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The Hall-Petch Relationship and Mechanisms of Fracture in B2 Compounds

Three-Year Progress Report

by

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MASTER

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INTRODUCTION

A study, funded by the 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 was aimed at increasing the understanding of grain boundaries in ordered alloys as a whole, and more specifically that of B2 compounds. This project continued from September 1st, 1990, with initial emphasis on CoAl, FeCo, and CuZn but with continued work on FeAl and NiAl. FeCo and CuZn are of interest since they exhibit order/disorder transformations. They were chosen as model materials because they exist over a wide range of compositions and can be expected to show significant variations in structure and properties over their composition range.

For these materials the effect of alloy composition on the structure/chemistry and properties of grain boundaries were examined by:

- 1) measuring the yield strength, σ_y , as a function of grain size, d , and, thus, determining the constant k in the Hall-Petch relationship, $\sigma_y = \sigma_0 + k d^{-1/2}$, where σ_0 is the lattice resistance;
- 2) measuring grain boundary composition using a scanning Auger electron microprobe (SAM);
- 3) examining dislocation/grain boundary interactions during *in-situ* straining in a TEM;
- 4) determining fracture modes, and assessing the extent of strain at the fracture surfaces using selected area channeling patterns (SACP's).

The progress during the last three years is reviewed below.

MATERIALS PROCESSING

Ingots (150 mm long; 50 mm dia.) of compositions shown below (in at.%) were cast from high-purity materials at the UTRC, East Hartford, CT, through the assistance of Dr. D. Anton:-

Co-47Al; Co-48.5Al; Co-50Al; Co-52Al

Fe-70Co; Fe-50Co; Fe-30Co

Fe-40Al; Fe-43Al; Fe-45Al; Fe-48Al; Fe-50Al

Fe-40Al-5Cr; Fe-45Al-0.05; BFe-45Al + 500 wt. ppm B

Ni-50Al; Ni-50Al-0.1B

Four inch sections of each of the ingots were canned in stainless steel and shipped to NASA-Lewis Research Center where, through the assistance of Dr. J.D. Whittenberger, they were extruded either once or twice. Extrusions of CoAl, Fe-45Al + 500 wt. ppm B and Ni-50Al-0.1B have not been successful, the resulting rods appeared to have suffered localized melting or contained numerous cracks. Large grain-sized specimens were prepared from homogenized (by annealing) sections of the remaining ingots.

The microstructures in the FeCo alloys, extruded at 1000°C, then re-extruded at 750°C, were examined in some detail, both optically and by transmission electron microscopy. It was found that increasing the Fe:Co ratio produces less complete recrystallization: Fe-70 at.%Co was completely recrystallized after both the 1000°C and 750°C extrusions, whilst Fe-30 at.%Co was partially recrystallized even after the 1000°C extrusion. Single dislocations were present in the extruded alloys except single-extruded Fe-30 at.%Co, in which dislocation pairs were observed.

The β -brass alloys Cu-46.3Zn Cu-48.8Zn and Cu-49.8Zn were cast, hot-rolled to 75mm by 300mm by 12.5mm sheets and chemically analyzed at the Metals Research Laboratories of the Olin Corporation, 91 Shelton Avenue, New Haven, Conn., through the assistance of Mr. P. Renschen.

FLOW AND FRACTURE OF FeCo

Compression tests were performed to study the relationship between the Hall-Petch parameter and the degree of order in the three FeCo alloys. Observations of surface slip traces showed that all the alloys exhibited wavy slip in their disordered states whilst planar slip occurred in the ordered states. Table 1 shows the largest grain size at which discontinuous yielding was observed for each alloy in the ordered and disordered states. Discontinuous yielding occurred at larger grain sizes in the ordered state than in the disordered state, and the largest grain size at which discontinuous yielding was observed was in the stoichiometric composition. These observations were rationalized by considering the work-hardening rate and the Hall-Petch slope, see Table 2.

Table 1 Largest grain size in which discontinuous yielding occurred.

Composition State	Fe-70 at.%Co	Fe-50 at.%Co	Fe-30 at.%Co
ordered	39 μm	170 μm	30 μm
disordered	20 μm	27 μm	not observed even in finest grain size, 25 μm .

