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Rapidly Solidified NiAl and FeAl

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ABSTRACT

Melt spinning was used to produce rapidly solidified ribbons of the B2 intermetallics NiAl and FeAl. Both Fe-40Al and Fe-45Al possessed some bend ductility in the as spun condition. The bend ductility of Fe-40Al, Fe-45Al, and equiatomic NiAl increased with subsequent heat treatment. Heat treatment at approximately 0.85 T_m resulted in significant grain growth in equiatomic FeAl and in all of the NiAl compositions. Low bend ductility in both FeAl and NiAl generally coincided with intergranular failure, while increased bend ductility was characterized by increasing amounts of transgranular cleavage fracture.

INTRODUCTION

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The B2 crystal structure aluminide intermetallics of Fe and Ni represent potential structural materials for use at elevated temperatures. These materials possess a high melting point, low density, and resistance to oxidation and high temperature deformation. Much work has been done in evaluating the mechanical properties of NiAl and FeAl in the cast [1], cast and wrought [2], cast and extruded [3,4], and P/M and extruded [5] forms. One problem limiting the potential use of these materials is their low room temperature ductility. Application of rapid solidification processing to these materials has already produced improvements in strength and ductility as compared to conventional processing [6,7,8].

Taub, Huang, and Chang [6] applied rapid solidification by melt spinning to Ni_3Al and B modified Ni_3Al and found that rapidly solidified material (5-10 μm grain size) had approximately twice the room temperature yield strength of cast material (200-1000 μm grain size) in both alloys. Although they found that increasing the grain size of the rapidly solidified material from 5-10 μm to 25-30 μm by heat treatment decreased the yield strength significantly in both alloys, they still stated that grain size alone could not explain the difference between rapidly solidified and as cast properties. Improvements in the room temperature ductility and strength of $L1_2$ compounds in Ni-(10-22)Al-X (X=12.5Cr, 6Mn, 10Fe, 20Co, or 12Si) alloys using the melt spinning technique were reported by Inoue, Tomioka, and Masumoto [7]. Although grain refinement was observed, the improvements in ductility and strength were attributed to the suppression of grain boundary segregation, and the low degree of ordered state with a high density of APB's, which was caused by suppression of ordering. Gaydosh, Jech, and Titran [8] also found that melt spun Ni-50Al having a grain size of 5.4 μm exhibited limited plastic deformation in a free bend test at room temperature. The fine grain size was thought to be at least partially responsible for the appearance of ductility.

This paper presents the results of an investigation into the effects of melt spinning on the microstructure and room temperature ductility of the B2 intermetallics NiAl and FeAl. The effects of annealing time and temperature on the melt spun structure and properties are discussed. Ductility of the melt spun ribbons was determined by using a simple bend test. Standard metallographic, X-ray diffraction, and electron microscopy techniques were used to evaluate the structures and fracture surfaces of the melt spun and heat treated ribbons.

MATERIALS AND PROCEDURES

Free jet melt spinning [9] was used to produce the rapidly solidified material. NiAl and FeAl melt stock was prepared from elemental materials by triple arc-melting in argon. For each run a charge of approximately 13 grams was placed into a dense alumina crucible in a melt spinning apparatus. The charge was heated above its melting point and ejected by pressurized argon onto an uncooled mild steel wheel rotated at a surface speed of 20 meters per second. Melt spinning was performed in a positive pressure argon atmosphere to suppress aluminum vaporization.

The compositions of the rapidly solidified materials are listed in Table I; these compositions were chosen to span the B2 phase fields in each alloy system. Ribbons of each composition were heat treated at two different temperatures for one hour in helium. The FeAl ribbons were treated at 775°C and 1000°C, which represent homologous temperatures of 0.64 to 0.68 and 0.77 to 0.83, respectively, based on the solidus temperatures of the FeAl system. The NiAl ribbons were treated at 1000°C and 1310°C, which represent homologous temperatures of 0.67 to 0.71 and 0.83 to 0.88, respectively, based on the solidus temperatures of the NiAl system.

