The Yield Strength Anomaly and the Environmental Effect in FeAl

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by

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DISCLAIMER

INTRODUCTION

The aim of the work here was to understand both the yield anomaly in FeAl, which was first noted by the P.I. and collaborators, and the effect of environment on the fracture of FeAl. The progress in these areas is outlined below. A model for the yield anomaly developed as part of this work served as the basis for a successful proposal to NSF to study the yield anomaly in other B2 compounds. The effects of vacancies and of boron on flow and fracture at room temperature were also addressed. Recrystallization and grain growth were studied in both FeAl and Ni_3Al. Strain-induced ferromagnetism was studied in FeAl and a model for the paramagnetic-to-ferromagnetic transition was developed based on antiphase boundary tubes. A successful proposal was also submitted to NSF on this topic. Finally, in addition to a number of papers and presentations on our experimental work, several invited presentations and published reviews, including a major review in International Material Reviews, on the mechanical properties of either FeAl or B2 compounds were made.

The Yield Anomaly

A phenomenological model was developed, with Dr. E.P. George of ORNL, to explain the yield strength anomaly in FeAl. The model incorporates hardening (frictional drag) by thermal vacancies at intermediate temperatures, and dislocation creep at elevated temperatures. Since the vacancy concentration increases exponentially with increasing temperature, the model predicts an exponential increase in strength with increasing temperature. This increasing strength is terminated by the onset of dislocation creep. The model captures the experimentally-observed strain rate dependency of the yield stress at high temperatures, and yields an activation enthalpy for vacancy formation which is in excellent agreement with a previously measured value.

One prediction of the model was that the yield anomaly should be observable even in stoichiometric FeAl (where it had not been observed previously) during rapid straining. We demonstrated that, at a strain rate of 1.6 s^{-1}, a yield peak is observed in both polycrystals and single crystals, whereas at a strain rate of 1×10^{-4} s^{-1} a slow decrease in yield stress was observed with increasing temperature. Compression tests (on polycrystals) at very high strain rates (2000 s^{-1}) caused the yield stress peak to shift to higher temperatures (1100 K) and stresses (1100 MPa).

Fracture

The effects of environment (moisture-induced hydrogen) on fracture strain and yield strength were studied in single-slip-oriented, iron-rich FeAl single crystals. During tensile tests, large fracture strains (>40%) were found when tests were conducted in oxygen. In contrast, in air, elongations were <10%, whereas in vacuum typical elongations were ~20-30%. Cathodic hydrogen charging had little effect on the ductility of crystals tested in air. Intermittent straining in air produced more ductility than continuous straining. Tests performed in air showed lower yield strengths than tests performed in vacuum. The yield strength in air increased with increasing strain rate up to 1×10^{-2} s^{-1}, when it was the same as in vacuum. Similarly, in interrupted tests, in which straining was performed alternately in either air or vacuum, higher flow stresses were observed under vacuum than in air. These effects could be explained by the effects of moisture-induced hydrogen.

Tensile tests performed in air and vacuum on Fe-40Al single crystals oriented so that predominately edge or screw dislocations accommodated the plastic strain showed that for both orientations that ductility was less in air than in vacuum. However, no discernable correlation was found between the character of the dislocations, and the effect of environment.

In both air and vacuum, the room-temperature fracture strengths of Fe-40Al single crystals are significantly increased, but the elongations are decreased by introducing a high vacancy concentration. The fracture mode was also found to be controlled by the vacancy concentration: with a low vacancy concentration, fracture is through cleavage on both {100} and a variety of other planes, whereas at high vacancy concentration, cleavage occurs mainly on {100} and {110}. The later fracture mode suggests that vacancies promote fracture along the slip planes.

Vacancy Effects

Single-slip-oriented single crystals and polycrystals of Fe-40Al were strained in compression at ~1×10^{-4} s^{-1} at room temperature after quenching-in different vacancy concentrations (C_v), and it was found that:

1. The yield stress increased approximately linearly with increasing C_v by ~350% up to C_v ~4×10^{-3}. This coincided with coarse [111] slip on {110}.

2. At higher C_v (> 4×10^{-3}) a much slower increase in strength occurred, a feature which coincided with the onset of slip on {211}.