The stoichiometric alloy had a higher yield strength in the disordered state than in the ordered state, irrespective of grain size whilst the cobalt-rich alloy Fe-70 at.% Co had a lower yield strength in the disordered state than in the ordered state, irrespective of grain size. In contrast, the iron-rich alloy Fe-30 at.% Co was stronger in the ordered state at fine grain sizes but weaker at coarse grain sizes. It was found that both compositional and thermal disordering decrease the Hall-Petch parameter although it has different effects on the lattice resistance: disordering increased σ_0 for Fe-50 at.% Co and Fe-30 at.% Co but decreased σ_0 for Fe-70 at.% Co, see Table 2.

Table 2 Values of k , σ_0 and work hardening rate in FeCo alloys.

composition	Fe-70 at.%Co		Fe-50 at.%Co		Fe-30 at.%Co	
state	ordered	disordered	ordered	disordered	ordered	disordered
k (MPa $\mu\text{m}^{1/2}$)	458	353	806	456	724	141
σ_0 (MPa)	404	261	172	309	149	237
W.H. rate (GPa)	4.3	6.5	7.6	6.7	7.7	5.8

Tensile tests were performed on the FeCo alloys and the resulting fracture surfaces examined in an SEM. It was found that ordered Fe-50 at.% Co and Fe-30 at.% Co were very

brittle, fracture occurring before yielding, with intergranular fracture occurring in most grains. In contrast, ordered Fe-70 at.% Co. showed about 18% elongation and exhibited a dimple-type fracture. It was also found that disordering improved the ductility of each composition, see Figure 1, but had little influence on the fracture mode.

Fig.1 Elongations of FeCo tensile samples.

TEM examinations showed that dislocation pairs were present in each ordered alloy deformed to about 2% strain. Slip trace observations showed that wavy slip was present in disordered FeCo whilst planar slip occurred in the ordered alloys, behavior which was consistent with the observed dislocations.

Combining the results of compression and tensile tests, it can be concluded that disorder always decreases the Hall-Petch parameter and improves ductility for FeCo.

AUGER ELECTRON SPECTROSCOPY

Auger electron spectroscopy studies of FeCo were initiated with Dr. E.P. George at ORNL and are still ongoing. (The work is co-funded by the SHaRE Program of Oak Ridge Associated Universities.)

The aim is to determine if

- i) the grain boundary Fe:Co ratio is different from the matrix,
- ii) ordering changes the Auger electron sensitivity factors,
- iii) any impurities have segregated to the grain boundaries, and
- iv) the impurity segregation (if present) depends on the degree of order.

The overall aim is to explain the observed flow and fracture behavior of FeCo described above. Thus far, our results have shown that the Fe:Co ratio on the grain boundaries and in the matrix is the same. We have clear evidence of sulfur segregation to the grain boundaries in stoichiometric and iron-rich FeCo; no data is available on cobalt-rich FeCo since this does not fracture intergranularly. However, since this alloy was produced from the same starting materials as the other alloys then similar segregation of sulfur would be expected. We have shown that intergranular fracture occurs at liquid nitrogen temperatures and so AES will be performed after *in-situ* fracture at this temperature to prove this point. This raises the question, why does sulfur embrittle some compositions but not others?

The segregation behavior also depends on the heat treatment. Surprisingly, the segregation was greater after a higher temperature anneal (followed by a quench) which produced the disordered state. This is so, even though the disordered state showed more ductility. Further work is underway to investigate why adding 2 wt.% V improves the ductility. The vanadium addition appears to improve the ductility but appears to impart environmental sensitivity to the alloy. Again, a point of further investigation.

TENSILE TESTING OF CuZn

The effect of strain rate and environment were examined at 23°C for stoichiometric CuZn. The yield stress and elongation at room temperature show little environmental effect; the yield stress and elongation obtained are 60 MPa, 18% and 69 MPa, 22% for in air and in vacuum, respectively, at $1 \times 10^{-4} \text{s}^{-1}$. At the higher temperature (300°C), the elongation of the specimen which was tested in vacuum increased from 90% in air to 120% in vacuum while the yield stress

increased from 15 MPa in vacuum to 29 MPa in air. The yield stress increased less with increasing strain rate at both room temperature and 300°C. For strain rates less than $1 \times 10^{-4} \text{s}^{-1}$, the yield stress decreases slowly. The strain rate had little effect on elongation at room temperature but at 300°C, the elongation decreased at the highest strain rate.

