CONF-961202--32
ANL/MSD/CP--91826

SYNTHESIS, CHARACTERIZATION AND MECHANICAL PROPERTIES OF NANOCRYSTALLINE NiAl*

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November 1996

To be submitted to the Materials Research Society, December 2-6, 1996, Boston, MA.

*Work supported by the U.S. Department of Energy, Basic Energy Sciences-Materials Sciences, under contract #W-31-109-ENG-38, the Alcoa Corporation, and by AFOSR Grant # FY9620-92-J.

Synthesis, Characterization and Mechanical Properties of Nanocrystalline NiAl

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ABSTRACT

Nanocrystalline NiAl has been produced from pre-cast alloys using an electron beam inert gas condensation system. In-situ compaction was carried out at 100 to 300°C under vacuum conditions. Energy dispersive spectroscopy was used to determine chemical composition and homogeneity. Average grain sizes in the range of 4 to 10 nm were found from TEM dark field analyses. A compression-cage fixture was designed to perform disk bend tests. These tests revealed substantial room temperature ductility in nanocrystalline NiAl, while coarse grained NiAl showed no measurable room temperature ductility.

INTRODUCTION

Intermetallic alloys are of interest for a variety of potential applications because of their high strength-to-weight and stiffness-to-weight ratios and because they often possess excellent elevated temperature properties. The fundamental limitation in the commercial utilization of these materials invariably is their inherent ambient temperature brittleness, which adversely affects material handling and fabricability.

Schulson and Barker [1] reported an improvement in tensile ductility at 400°C in ordered NiAl as a result of grain size refinement to sizes as small as 20 μm. While Schulson's and Barker's results demonstrated that grain size refinement can increase ductility, these results did not give any information about the room temperature deformation behavior of NiAl. A number of recent studies have begun investigating the possibility of improving room temperature ductility of intermetallic alloys through further size refinement to the nanocrystalline regime. In this respect, investigators [2,3] produced n-NiAl through different processing routes and concluded that some evidence of room temperature ductility is present in these materials. In case of [2], NiAl was ball milled to obtain grain sizes in the μm to nm regime. They used miniaturized disk bend testing and observed that materials that had low carbon content showed some evidence of ductility while materials with higher carbon content showed no ductility. In addition to carbon, their materials also contained substantial amounts of other impurities and questions remain regarding the possible effects of these impurities, as well as the broad grain size distribution of their materials on the observed properties. On the other hand, the investigation by Haubold and co-workers [3] was done on cleaner nanocrystalline NiAl produced by the inert gas condensation (IGC) method, but their mechanical properties evaluation was limited to microhardness tests done on a single sample annealed at various temperatures. A more comprehensive study of mechanical behavior of nanocrystalline NiAl with low impurity levels is clearly needed and motivates the present study.

In the present investigation, the ambient temperature deformation behavior of n-NiAl was investigated using biaxial disk bend testing (BDBT). Disk bend testing offers possible advantages over tensile testing in characterizing nanocrystalline samples, particularly since thin disk-shaped samples can be tested in as-produced form. This method eliminates the need to machine dogbone-shaped samples and subsequently attach grips and strain gauges to typically small samples produced by the IGC method. Previous mechanical tests on nanocrystalline NiAl [2] used the miniaturized disk bend testing technique with 3 mm diameter specimens. In the present study, biaxial disk bend testing is used with 9 mm diameter disks produced directly from our synthesis facility without any post production sample machining. This avoids the potential introduction of stresses in the specimen. The load was applied through a flat punch, as seen in Fig. 1, which also eliminated the uncertainties associated in determining the exact contact area between the specimen and the punch. Knowing the contact area accurately is important in calculating the exact value of yield stress from the measured displacements.

EXPERIMENTAL

Nanocrystalline NiAl was produced by the inert gas condensation process using an electron beam evaporation system [4]. Nickel and NiAl were evaporated from separate crucibles with dwell times chosen to yield the desired 50:50 composition. A Ni source was required to compensate for the larger vapor pressure of Al compared to Ni. The powder produced was transported under vacuum to an adjoining chamber where 9 mm diameter disks were compacted at 1.4 GPa. High vacuum compactions at temperatures of 100 - 300°C were performed, resulting in sample densities that varied from 70 - 90% of theoretical. Chemical analysis was done by energy dispersive spectroscopy (EDS) and average grain size was determined by dark field transmission electron microscopy (TEM).