3. The data suggest that vacancies harden FeAl by frictional strengthening and that this is much greater on {110} than on {211}.

4. The work hardening rate increased only slightly with increasing vacancy concentration.

5. Numerous dislocation loops, debris and pinning points are produced by slip.

Boron Effects

X-ray studies and mechanical tests were performed on low-temperature annealed FeAl with and without boron and, together with analysis of other data, it was shown that:

1. Boron does not affect the order, vacancy concentration or anti-site atom concentration in FeAl.

2. Boron increases the Hall-Petch Slope.

3. Boron increases the temperature and magnitude of the yield stress peak in FeAl.

4. The strengthening effect of boron in FeAl is roughly independent of temperature from 300K to 700K, above which it rises rapidly.

5. The compositional dependence of the effect of boron on the strength of FeAl depends on whether vacancies are present: for FeAl containing few vacancies (□ 45 at. % Al) the strength increase per atomic percent boron increases with increasing aluminum concentration; when vacancies are present (□ 48 at. % Al), boron strengthening shows little change with aluminum concentration, suggesting that the vacancies significantly interact with boron.

6. The strength increase due to boron per unit increase in lattice parameter at room temperature is 0.22G - 0.80G depending on the aluminum concentration, is an order of magnitude greater than the boron strengthening of nickel-based $L1_2$ compounds.

Strain-Induced Ferromagnetism

A calorimetric study, through continuous heating, of a cold-rolled Fe-40Al single crystal showed three exothermic peaks, with the peak magnitudes and temperatures depending on the rolling strain. The lowest temperature (440-550K) exothermic peak appears to be associated with a disorder-order transition, probably associated with the removal of APB tubes. At strains > 14%, a transition from paramagnetism to ferromagnetism was observed at room temperature after deformation, which disappeared after annealing above the lowest temperature exothermic peak. However, the annealed specimen still showed enhanced magnetic susceptibility at temperatures below 225K compared to the unstrained crystal. Analysis suggested both that the magnetic transition at *room temperature* is not from the APB's between two a/2<111> partial dislocations but due to the formation of a high density of APB tubes, and that both types of APB contribute to ferromagnetism in off-stoichiometric FeAl, with the greater contribution from APB tubes. A model for this behavior was developed. Further, less detailed measurements were performed on FeAl single crystals of other compositions.

Recrystallization

The recrystrallization and subsequent grain growth of FeAl and Ni3Al both with and without boron were studied using a hardness indentation technique, and it was shown both that the recrystallization kinetics of FeAl alloys correlate with the degree of constitutional order, i.e. with increasing degree of order the recrystallization temperature increases and the grain growth rate decreases; and that boron retards recrystallization in both FeAl and Ni_3Al, leading to slower grain growth in FeAl, but not in Ni_3Al.

PERSONNEL

Supported by funding from this grant, Yong Yang completed his Ph.D. in June, 1999 with a thesis entitled "Mechanical and Magnetic Properties of Single Crystals of FeAl". He is currently employed as a Research Engineer with Hypertherm, Inc of Hanover, NH, the World's leading maker of plasma cutting torches. Dr. X. Pierron was supported as a Post-Doctoral Fellow until May, 1997, when he joined Special Metals, Inc of New Hartford, NY as a Senior Research Engineer. Markus Wittman, Dongmei Wu (Ph.D. candidates) and the P.I. were all partly supported by the grant.

REFEREED JOURNAL ARTICLES

1. "Recrystallization of FeAl and Ni_3Al with and without boron", Y. Yang and I. Baker, Scripta Metallurgica et Materialia, **34** (1996) 803-807.

2. "The Effect of Boron on the Hall-Petch Behavior of Fe-45Al", X. Li and I. Baker, Scripta Metallurgica et Materialia, **34** (1996) 1219-1223.

3. "On the Yield strength Anomaly in Stoichiometric FeAl", I. Baker and Y. Yang, Materials Science and Engineering, **A239-240** (1997) 109-117.

4. "The Effect of Strain Rate on the Yield Strength Peak in Fe-45Al", X. Li and I. Baker, Scripta Metallurgica et Materialia, **36** (1997) 1387-1390.

