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The Structure and Properties of Grain Boundaries in B2 Ordered Alloys

A Progress Report

by

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ABSTRACT

This report covers the period from April 1st 1988 to March 30th 1989. During this period a number of fine-grained alloys of FeAl and NiAl with different aluminum contents were produced by multiple hot extrusion. Thus far, researchers have

1) characterized the extruded microstructures of the alloys;
2) initiated mechanical testing which confirms that with increasing deviation from stoichiometry the yield strength of FeAl decreases while that for NiAl increases and which shows that the grain boundary strengthening in NiAl decreases with increasing deviation from stoichiometry;
3) performed grain growth studies of NiAl and FeAl. For FeAl alloys it was found that grain growth is more rapid in the iron-rich composition;
4) continued an atom probe field ion microscope (APFIM) study of the grain boundary chemistry of NiAl with Dr. M.K. Miller at Oak Ridge National Laboratory;
5) studied boundary chemistry in near-stoichiometric NiAl and FeAl using scanning Auger electron spectroscopy at Oak Ridge National Laboratory, with the aid of Mr. R.A. Padgett, Jr; and
6) initiated in-situ straining experiments in the transmission electron microscope (TEM) to examine dislocation/grain boundary interactions.

INTRODUCTION

A study, funded by the U.S. Department of Energy (grant no. DE-FG02-87ER45311), was initiated on September 1st 1987 to examine the structure and properties of grain boundaries in the B2 ordered alloys FeAl and NiAl. The project is aimed at increasing the understanding of grain boundaries in ordered alloys as a whole. These two compounds were chosen as model materials largely because they exist over a wide range of compositions and can thus be expected to show significant variations in structure and properties over their composition range.

The effect of alloy composition on the structure/chemistry and properties of grain boundaries is being examined:-

1) by measuring yield strength, σ_y; as a function of grain size, d, and, thus, determining the constant k_y in the Hall-Petch relationship, $\sigma_y = \sigma_0 + k_y . d^{-1/2}$ (where σ_0 is the lattice resistance), which is a measure of the ability of the grain boundaries to transmit slip;

2) by measuring grain boundary composition using an APFIM, a scanning Auger electron microprobe (SAM) and a high resolution scanning transmission electron microscope (STEM) at the Oak Ridge National Laboratory;

3) by examining the dislocations in the grain boundaries of lightly-strained compression samples using (TEM);

4) by examining dislocation/grain boundary interactions during in-situ straining in a TEM;

5) by measuring grain growth kinetics;

The progress in each of these areas is reviewed overleaf. First, though, the results of material processing are outlined.

MATERIAL PROCESSING

All alloys have been characterized microstructurally after first, second and third extrusions, using optical microscopy, transmission electron microscopy and hardness measurements. The microstructure following the first two extrusions of the FeAl alloys has been described in a paper to be published in the Journal of Materials Science.

Briefly, the FeAl alloys had equiaxed grains after the first extrusion at 1000°C containing few dislocations. After the second and third extrusions at temperatures from 700°C to 750°C, the alloys showed some evidence of an elongated grain structure in longitudinal sections in a few regions, see figure 1. TEM observations of transverse sections found evidence of a <111> fiber texture and hardness measurements indicated consistently higher hardness values on longitudinal sections than on transverse sections, see Table 1. The texture will be characterized more fully by x-ray measurements. TEM examination also showed that the the second and third extrusions contained numerous dislocations. The dislocations mostly had <100> Burgers' vectors except in the iron-rich alloys where they were <111>, see figure 2. It was found that the residual dislocations could be removed by annealing at ~775°C for 24 hours; a heat treatment which does not result in significant grain growth. This heat treatment is being performed on samples prior to performing in-situ straining experiments in the TEM and mechanical testing.

All NiAl alloys were successfully extruded at 1000°C except Ni-55Al. The microstructure of the NiAl alloys after the first extrusion consisted of recrystallized equiaxed grains containing a few <001> dislocations. However, after the second and third extrusion at ~750°C the microstructure had only partially recrystallized (note Ni-42Al has not been successfully extruded for a third time thus far): no evidence of a texture was found after subsequent recrystallization and hardness values are similar on transverse and longitudinal sections, see Table 2. The residual dislocations in all the alloys except the most nickel-rich (Ni-42 at.%) alloy had <001> Burgers vectors. In the most nickel-rich NiAl dislocations of both b = <001> (together with some <011> junction dislocations) and b = <111> were observed, this significant observation has been described in a paper published in Scripta Metallurgica.