BDBT was performed to characterize the mechanical behavior of both nanocrystalline and coarse grained NiAl samples. All samples were mechanically polished prior to testing to produce a $0.05\mu m$ surface finish. A compression cage fixture was designed and built for these measurements. A schematic representation of the BDBT apparatus is shown in Fig. 1. The 9 mm diameter specimens were freely supported on a 7 mm diameter ring and load was applied to the center of the disk through a flat punch having a 1.15 mm diameter. The BDBT load-displacement data were analyzed following the procedures outlined in [5].

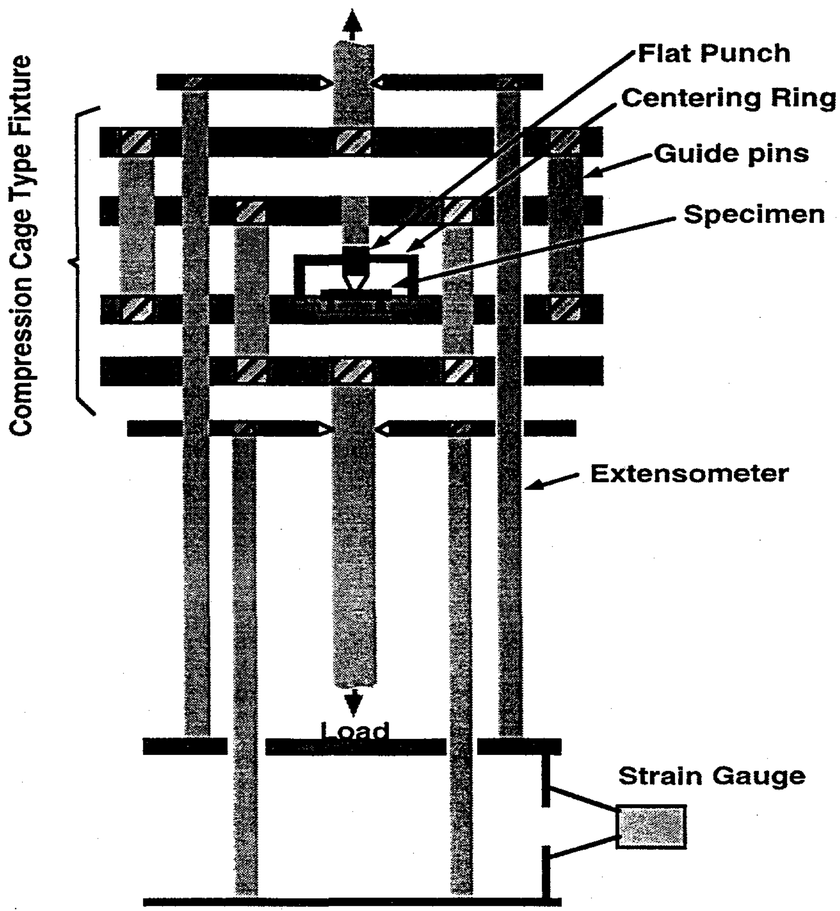

Figure 1. Schematic drawing of a cross section of the BDBT apparatus.

RESULTS AND DISCUSSION

Chemical analysis results for two nanocrystalline specimens and one coarse grained (CG) NiAl specimen are shown in Table I. From each specimen, an EDS/SEM analysis was done on three random locations from a relatively large area. EDS/TEM analysis was done in a nanoprobe mode with a nominal probe size of ~10 nm, thus producing information on a much more local scale. RBS scans covering an area of approximately one mm^2 were used to obtain macroscopic compositions. Clearly the small scatter in the composition obtained by all three methods indicates that the samples are very homogenous in composition on a microscopic as well as macroscopic level. Also, the good agreement between all three methods is further evidence of sample homogeneity. This behavior differs from that of nanocrystalline Al-Zr [6] produced in the same system, where large variations in the composition were obtained within the same sample. It is possible that in the case of Al-Zr, the limited diffusion rate of Zr in Al during evaporation and/or consolidation prevents homogenization. Faster diffusion of Ni and Al in NiAl than of Zr in Al is believed to result in a more uniform composition in the present case.