5. "The Mechanical Properties of FeAl", I. Baker and P.R. Munroe, International Materials Reviews, **42** (1997) 181-205. *(Invited)*

6. "The Influence of Vacancy Concentration on the Yield and Flow Behavior of Fe-40Al", Y. Yang and I. Baker, Intermetallics, **6** (1998) 167-175.

7. "The Strengthening Effect of Boron as a function of Aluminum Concentration in FeAl", I. Baker, X. Li, H. Xiao, R. L. Carleton and E. P. George, Intermetallics, **6** (1998) 177-183.

8. "A Model for the Temperature Dependence of the Yield Strength of FeAl", E.P. George and I. Baker, Philosophical Magazine, **77** (1998) 737-750.

9. "Thermal vacancies and the Yield Anomaly of FeAl", E.P. George and I. Baker, Intermetallics, **6**

(1998) 759-763.

10. "On the Mechanism of the Paramagnetic to Ferromagnetic Transition in FeAl", Y. Yang, I. Baker, and P. Martin, Philosophical Magazine B, 79 (1999) 449-461.

11. "The Yield Stress Anomaly in Stoichiometric FeAl at High Strain Rate" Y.Yang, I. Baker, R.T. Gray, III and C. Cady, Scripta Materialia, 40 (1999) 403-408.

12. "Effect of Vacancies on the Tensile Properties of Fe-40Al Single Crystals in Air and Vacuum", Y. Yang, I. Baker and E.P. George, Materials Characterization, 42 (1999)161-168.

13. "Mechanical Properties of Strongly-Ordered B2 Compounds", I. Baker, Transactions of Nonferrous Metals Society of China, 9 (1999) 146-156. *(Invited)*

14. "Improving the Ductility of Intermetallic Compounds by Particle-Induced Slip Homogenization", I. Baker, Scripta Materialia, 41 (1999) 409-414.

15. "Recovery, Recrystallization and Grain Growth in Ordered Alloys", I. Baker, Intermetallics, in press. *(Invited)*

16. "The Effect of Environment and Strain Rate on the Room Temperature Tensile Properties of FeAl Single Crystals", D. Wu and I. Baker, Intermetallics, in press.

17. "The Role of Edge and Screw Dislocations on Hydrogen Embrittlement of Fe-40Al". M. Wittmann, D. Wu, I. Baker, E.P. George, and L. Heatherly, Materials Science and Engineering, in press.

18. "Strain-Induced Ferromagnetism in FeAl Single Crystals", D. Wu and I. Baker, submitted to Materials Science and Engineering.

19. "The Effects of Environment on the Room-Temperature Mechanical Behavior of Single-Slip Oriented FeAl Single Crystals", I. Baker, D. Wu, S. O. Kruijver, and E.P. George, submitted to Materials Science and Engineering.

20. "Mechanical Properties of Soft Magnetic FeCo Alloys", E. P. George, A. N. Gubbi, and I. Baker, Materials Science and Engineering, in preparation.

BOOK CHAPTERS

1. "Intermetallics: iron aluminides", E. P. George and I. Baker, in The Encyclopedia of Materials, in press.

2. "Intermetallics: nickel aluminides", I. Baker and E. P. George, The Encyclopedia of Materials, in press.

CONFERENCE PAPERS

1. "An Overview of Environmental Effects in Iron Aluminides", X. Pierron and I. Baker, Design Fundamentals of High Temperature Composites, Intermetallics and metal-Ceramic Systems", Editors - R.Y. Lin, Y.A. Chang, R.G. Reddy and C.T. Liu, The Metallurgical Society, Warrendale, Pa., 1996, 271-286. *(Invited)*

2. "Deformation and Fracture of B2 Compounds", I. Baker, in Deformation and Fracture of Ordered

Intermetallic Materials, Editors - W.O. Soboyejo, H.L. Fraser and T.S. Srivatsan, The Metallurgical Society, Warrendale, Pa., (1996) 27-44. *(Invited)*

3. "Aluminides: Processing Properties and the Mechanical Properties of FeAl", I. Baker and E.P. George, In Proceedings of the International Symposium on Nickel and Iron Aluminides: Processing, Properties and Applications, Editors - S. Deevi and V. Sikka, The Metallurgical Society, Warrendale, Pa., (1997) 145. *(Invited)*

4. "Annealing of Cold-Rolled Fe-40Al single Crystals", Y. Yang and I. Baker, Proceedings of the Material Research Society, 460 (1997) p367.