The observation of <111> slip dislocations in polycrystalline NiAl was unexpected, had not been observed before and clearly warranted further investigation. Thus, TEM examination of Ni-42Al after straining 2% in compression at room temperature was performed. This revealed that all the dislocations present had <001> Burgers vectors, that is, no <111> slip was observed. Interestingly, this alloy could not be electropolished after straining and was observed to pit, whereas in the as-extruded form it could be electropolished easily: several polishing solutions were used, none successfully. It was thought that the alloy may have undergone a strain-induced martensitic transformation, but no martensite was observed after foil preparation by ion-beam thinning.

YIELD STRENGTH MEASUREMENTS

In the previous progress report, it was noted that except for Fe-34Al as-received single extruded FeAl had a much lower yield strength than annealed FeAl (of a much larger grain size). this has been investigated further and it was found that the above effect arose due to differences in cooling rate. Figure 3, shows the hardness of discs of FeAl after annealing at 1000°C and either water quenching, air cooling or furnace cooling. For Fe-40Al, Fe-45Al and Fe-50Al no differences in hardness between water-quenched and air-cooled samples were found but the hardness values were significantly greater than furnace-cooled samples. Air cooling gave a greater hardness in Fe-51Al but not as great as water quenching, and the cooling rate has only a smaller effect on the hardness of Fe-34Al. The cause of this cooling rate effect may be retained vacancies. All samples of FeAl are now furnace cooled prior to mechanical testing. Even so, after yield strength measurements on approximately 20 FeAl samples, the results clearly show that the strength changes with composition, but the scatter in data have not allowed any effect of grain size on yield strength to be discerned. However, it is clear that the grain size strengthening effect in FeAl is small.

The effect of cooling rate on the hardness of NiAl after annealing at 1000°C was also examined. Whilst the hardness was greater after water quenching, no increase in hardness was observed after air cooling. Preliminary yield strength measurements on eight samples of NiAl (on Ni-45Al, Ni-48Al and Ni-50Al) have indicated that the grain size strengthening effect decreases with increasing deviation from stoichiometry, see figure 4. Further work is needed before this observation is confirmed.

RECRYSTALLIZATION AND GRAIN GROWTH KINETICS

The recrystallization and grain growth kinetics of Ni-45Al, Ni-48Al and Ni-50Al have been measured using optical microscopy after the third extrusion at a number of temperatures. However, no significant differences in either process have been observed.

By comparison, determination of the grain growth kinetics for double-extruded FeAl at 900°C and 1200°C has shown that grain growth rate increases with decreasing Al content. (Note that, although the melting temperature of FeAl increases with decreasing Al content, that is, annealing was performed at a lower homologous temperature with decreasing Al content, grain growth still occurs faster in more iron-rich alloys.) These results have been reported in a paper presented at the 1988 Fall MRS meeting

Measurement of grain growth kinetics on triple-extruded FeAl is now underway.

ATOM PROBE FIELD ION MICROSCOPY

APFIM work has continued with Ph.D. candidate Pavan Nagpal performing initial sample preparation at Dartmouth College and performing final sample preparation and examination at ORNL. The next trip to ORNL will be in June. Travel expenses and accommodation associated with this work have been covered by ORAU's SHaRE Program.

TEM IN-SITU STRAINING

A 'Tenupol' holder has been modified to allow the preparation of thin foils for in-situ deformation in the TEM. All the successfully-extruded alloys except Fe-51 at.%Al have been ground into an appropriate form for in-situ deformation: Fe-51 at.%Al proved to be too brittle. In-situ straining samples of the most iron-rich alloy Fe-34Al have been prepared and strained. During straining, transgranular cracks were observed to grow approximately perpendicular to the straining direction . <111> dislocations have been observed to glide toward grain boundaries, but none have been observed to be transmitted across them thus far, see figure 5.