IN-SITU STRAINING EXPERIMENTS

Samples of a range of compositions of NiAl and FeAl, and of the B2 + f.c.c/L1₂ alloy Ni-30Fe-20Al were studied by TEM *in-situ* straining experiments. The work was performed in collaboration with Dr. J.A. Horton at ORNL through the SHaRE Program. Experiments performed at 300 keV in a Philips 430 TEM at ORNL showed that FeAl undergoes radiation damage at this electron energy.

In-situ straining experiments on FeAl could not find any differences in slip or crack growth behavior in the thin foil with changes in Fe:Al ratio. Dislocation activity is intense during *in-situ* straining. Dislocations are emitted all along the thin foil edge, run into the grain boundary and pass through easily. Surprisingly, dislocation debris is left behind the gliding dislocations (c.f., Ni₃Al) even though no dislocation interactions can be observed. Crack growth did depend on prior deformation. In initially crack-free thin foils significant dislocation activity occurred before a crack grew down a thinned slip band. These cracks were quite blunt. On the other hand, cracks present after specimen preparation which grew into largely dislocation-free material were quite sharp.

In-situ straining of stoichiometric NiAl produced a cell structure very similar to that found in bulk-strained samples, indicating very easy cross-slip. Interestingly, even though in bulk stoichiometric NiAl fractures intergranularly, during thin foil deformation cracks start at grain boundaries, then blunt and run through the matrix. *In-situ* straining of off-stoichiometric NiAl showed planar slip and transgranular cleavage. The clear change in dislocation behavior ties in well with the observed bulk flow and fracture behavior. Loop debris was observed in both cases but was more prevalent in stoichiometric NiAl.

In-situ straining of Ni-30Al-20Fe found evidence of slip transmission both from the f.c.c. phase containing fine L1₂ precipitates to the B2 phase and vice-versa. Loops were formed in the B2 phase during glide of <100> dislocations. These appear to have formed by a double cross-slip mechanism since they formed on dislocations which appeared not to have interacted with other dislocations. Slip plane disordering was observed to occur in the f.c.c. phase due to cutting of the L1₂ precipitates. No loops were formed in this phase, presumably due to the lack of cross-slip.

FRACTURE OF FeAl/NiAl/CoAl

A series of studies on FeAl and NiAl have shown that in both cases room temperature fracture is intergranular at the stoichiometric composition with an increasing amount of transgranular cleavage with increasing deviation from stoichiometry. In contrast, CoAl shows intergranular fracture irrespective of composition. Selected area channelling patterns (SACP's) at 200 keV could be obtained from fracture surfaces of all NiAl and CoAl compositions, the quality decreasing with increasing deviation from stoichiometry for NiAl but did not vary with composition for CoAl. For FeAl, SACP's could only be obtained from the stoichiometric composition. It has also been observed that the fracture strains of FeAl increase with increasing deviation from stoichiometry whilst those for NiAl decrease with increasing deviation from stoichiometry. For CoAl, fracture occurred before any evidence of plastic strain irrespective of composition.

ENVIRONMENTAL EFFECTS ON FeAl

Liu and George, Scripta Metall., 24, 1285 (1990), and Gaydosch and Nathal, Scripta Metall., 24, 1281 (1990), demonstrated an environmental effect in Fe-40 at. % Al, which showed higher ductility (~5 %) in vacuum in comparison with tests performed in air (~1%). Liu and George suggested that the embrittlement is due to the atomic hydrogen arising from a reaction between water vapor and aluminum atoms, which produces Al₂O₃. The idea is that hydrogen atoms diffuse to the

crack tip and thus reduce the stress to fracture. Since hydrogen diffusion is time-dependent, a strain-rate dependence of the ductility is expected. This was examined in Fe-45Al and Fe-40Al-5Cr.