Optical metallography, X-ray diffraction, scanning electron microscopy (SEM), and transmission electron microscopy (TEM) were used to assess the structure of both the as spun and heat treated ribbon. Grain size was measured using the linear intercept method, where the reported values are the average grain width measured across a longitudinal section, and do not account for the length or depth of the grains. Measurements were made at three different places on each of two different ribbons for every composition. Disks 3 mm in diameter for TEM observation were cut from the ribbons using a Gaton ultrasonic disk cutter. The disks were electropolished to thin foils at -30°C in a 33 percent HNO_3 in methanol solution at 12 volts. Finally, room temperature bend testing was used to provide a relative measure of the ductility of the ribbons.

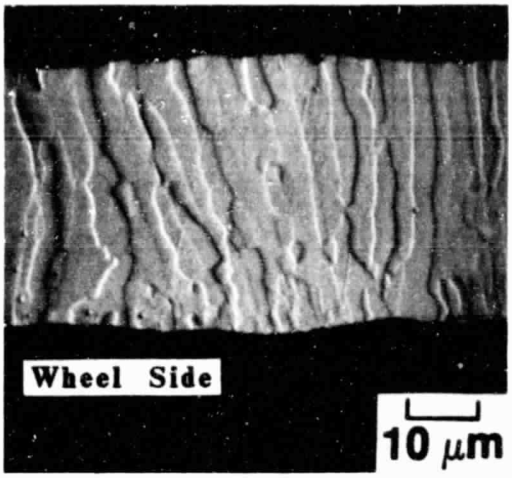
RESULTS AND DISCUSSION

Rapidly solidified ribbon was produced for every composition except Ni-53Al and Ni-55Al. Melt spinning these compositions resulted in only discontinuous flake and powder product. The melt spun ribbon was 2 to 3 mm wide and ranged from 0.025 to 0.038 mm in thickness. A rough surface and serrated edges resulted from the use of an argon atmosphere. Gas entrapment between the wheel and the ribbon caused some surface roughness on the wheel side of the ribbon. The serrated edges and surface roughness on the ribbon's free side were caused by interaction of the solidifying metal with the gas boundary layer. Because of this surface roughness and the small dimensions of the ribbon, tensile testing was not possible.

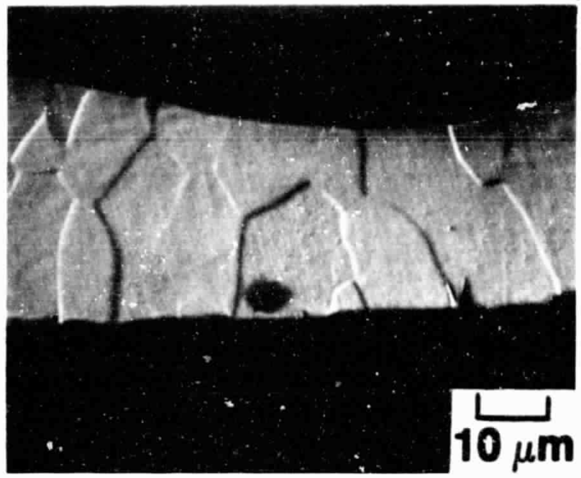
TABLE I

Compositions of Melt Spun Ribbons
Al, at.%

NiAl	42	46	50	53	55
FeAl	40	45	50	50.5	51

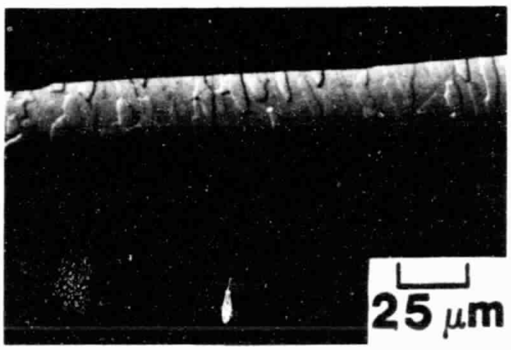


(a)