In one particularly interesting area a large number of dislocations were observed at a small crack at the edge of the foil; the crack did not grow during straining (other cracks grew rapidly) but the dislocation density around the crack decreased. Dislocations were observed to glide toward both the crack and the foil edge.

AUGER ELECTRON SPECTROSCOPY

So far, two alloys have been examined, Ni-49Al and Fe-45Al. These are the only compositions out of the range of alloys being studied which fracture by a mixture of transgranular cleavage and intergranular fracture at room temperature. Comparison of Auger spectra from intergranular and transgranular surfaces, after in-situ fracture in the SAM, found that the grain boundary composition was identical to that of the matrix in both cases, see figure 6 for example.

It has recently been determined that both NiAl and FeAl alloys containing lower levels of aluminum than those noted above can be made to fracture in a mixed mode at liquid nitrogen temperatures. Samples are now being prepared from compositions further from stoichiometry, than Ni-49Al and Fe-45Al, for examination in the SAM after in-situ fracture at liquid nitrogen temperatures.

OXIDATION KINETICS

The oxidation kinetics of FeAl have been briefly examined at 900°C and 1200°C. At 900°C, little oxidation occurs. At 1200°C, oxidation is still relatively slow but the rate increases with decreasing aluminum concentration. Interestingly, the hardness of the FeAl below the oxidation layer changes with oxidation time (it decreases for all compositions except Fe-34Al) in a manner which is consistent with a depletion of aluminum near the surface. These results have also been reported in a paper presented at the 1988 MRS meeting.

PERSONNEL

The following personnel were supported during the previous budget period:-

Ian Baker - P.I.

Paul R. Munroe - Research Assistant Professor

Pavan Nagpal - Ph.D. candidate

Pavan Nagpal has completed his Ph.D. qualifying exams. Bernd Schmidt, whose thesis topic was "Annealing Studies of FeAl", was awarded a B.E. in June 1988. Two students, Fuping Liu and Jianxing Fang, worked on this project as part of their first year doctoral work (prior to starting their thesis project).

PAPERS PUBLISHED OR IN PRESS DURING YEAR -2

"Structural Intermetallic Compounds", P.R. Munroe and I. Baker, Metals and Materials, 4 (1988) 435.

"On Grain Boundary Disorder and the Tensile Ductility of Polycrystalline Ordered Alloys : a Hypothesis", I. Baker and E.M. Schulson, Scripta Met., 23 (1989) 345.

"Observation of <111> Slip in NiAl", P.R. Munroe and I. Baker, Scripta Met. 23 (1989) 495.

"Grain Boundary Chemistry of NiAl", P.P. Camus, I. Baker, J.A. Horton and M.K. Miller, J. de Physique 49 C6 (1988) 329.

"The Microstructure of Extruded FeAl", P.R. Munroe and I. Baker, J. Mat. Sci. (in press).

"Annealing Studies of B2 FeAl", B. Schmidt, P. Nagpal* and I. Baker, 'High Temperature Ordered Intermetallic Alloys' MRS Symp. Proc. (1989) (in press).

PAPERS TO BE PRESENTED

The following two papers are to be presented at conferences and published in the relevant proceedings:

"Dislocation Structures in Ordered B2 Alloys", I. Baker, 47th Proc. EMSA, San Antonio, TX, August 1989.**

"A Review of the Properties of B2 Ordered Alloys", I. Baker, Symposium on 'High Temperature Aluminides and Intermetallics', Fall TMS meeting, Indianapolis, IN, October 1989.**

* Presented as a poster by P. Nagpal.