Table I Chemical composition, density, and mechanical properties of two nanocrystalline NiAl and one coarse grained (CG) NiAl sample.

Sample #	EDS(SEM) at.% Ni.	EDS(TEM) at.% Ni.	RBS at.% Ni.	Density % Theor.	σ_y MPa	Fracture Mode
NiAl-1	48.9±0.3	48.1±1.3	48.6±0.7	84.67	91.32	Ductile
NiAl-2	48.6±0.4	48.8±0.9	48.1±0.5	90.05	134.67	Ductile
CG- NiAl	50.3±0.1	-	-	100	-	Brittle

Figs. 2(a) and (b) show a dark field TEM image of n-NiAl-1 and a histogram of the grain size distribution. The grain size distribution is quite narrow and uniform throughout the specimen with an average size of ~6 nm in this particular case.

Before disk bend testing nanocrystalline NiAl specimens, the performance of the BDBT apparatus was evaluated by testing standard coarse grained stainless steel and aluminum samples. These materials have very well characterized mechanical properties. Load-displacement curves obtained were similar to load-displacement curves obtained by other investigators on similarly ductile materials when subjected to disk bend testing [7]. Also, yield stress and modulus values obtained were within ±5% of the literature values for the same materials tested by more conventional tensile test methods.

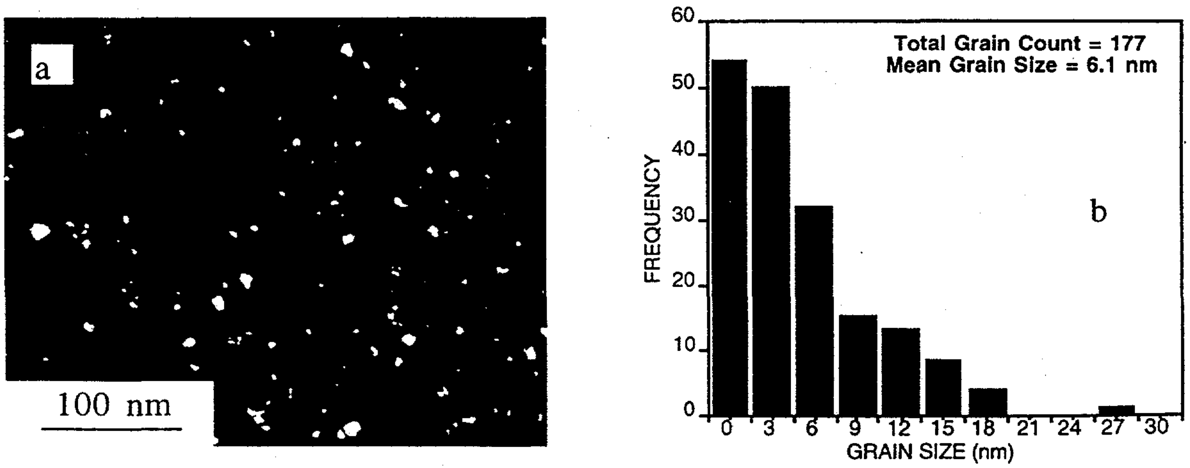

Figure 2. (a) Dark field TEM image of nanocrystalline NiAl-1, and (b) histogram showing the grain size distribution for the same specimen.

A representative room temperature load-displacement curve for n-NiAl-1 and coarse grained NiAl sample strained at 1.4×10^{-6} sec^{-1} is shown in Fig. 3. After correction for the expected initial non linear region [7], the yield stresses of nanocrystalline samples were calculated from the total loads at yielding (taking into account the sample thicknesses according to the procedures described in [5]). Yield stress values are given in Table I. Coarse-grained NiAl samples fractured prior to yielding and the stresses at fracture were typically approximately 75 MPa, well below the yield stresses of nanocrystalline samples.