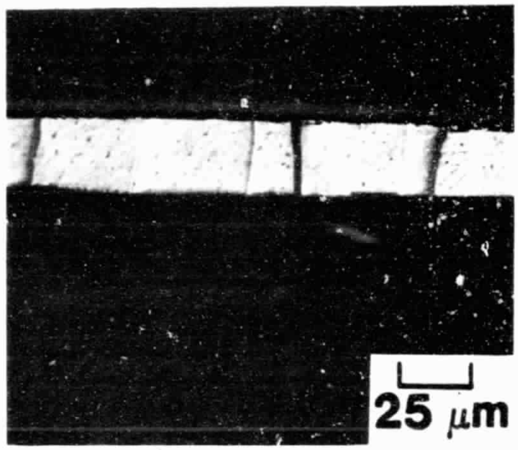


(b)

Figure 1. Microstructure of as spun Ni-50Al.



(a)



(b)

Figure 2. Microstructure of Fe-50Al (a) in the as spun condition and, (b) treated at 1000°C for 1 hour in He.

Microstructure

Optical metallography revealed that both NiAl and FeAl possessed a columnar grain structure extending outward from the wheel side of the ribbon. A typical photomicrograph of the melt spun microstructure of NiAl is shown in Figure 1(a), while that of FeAl is shown in Figure 2(a). The as spun grain width of the NiAl series ranged from 4 to 6 μm, while that of the FeAl series ranged from 6 to 9 μm. Because the grain length is limited by an approximately constant ribbon thickness, the larger grain width in the FeAl ribbons represents a decrease in the grain aspect ratio in comparison to that of NiAl. In addition, small equiaxed grains were observed on the wheel side of the NiAl ribbons. Both the difference in grain size and the presence of equiaxed grains in NiAl indicate differences between the solidification of NiAl and FeAl during melt spinning.

Figure 1(b) is another photomicrograph of equiatomic NiAl from the same melt spin run as the ribbon shown in Figure 1(a). The change in ribbon thickness, grain size, and grain shape illustrate the major problem with this melt spun material; heterogeneity. Differences in microstructure such as those shown here have been observed within a 2 cm length of ribbon. Statistical analysis of a number of specimens was therefore used to determine the grain size and bend ductility of the materials.