** Review paper, partly funded by DOE.

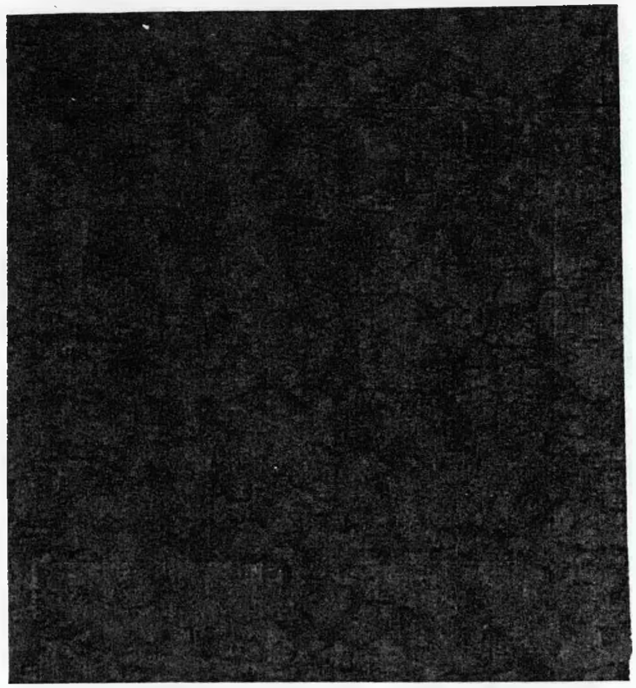

Figure 1. Optical micrograph of the elongated grain structure observed in some longitudinal sections of Fe-51Al after the third extrusion.

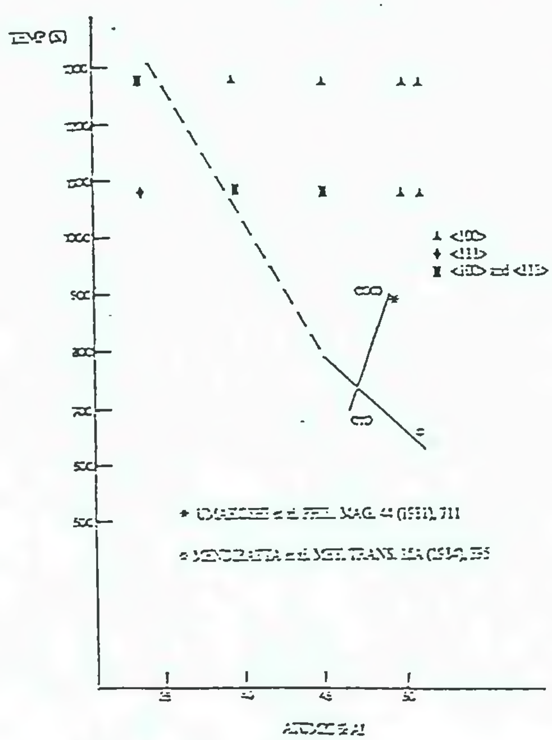

Figure 2. Graph showing the Burgers' vectors of dislocations in FeAl for different temperatures and aluminum contents. The two lines indicate the boundaries previously observed by other workers between <111> slip at low temperature and <100> slip at high temperature.

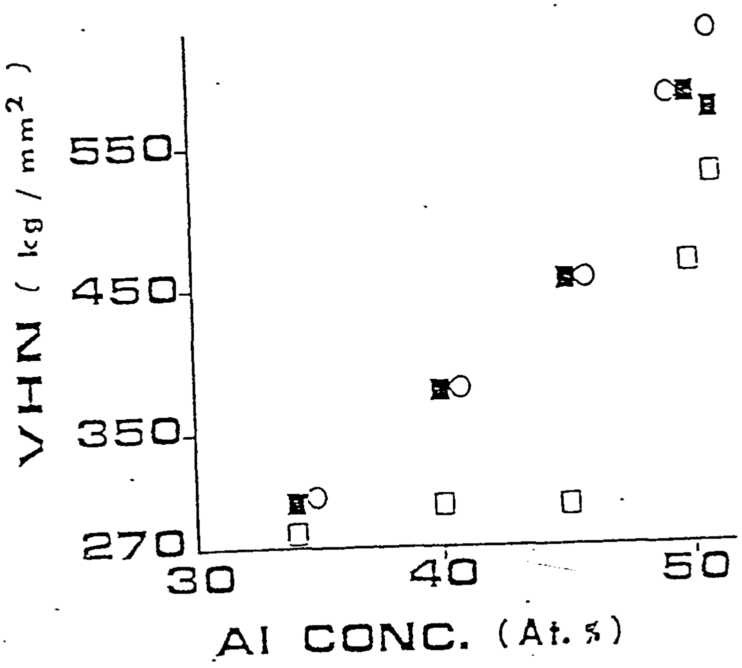

Figure 3. Hardness as a function of composition for discs of FeAl annealed at 1000°C and (○) water-quenched, (■) air-cooled and (□) furnace-cooled.