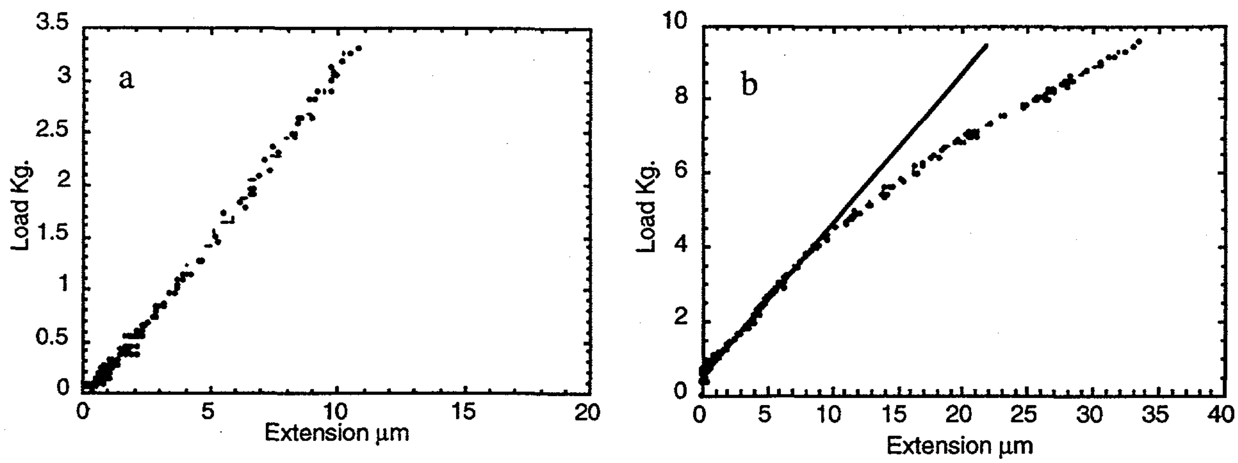

Figure 3. Load-displacement curves obtained at room temperature for (a) coarse grained NiAl (sample failed prior to yielding), and (b) nanocrystalline NiAl-1.

It has been shown by Li et al. [7] that for disk bend tests plastic yielding occurs at the onset of the deviation from linearity in the load-displacement curve and that the yield strength obtained by disk-bend testing is comparable to the tensile yield strength. A significant and most important observation in the present study is that a deviation from linearity is observed in the load-displacement curve for nanocrystalline NiAl, indicating that NiAl in nanocrystalline form exhibits yield behavior and thus measurable room temperature ductility. A deviation from linearity was not observed in eight CG-NiAl samples tested under similar conditions. Strains-to-failure of 0.1 - 0.2% and 0.016 - 0.06% were observed at room temperature in the present study for n-NiAl and CG-NiAl, respectively. The values of total strain obtained for n-NiAl samples may be hampered by the residual porosity since stress concentrations could develop at the voids resulting premature fracture. Therefore, the measured strain-to-failure values in the present case are most likely lower limits. Further improvements in ductility may be obtained if denser specimens can be obtained in the future. The failure mode of n-NiAl was determined to be ductile from postmortem examination of broken pieces using SEM, as seen in Fig. 4.

The apparent improvement in the room temperature ductility in n-NiAl can be understood by examining the potential deformation mechanisms in nanocrystalline materials. For materials that are normally ductile at ambient temperature in coarse grained form (e.g. fcc metals) a significant reduction in ductility is observed when grain sizes are reduced to the nanometer range [6,8]. In these fcc metals, deformation occurs by dislocation generation and motion. It has been pointed out by numerous authors (e.g., [9]) that dislocation generation is increasingly difficult as grain sizes decrease, leading to a decrease in dislocation-based ductility in nanocrystalline materials. Likewise, diffusional mechanisms are expected to be more active in nanocrystalline materials than in coarse grained materials due to the large volume fraction of atoms located in or near grain boundaries [10], which display far higher diffusion rates than the bulk. In normally ductile materials, it appears that any increase in ductility afforded by diffusional mechanisms is insignificant compared to the competing loss of ductility due to the hindrance of dislocation motion [11]. In contrast to the behavior of materials that are normally ductile in coarse grained form, the present studies indicate that in the case of a normally brittle material such as NiAl where dislocation-based ductility is limited even for coarse-grained material, deformation of nanocrystalline samples is likely affected to a measurable extent by enhancements of diffusional mechanisms (such as grain boundary sliding and Coble creep) that accompany grain size refinements.