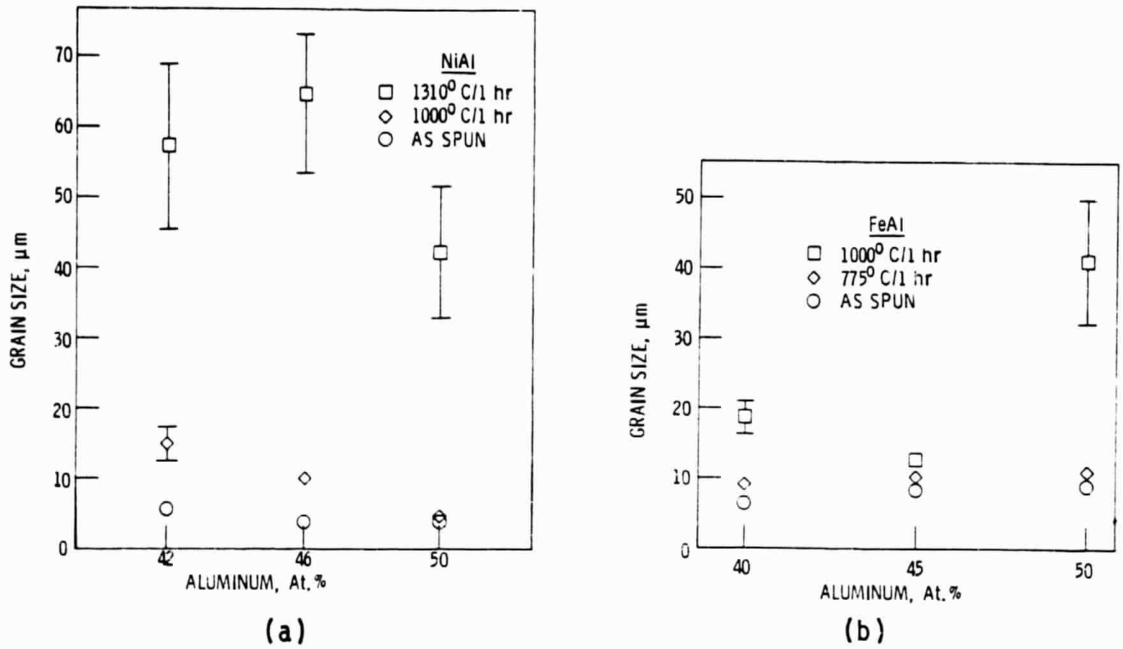


Figure 3. Grain size as a function of heat treatment temperature and composition for melt spun (a) NiAl and, (b) FeAl.

Grain Size

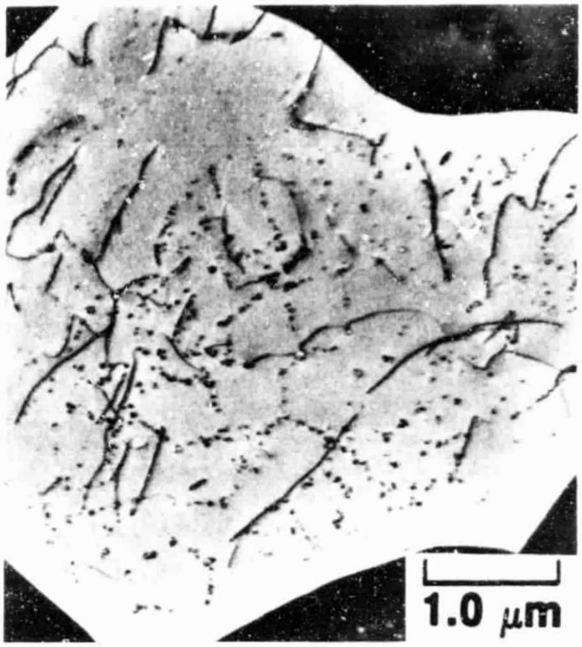
Grain widths measured for the as spun and heat treated ribbons of NiAl and FeAl are shown in Figure 3. Error bars indicate the 95 percent confidence interval. Where no error bars appear, they are smaller than the symbols. Heat treatment at 1000°C for 1 hour produced some grain growth. Although not shown in the figure, the grain size of equiatomic NiAl grew from 8 to 26 µm when treated at 1000°C for 23 hours, indicating the effect of time on grain growth. Heat treatment at 1310°C for 1 hour resulted in large increases in grain width up to 40 to 60 µm.

Heat treatment of the FeAl series at 775°C resulted in little if any growth, while a 1310°C treatment increased the grain size of Fe-40Al and Fe-50Al. The 45 at.% Al composition, however, had some resistance to grain growth. The 30 µm increase in grain width caused by the 1000°C treatment of equiatomic FeAl is illustrated in Figure 2(b). Note how each grain has grown completely through the thickness of the ribbon. This is typical for grain growths of 30 µm and above in all of the compositions studied. Width increases of about 10 µm resulted in nearly 80 percent of the grains growing through the ribbon thickness.