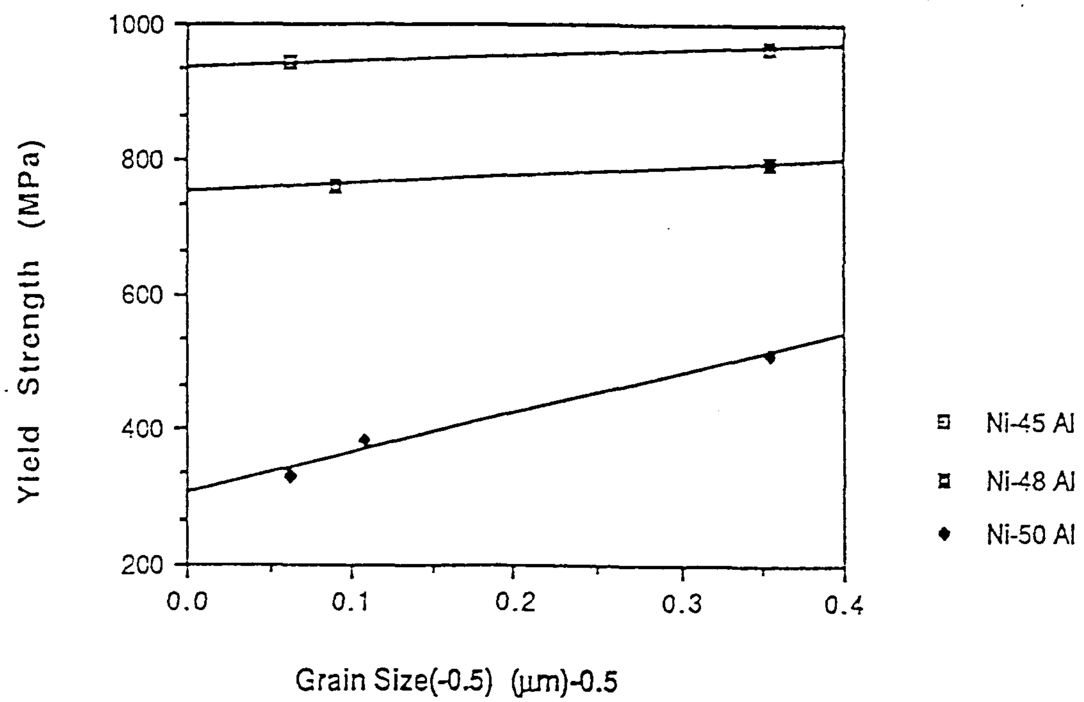

Figure 4. Yield strength as a function of grain size for three different compositions of NiAl.

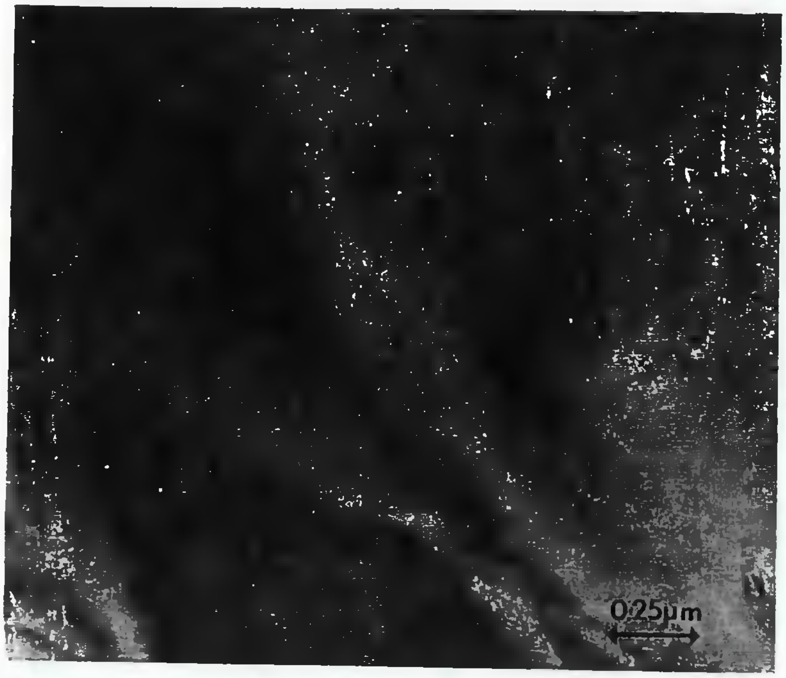

Figure 5. Bright-field transmission electron micrograph of in-situ strained Fe-34Al.

