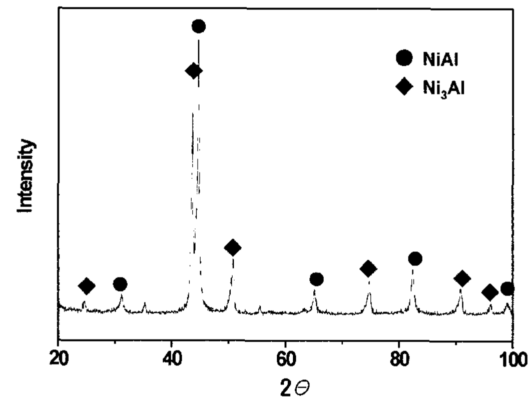

Fig. 5. XRD result for sintered body of MA-NiAl.

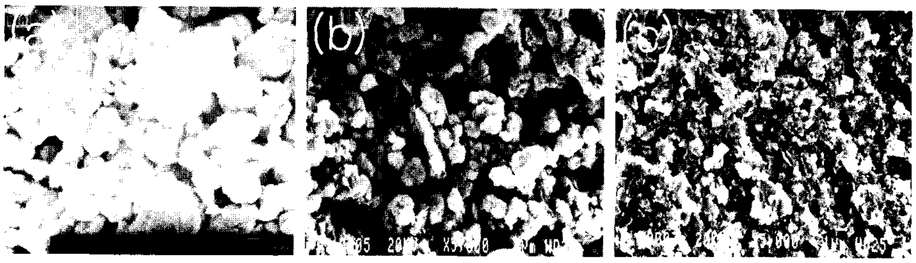

Fig. 6. SEM images of fracture surface of sintered body of MA-NiAl powder compact : (a) Mechanically alloyed powder, (b) sintered at 900°C for 0 min and (c) at 1150°C for 5 min.

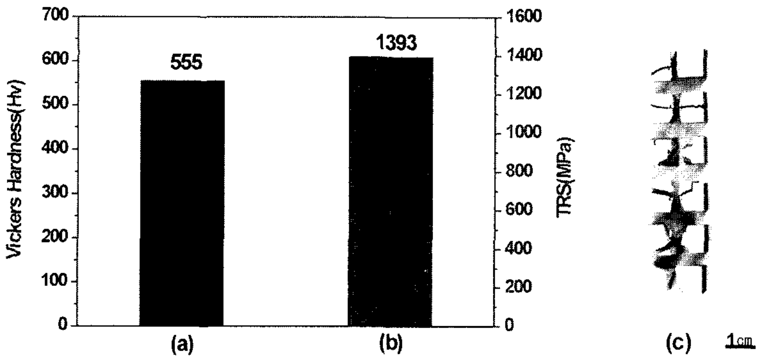

Fig. 7. The average (a) Vickers hardness and (b) TRS (Transverse Rupture Strength) value of sintered body and (c) a photograph of fractured specimens.

Fig. 4 shows the curve of change in densification rate during sintering process for the specimens given in Fig. 3. The densification rate increased gradually up to 800°C and drastically to 900°C, forming a single maximum on curve.

XRD analysis for sintered bodies of MA-NiAl revealed that NiAl exists as major phase with Ni_3Al as secondary phase (Fig. 5).

SEM observation on fracture surface of sintered body of MA-NiAl powder compact (Fig. 6) 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.

Fine grain size of mechanically alloyed NiAl powder compact resulted in a good mechanical propertiey. Fig. 7 is a summarized result of Vickers hardness and transverse rupture strength. It showed an average Hv value of 555 and TRS of 1393 MPa. The TRS specimens were broken with multi-fracture paths.

4. Conclusions

From the results of spark-plasma sintering experiments on mechanically alloyed NiAl powder it could be concluded as follows:

(1) Mechanically alloyed NiAl powder compacts showed a sintered density of 97%. The grain size was very fine which ranged submicron to several tens nanometer.

(2) Sintered body of mechanically alloyed NiAl powder compact showed a good mechanical property: A high Vickers hardness and TRS of 555 H_v and 1393 MPa.

Acknowledgements

This work was supported by the Korea Science and Engineering Foundation (KOSEF) through the Research Center for Machine Parts and Materials Processing (ReMM) at University of Ulsan.

References

1. K. Enami and S. Nenno: Metall. Trans., **2** (1971) 1487.
2. Y. D. Kim and C. M. Wayman: Script. Metall., **24** (1990) 245.
3. U. D. Hangen and G. Sauthoff: Intermetallic, **7** (1999)

501.
4. Y. K. Au and C. M. Wayman: Scipt. Metall., **6** (1972) 1209.
5. J. L. Smialek and R. F. Hehemann: Metall. Trans., **4** (1973) 1571.
6. P. Kimura and S. Kobaysi: J. Metals and Materials Japan, **58** (1994) 201.
7. Y. H. Park and T. Y. Eam: J. Japan Soc. of Powder and Powder Metallurgy, **44** (1997) 530.
8. M. Omori, H. Sakai, A. Okubo, M. Kawahara, M. Tokita and T. Hirai: Preparation and Properties of ZrO_2 (3Y)/Ni FGM, Proc. the 3rd Int'l. Symp. on Structural and FGM, Lausanne, Switzerland (1994) 99.
9. M. Omori, H. Sakai, A. Okubo, M. Kawahara, M. Tokita and T. Hirai: Preparation of Functional Gradient Materials by SPS, Symp. Mater. Research Society, Japan (1994).
10. G. A. Weissler: Resistance Sintering with Alumina Dies, Int'l. J. Powder Metallurgy & Powder Technology, **17** (1981) 107.
11. Y. Goto, M. Sasaki, K. Mukaida, M. Omori, A. Okubo, T. Hirai and T. Nagano: J. Japan Soc. of Powder and Powder Metallurgy, **45** (1998) 1061.
12. N. Tamari, T. Tanaka, K. Tanaka, I. Kondo, M. Kawahara and M. Tokita: J. Ceramic Soc. Japan, **103** (1995) 740.
13. M. Omori, A. Okubo and T. Hirai: Proc. of 1993 Powder Metallurgy World Congress, Kyoto (1993) 935.