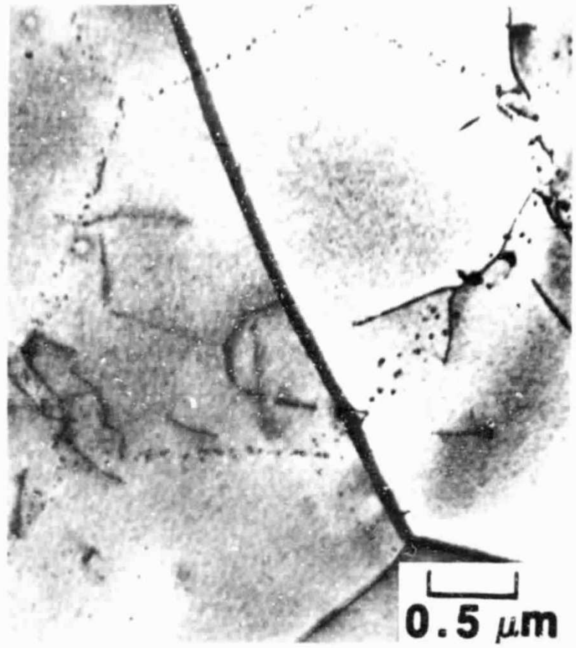
Transmission Electron Microscopy

The substructure of the Ni-50Al ribbons observed by TEM consisted primarily of dislocations and precipitates. In the as spun condition (Figure 4(a)), the dislocations occurred throughout the grains with no apparent orientation preference. Precipitates were distributed in an apparently prior subgrain structure and had little obvious effect on the dislocations. Upon annealing, the Ni-50Al ribbon (Figure 4(b)) showed a decrease in dislocation density. Again, the dislocations were randomly oriented, and the precipitates outlined prior subgrain boundaries.

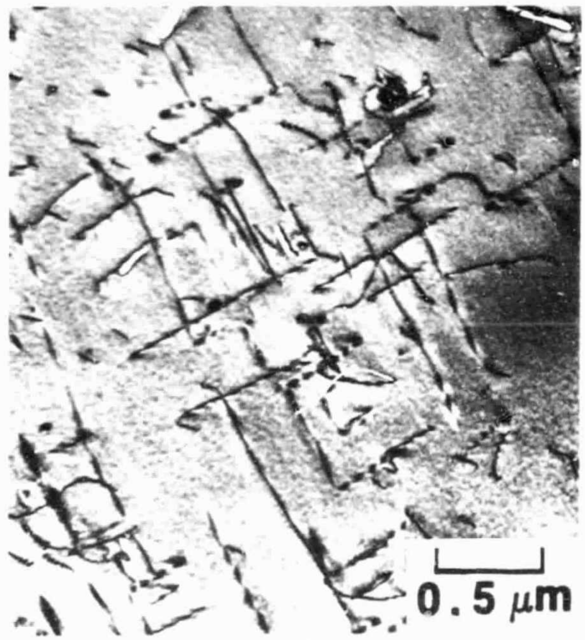
Examination of as-spun Fe-50Al ribbon revealed a relatively large dislocation density, as shown in Figure 4(c). These dislocations were long and straight and assumed three distinct orientations. Precipitates,



(a)



(b)



(c)

Figure 4. TEM substructure of (a) as spun Ni-50Al, (b) Ni-50Al heat treated at 1000°C for 1 hour in He, and (c) as spun Fe-50Al.

which are much larger than those in NiAl, occurred randomly in the structure and some dislocation-precipitate interactions were observed. The as spun Fe-40Al and Fe-45Al ribbons displayed similar structures.

Additional work is required to determine the composition, structure, and origin of the precipitates in both NiAl and FeAl.

X-Ray Diffraction

X-ray diffraction of as spun materials revealed a {100} texture perpendicular to the ribbon surface on the free side of the ribbons, and little or no preferred orientation on the wheel side. All three NiAl compositions exhibited a definite {100} preferred orientation on the free side. Fe-40Al and Fe-50Al had a minor {100} texture, while Fe-45Al possessed little or no preferred orientation. In general, heat treatments which caused grain growth also caused the {100} texture to appear on the wheel side of the ribbon, and heat treatments which had no affect on grain size did not change the original texture. It appears that directional growth occurred in the solidifying ribbons after an initial solidification next to the wheel. These oriented grains then grew through the ribbon when heat treatment caused grain growth.