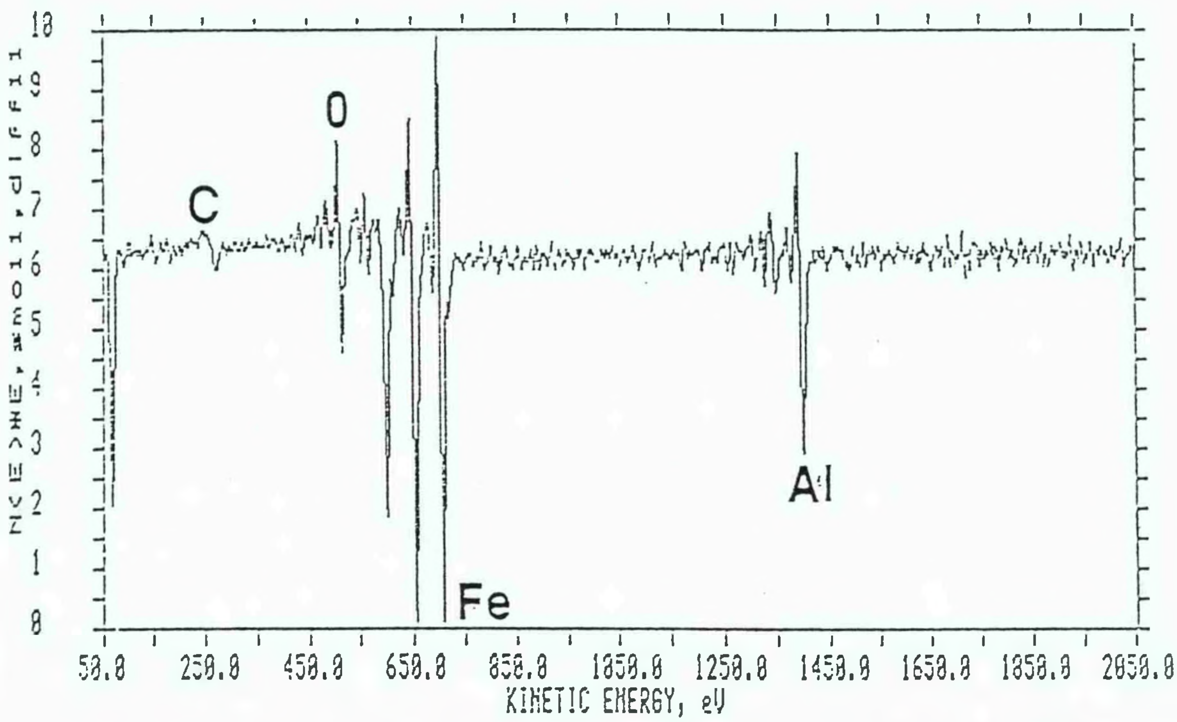

Figure 6. Auger electron spectrum from fracture surface of Fe-45Al .

Composition	Section	Grain Size (μm)	Hardness (R_A)
Fe-34Al	Transverse	13	60.3
	Longitudinal	14	61.5
Fe-40Al	Transverse	8	64.8
	Longitudinal	8	66.5
Fe-45Al	Transverse	11	67.3
	Longitudinal	11	68.3
Fe-50Al	Transverse	10	73.7
	Longitudinal	12	74.5
Fe-51Al	Transverse	11	75.7
	Longitudinal	12	76.4

TABLE 1. Grain Size and Rockwell Hardness of As-received Triple-extruded FeAl

Composition	Section	Hardness (R_A)
Ni-45Al	Transverse	69.8
	Longitudinal	71.3
Ni-48Al	Transverse	69.3
	Longitudinal	69.1
Ni-50Al	Transverse	59.2
	Longitudinal	59.9

TABLE 2. Rockwell Hardness of As-received Triple-extruded NiAl