5. "A Model for the Yield Strength Anomaly in FeAl", I. Baker and E. P. George, Proceedings of the Material Research Society, 460 (1997) p373.

6. "Effect of Iron Content and Heat-Treatment on The Precipitation of a New tetragonal boride in FeAl + B", X. Pierron and I. Baker, Proceedings of the Material Research Society, 460 (1997) p331.

7. "Brittle Fracture in B2 Compounds", P.R. Munroe and I. Baker, George R. Irwin Symposium on Cleavage Fracture, The Metallurgical Society, Warrendale, Pa., (1997) 329-346. *(Invited)*

8. "Boron Strengthening in FeAl" I. Baker, X. Li, H. Xiao, O. Klein, C. Nelson, R. L. Carleton and E. P. George, The Metallurgical Society, Warrendale, Pa., (1998), 23-38.

9. "The Mechanical Behavior of FeAl", I. Baker and E.P. George, "High Temperature Ordered Intermetallic Alloys VIII", Proceedings of the Material Research Society, (1999) KK4.1.1. *(Invited)*

10. "Annealing of Quenched FeAl alloys with and without B and Ti, Y. Yang and I. Baker, "High Temperature Ordered Intermetallic Alloys VIII", Proceedings of the Material Research Society, (1999) KK8.22.1.

INVITED PRESENTATIONS IN UNIVERSITIES AND INDUSTRY

"The Mechanical Properties of FeAl", DOE Workshop on Intermetallics, Atlanta (1996)

"The Mechanical Properties of FeAl", University of Tennessee (1997)

"The Mechanical Properties of FeAl", General Electric Corporate R&D Center (1997)

"The Mechanical Properties of FeAl", Brown University (1998)

UNPUBLISHED CONFERENCE PRESENTATIONS

1. "An Overview of the Mechanical Properties of FeAl", I. Baker and E.P. George, presented at the Fall TMS meeting, Cincinnati, OH, Oct., 1996. *(Invited)*

2. "Grain Boundary Structure and Chemistry in Ordered Alloys", E.P. George and I. Baker, Symposium in honor of the Late David A. Smith, presented at the Fall TMS meeting, Indianapolis, IN, Sept. 15-17th, 1997.

3. "The Mechanical Behavior of FeAl", I. Baker, Annual TMS Meeting, San Antonio, TX, February, 1998. *(Invited)*

4. "Effects of Thermal Vacancies and Deviations from Stoichiometry on the Yield strength Anomaly of FeAl" E.P. George and I. Baker, presented at the meeting on Nonstoichiometric Ceramics and Intermetallics, Kona, Hawaii, April 26-30, 1998.

5. "Thermal vacancies and the Yield Anomaly of FeAl", E.P. George and I. Baker, Kyoto Workshop on High-Temperature Intermetallics, Kyoto, Japan, May 11-13, 1998.

6. "On the Mechanism of Deformation-Induced Magnetic Transition in FeAl", Y. Yang, I. Baker and P. Martin, Annual TMS meeting, San Diego, CA, March 1-5, 1999.

7. "The Yield Strength Anomaly in FeAl", E.P. George and I. Baker, International Symposium on "Intermetallics for the Third Millenium", Fall ASM meeting, Cincinnati, OH, Nov. 1-4, 1999. *(Invited)*

8. "Advanced Intermetallic Alloys", I. Baker, Materials Technology 99, University of Waterloo, Waterloo, Ontario, November 10th, 1999. *(Invited)*

9. "On the Yield Strength Anomaly in CoTi and CoHf", I. Baker, M. Wittmann and P. Bove, Annual TMS meeting, Nashville, TN, 12-16 March, 2000.

10. "The Strain-Induced Paramagnetic to Ferromagnetic Transition in FeAl", I. Baker, Y. Yang, D. Wu and P. Martin, Annual TMS meeting, Nashville, TN, 12-16 March, 2000.