Bend Test

Room temperature bend testing of both as spun and heat treated ribbons was conducted by free bending the ribbon around a mandrel of successively smaller diameter until failure occurred. Total strain at failure e_f was calculated from the minimum bend radius r and the ribbon thickness t [10].

$$e_f = \frac{t}{(2r + t)} \quad (1)$$

For each condition, half of the tests were conducted with the wheel side of the ribbon as the outer bend surface, and the other half with the free side of the ribbon as the outer bend surface. Typically, a total of eight tests were conducted for each condition. It should be emphasized that bending of these ribbons produced large tensile stresses perpendicular to the columnar grain boundaries. This resulted in a more severe test than if the grain boundaries had been randomly oriented.

The results of the bend test for NiAl and FeAl are shown in Figure 5. The 95 percent confidence interval was calculated for each failure strain, and it lies within the symbols for each average value. The primary type of deformation during the bend test as determined by visual observation is also listed in the two figures. For NiAl, as spun failure strains ranged from 0.9 to 1.3 percent. Heat treatment had relatively little affect on the low aluminum content (42 and 46 at.%) compositions, while it increased the failure strain up to 2.25 percent for the equiatomic composition. Also, deformation in the low aluminum content compositions was primarily elastic, while limited plastic deformation was observed for equiatomic NiAl. Heat treatment at 1000°C either increased or had no effect on ductility. However, exposure at 1310°C seemed to lower the observed failure strain in comparison to the 1000°C heat treatment. Because the only effect of heat treatment on ductility occurred at the equiatomic composition, and because grain growth was relatively uniform for NiAl (Figure 3(a)), it is reasoned that grain growth had no direct effect on the measured bend ductility.

The failure strains of FeAl decreased with increasing Al content between 40 and 50 at.% Al and remained constant for hyperstoichiometric Al