Figure 2 shows the strain to failure versus strain rate for Fe-45Al and Fe-40Al-5Cr. For Fe-45Al, at strain rates up to $1 \times 10^{-2} \text{ s}^{-1}$ elongations of 2 to 3% were obtained, independent of strain rate. For strain rates greater than $1 \times 10^{-2} \text{ s}^{-1}$, as the strain rate increased the ductility increased, and the highest elongation (9%) was obtained for the strain rate of 1 s^{-1} . For Fe-40Al-5Cr straining at rates up to $1 \times 10^{-3} \text{ s}^{-1}$ gave ~2% elongation. Whereas for higher strain rates, increases in strain rate gave greater elongations. The fracture mode was mainly intergranular for both alloys, independent of strain rate. The yield stress was independent of strain rate for both alloys. The results appear to confirm the suggestion by Liu and George that the environmental embrittlement of FeAl is due to diffusion-assisted processes.

Figure 2. Graph of strain to failure versus strain rate for Fe-45Al and Fe-40Al-5Cr.

Fracture toughness tests were performed on FeAl at different loading rates. Values of $\sim 20 \text{ MPam}^{1/2}$ were obtained at slow loading rates whereas values of $\sim 30 \text{ MPam}^{1/2}$ were obtained at higher loading rates. This difference is consistent with the results of the tensile tests that there is an environmental effect.

TENSILE BEHAVIOR OF POLYCRYSTALLINE STOICHIOMETRIC NiAl

The room temperature yield strength and fracture strain of stoichiometric NiAl were examined as a function of grain size under tension. It was found that over the grain size range examined, $13\mu\text{m}$ to $270\mu\text{m}$, the yield strength of NiAl is independent of grain size, consistent with similar earlier studies performed in compression, see Figure 3. One difference was that the yield strengths in tension are consistently lower than those in compression by around 10-20 MPa, which is ~ 10 to 20% of the yield strength.

Figure 3. Graph of yield stress versus grain size for stoichiometric NiAl.

The fracture strains, at 1-2%, were essentially independent of grain size also, at least over the grain size range examined, see Figure 4.

Figure 4. Graph of strain to failure under tension versus grain size for stoichiometric NiAl.

FRACTURE MECHANISM

Previously, a mechanism for producing $\langle 100 \rangle$ dislocations by the collision of two different gliding $\langle 111 \rangle$ dislocations was presented for binary FeAl (P.R. Munroe and I. Baker, *Acta Metallurgica et Materialia*, 39 (1991) 1011-1017). The resulting $\langle 100 \rangle$ dislocations lie on a $\{001\}$ and had a coat-hanger shape. Further work has shown the presence of these dislocations in room temperature deformed FeAl containing Cr and in DO_{19} -structured Fe-34Al and Fe-28Al+6Cr. Work is underway looking for the presence of such dislocations in FeCo, which also deforms by $\langle 111 \rangle$ slip.

PERSONNEL

The following personnel were supported during this budget period:-

Ian Baker - P.I.

Lie Zhao - M.S. awarded (1992); Ph.D. candidate

Oliver Klein - M.S. candidate, expected completion date: March 1993

H. Xiao - M.S. candidate expected completion date: June 1993

P. Nagpal - Ph.D. awarded (1992)

PAPEK, PUBLISHED UNDER DOE SUPPORT DURING THE LAST THREE YEARS

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"Hard Sphere Modelling of Interstitial Sites in B2 Compounds", I. Baker, submitted to Journal of Materials Research.

"TEM *In situ* Straining of NiAl", P. Nagpal, I. Baker and J.A. Horton, submitted to Philosophical Magazine.

"Extrusion processing of FeCo", L. Zhao and I. Baker, submitted to Journal of Materials Science.

"The Effect of Grain Size and Fe:Co ratio on the Room Temperature Flow of Polycrystalline FeCo", L. Zhao and I. Baker, submitted to Acta Metallurgica et Materialia.

PRESENTATIONS

- * "Heat Treatment and Processing of Nickel Aluminides", I. Baker, ASM Symposium on Heat Treatment and Annealing of Ordered Alloys, Detroit, MI, Fall 1990.

"Observation of $\langle 001 \rangle$ Dislocations in B2-Structured FeAl", P.R. Munroe and I. Baker, TMS Fall meeting, Detroit, MI, Oct. 1990.

The research on FeAl and NiAl was outlined at invited presentations at:

Oregon Graduate Center (1990),
United Technologies Research Center (1990),
Sherritt - Gordon Ltd. (1990),
Oak Ridge National Laboratory (1992),
Monash University (1992),
University of Auckland (1992), Rolls Royce (1992),
University of Sheffield (1992),
University of Pittsburgh (1992).

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