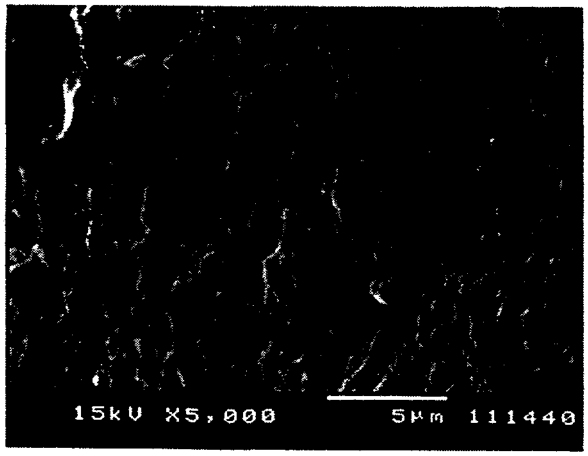

Figure 4. SEM micrograph showing a ductile-appearing fracture surface of n-NiAl-1.

Another aspect of the observed improved ductility to consider is the test method itself. Since the stress state during a disk bend test is complex and involves both tensile and compressive stresses, it is possible that this may have an effect on the measured ductility of nanocrystalline materials. For example, significantly larger ductilities are seen for nanocrystalline Cu tested in compression [12] than in tension [8]. However, this cannot be the only reason for the measured enhanced ductility in case of n-NiAl, since coarse grained specimens did not show any evidence of ductility when also tested by BDBT under similar conditions.

CONCLUSIONS

The conclusions drawn from this study are:
1) Ordered single phase nanocrystalline NiAl can be successfully synthesized using an electron beam inert gas condensation system. The chemical composition and grain size distribution is quite uniform on both microscopic and macroscopic scales.
2) The substantial room temperature ductility observed in nanocrystalline NiAl samples suggests that diffusional mechanisms such as grain boundary sliding controlled by grain boundary diffusion are contributing substantially to deformation.

ACKNOWLEDGMENTS

This research was supported by AFOSR Grant# FY9620-92-J, and by U.S. Department of Energy BES-DMS Contract# W-31-109-ENG-38. The authors gratefully acknowledge the help of Dr. John Kramer for Instron operation and experimental setup, Bernie Kestle for TEM specimen preparation, Pete Baldo for performing the RBS experiment, and Dr. Stanslaw Dymek for TEM operation.

REFERENCES

1. E. M. Schulson and D. R. Barker, Scripta Met. **17**, 519 (1983)
2. T. R. Smith, Nanostruct. Mater. **5**, 337 (1995)

3. T. Haubold, R. Bohn, R. Birringer and H. Gleiter, Mater. Sci. and Eng. **A153**, 679 (1992)
4. J. A. Eastman, L. J. Thompson and D. J. Marshall, Nanostruct. Mater. **2**, 377 (1993)
5. S. Timoshenko and S. Woinowsky-Krieger, Theory of Plates and Shells, 2nd ed., McGraw-Hill, New York, NY, (1959)
6. M. N. Rittner, T. R. Weertman, J. A. Eastman, K. B. Yoder and D. S. Stone, Accepted for publication in Materials Science and Engineering A, (1996)
7. H. Li, F. C. Chen and A. J. Ardell, Metall. Trans. **A22**, 2061 (1991)
8. P. G. Sanders, Ph.D. Thesis, Northwestern University, (1996)
9. G.W. Nieman, J. R. Weertman and R.W. Siegel, J. Mater. Res. **6**, 1012 (1991)
10. G. Palumbo, S. J. Thrope and K. T. Aust, Scripta Metall. et Mater. **24**, 1347 (1990)
11. J. A. Eastman, M. Choudry, M. N. Rittner, C. J. Youngdahl, M. Dollar, J. R. Weertman, R. J. DiMelfi and L. J. Thompson, Submitted to the proceedings of the annual meeting of the Minerals, Metals, and Materials Society, Orlando (1997)
12. C. Y. Youngdahl, J. R. Weertman and J. A. Eastman, Submitted to Scripta Met. (1996)