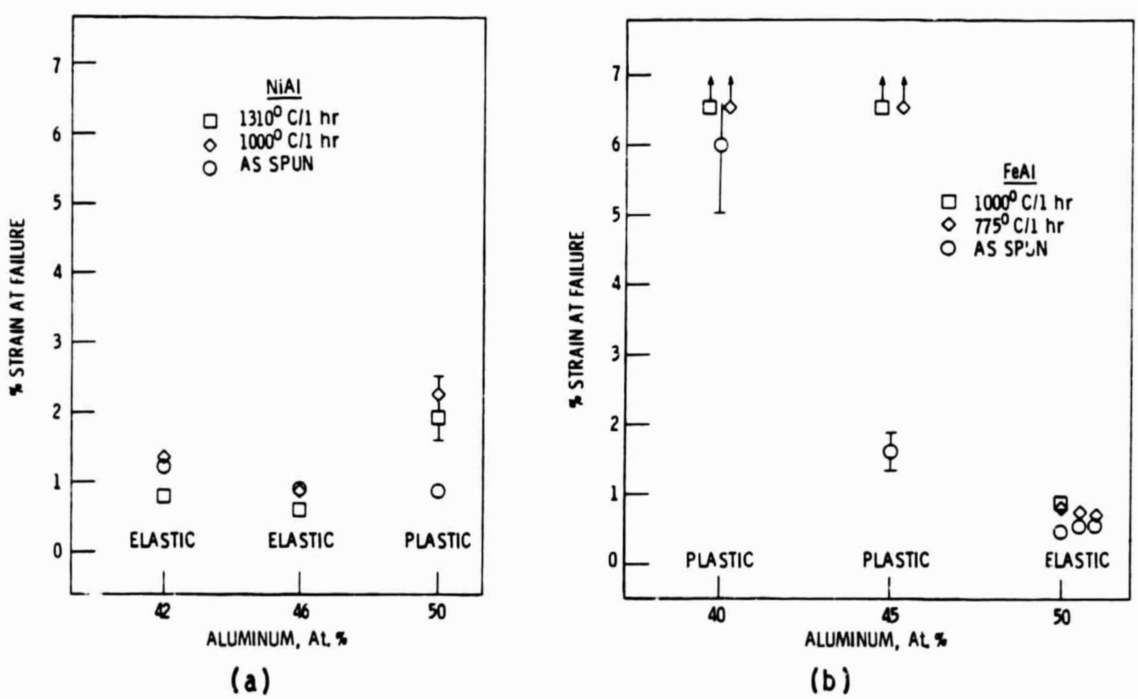


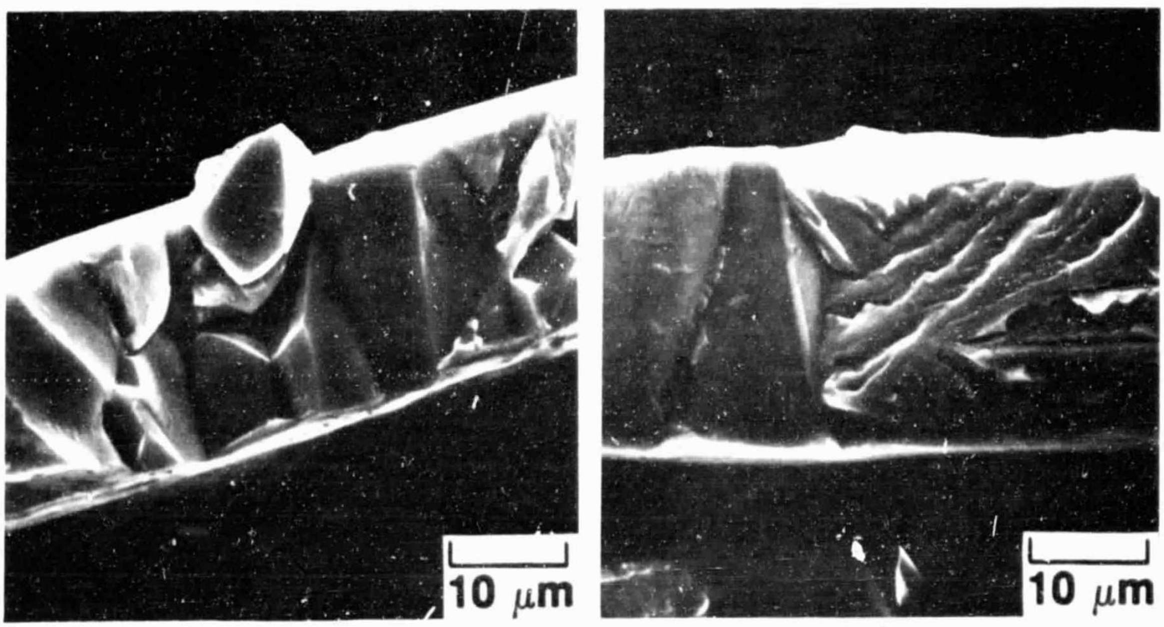
Figure 5. Room temperature bend test results as a function of composition for melt spun (a) NiAl, and (b) FeAl.

levels (Figure 5(b)). The strain to failure for as spun Fe-40Al was quite high (6 percent) and was accompanied by considerable plastic deformation; such ductility was considerably greater than that observed in any NiAl material. The effect of heat treatment with composition was opposite to that in NiAl. The failure strains of the low Al compositions increased dramatically, while the near equiatomic compositions were not affected. In fact, the 40 and 45 Al compositions could be plastically bent 180° and flattened without breaking (This is indicated by the upward arrows in Figure 5(b)), while the near stoichiometric materials failed in a brittle elastic manner. Finally, comparison of Figures 3(b) and 5(b) revealed no direct correlation between grain growth and the measured bend ductility, even though grain growth in FeAl as a function of composition was not uniform.

Fracture Surfaces

Fracture surfaces were examined for ribbons in the as spun and heat treated conditions. Equiatomic NiAl in the as spun condition exhibited almost entirely intergranular fracture with rare instances of transgranular cleavage fracture. The fracture of ribbon heat treated at $1000^{\circ}C$ for one hour was also primarily intergranular, but there was a noticeable increase in the amount of transgranular cleavage. Note that the bend ductility of the heat treated ribbon was twice that of the as spun material. Ni-42Al exhibited a mixture of intergranular and transgranular cleavage fracture in both the as spun and heat treated conditions. Grain growth during heat treatment made any changes in fracture mode hard to quantify for this composition.

Equiatomic FeAl exhibited completely intergranular failure in both the as spun and heat treated conditions. Note that this composition exhibited a low initial value of bend ductility which did not change with heat treatment. Figure 6 shows Fe-40Al in the as spun condition and after heat treatment at $1000^{\circ}C$ for one hour. The as spun material possessed a



(a) (b)

Figure 6. Scanning electron photomicrograph of the fracture surface of (a) as spun Fe-40Al: e_f = 6%, and (b) Fe-40Al treated at $1000^{\circ}C$ for 1 hour in He.

primarily intergranular fracture with some transgranular cleavage; whereas the fracture surface of the heat treated ribbon was primarily transgranular cleavage with some instances of intergranular failure. Although this was the most ductile of the compositions examined, some intergranular failure always occurred in failed specimens.

For both NiAl and FeAl, low ductility was accompanied by primarily intergranular failure, while increases in ductility generally coincided with an increase in transgranular cleavage fracture. Schulson [3] also observed a similar mixture of intergranular and transgranular cleavage fracture in Ni-49Al that exhibited tensile elongations of 14 and 41 percent at 400^{o}C.

One possible explanation for the appearance of ductility and change in fracture mode with heat treatment involves the effects of grain boundary segregation and hardening [11]. These effects could be suppressed by rapid solidification, but reintroduced by subsequent heat treatment. If segregation strengthens the grain boundaries sufficiently, deformation and failure occur through the bulk material instead of along the grain boundaries, and a more ductile failure can result.

SUMMARY OF RESULTS

Melt spinning of NiAl and FeAl produced a heterogeneous ribbon with some variation in both thickness and microstructure. Both Fe-40Al and Fe-45Al possessed some bend ductility in the as spun condition. The bend ductility of Fe-40Al and Fe-45Al increased dramatically with subsequent heat treatment, while Ni-50Al exhibited some increase in ductility with heat treatment. The measured bend ductility itself was a result of the special situation of producing tensile stresses perpendicular to columnar grain boundaries. This resulted in a more severe test than if the grain boundaries had been randomly oriented. Heat treatment at approximately 0.85 Tm also resulted in significant grain growth in equiatomic FeAl and in all of the NiAl compositions. Low bend ductility in both FeAl and NiAl was generally characterized by essentially complete intergranular failure, while increases in transgranular cleavage fracture occurred when the amount of bend ductility increased.

REFERENCES

1. A. Ball and R. E. Smallman: Acta Met. 14 (1966) p. 1349.
2. A. Lawley, J. A. Coll, and R. W. Cahn: Trans. Metall. Soc. AIME, 218 (1960) pp. 166-176.
3. A. G. Rozner and R. J. Wasilewski: J. Inst. Metals, 94 (1966), 930-944.
4. E. M. Schulson and D. R. Barker: Scripta Met. 17 (1983) p. 519.
5. J. D. Whittenberger: Mat. Science and Eng., 57 (1983) pp. 77-85.
6. A. I. Taub, S. C. Huang, and K. M. Chang: Met. Trans., 15A (1984) pp. 399-402.
7. A. Inoue, H. Tomioka, and T. Masumoto: Met. Trans. 14A (1983) p. 1367.
8. D. J. Gaydosh, R. W. Jech, and R. H. Titran: Journal of Mat. Science Letters, In Press.
9. R. W. Jech, T. J. Moore, T. K. Glasgow, and N. W. Orth: Journal of Metals, 36 No. 4 (1984) pp. 41-45.
10. G. E. Dieter: "Mechanical Metallurgy, Second Edition," (McGraw-Hill, Inc., 1976) p. 681.
11. R. Bakish, "Intermetallic Compounds," J. H. Westbrook, ed. (John Wiley and Sons, 1967) p. 298.