

DOE/ER/45354--5

DE93 007971

## CORROSION FATIGUE OF IRON-CHROMIUM-NICKEL ALLOYS: FRACTURE MECHANICS, MICROSTRUCTURE AND CHEMISTRY

### Progress Report

for Period January 1, 1992 - December 31, 1992

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January 25, 1993

Prepared for

THE U. S. DEPARTMENT OF ENERGY  
AGREEMENT NO. DE-FG02-88ER45354

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15 January 1993

TO: Division of Materials Sciences  
Office of Basic Energy Sciences  
Department of Energy  
Washington, DC 20545

FROM: Dr. Robert P. Wei  
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REFERENCE: Grant No. DE-FG02-88ER45354, "Corrosion Fatigue of Iron-Chromium-Nickel Alloys: Fracture Mechanics and Chemistry"

SUBJECT: Progress Report for Period 1 January to 31 December 1992

This progress report briefly summarizes the research performed under the referenced grant for the period from 1 January to 31 December 1992, and contains a cumulative listing of technical presentations and publications dating back to 1 June 1988.

Technical Report of Progress

Under this grant, a multi-disciplinary research program is undertaken to address certain fundamental issues relating to corrosion fatigue crack growth in structurally important alloys in aqueous environments. The principal goal of the research is to develop and expand the scientific understanding of the processes that control corrosion fatigue crack growth, particularly for ferrous alloys in terms of the controlling mechanical and chemical/electrochemical processes and their interactions with the microstructure.

Focus is placed upon the austenitic iron-chromium-nickel (FeCrNi) alloys because of the need to resolve certain mechanistic issues and because of extensive utilization of these alloys in the power generation and chemical industries. Emphasis is given to the growth of short (small) cracks at low growth rates because crack growth in this regime is expected to be more sensitive to changes in external chemical/electrochemical variables. The slower growth rates would also facilitate critical testing of mechanistically based models in terms of their ability to predict long-term response from short-time data. The ongoing research utilizes a combined mechanics, materials science and chemistry/electrochemistry approach.

The program was initiated on 1 June 1988, and was extended for an additional 3 years, effective 1 June 1991. Principal efforts during the past 12 months are as follows: (1) studies of hydride decomposition and cracking in an electrolytically charged Fe18Cr12Ni alloy during aging at room temperature; (2) quantitative assessment and modeling of the role of microstructure on corrosion fatigue crack growth; and (3) assessment of a mechanistically based probability approach for service life prediction. Progress in each of the areas is given briefly in the following executive summary. Specific aspects are given in the attached reprints or preprints.

Executive Summary

Principal findings from research conducted during this period are as follows:

1. To assist in the understanding of hydrogen embrittlement of austenitic stainless steels, phase transformation and cracking during room temperature aging of electrolytically charged, high-purity Fe18Cr12Ni alloy and commercial-grade 304 (Fe18Cr10Ni) stainless steel were examined. The results showed that  $\epsilon^*$  (hcp) hydride was formed in the Fe18Cr12Ni alloy upon charging. The newly formed hydride was unstable and decomposed rapidly to form  $\epsilon$  martensite, which further transformed to  $\alpha'$  martensite during aging. The kinetics of hydride decomposition ( $\epsilon^* - \epsilon$ ) were more rapid than the associated  $\epsilon - \alpha'$  phase transformation. Two types of cracking were observed; namely, austenite grain and twin boundary cracking and transgranular cracking through the austenite grains. Boundary cracks formed mainly during hydrogen charging, whereas transgranular cracking evolved with aging time. The evolution of transgranular cracks correlated well with the kinetics of  $\epsilon - \alpha'$  transformation during aging. These results are published in *Scripta Metallurgica et Materialia* (see Attachment 1).

Similar results were obtained on the 304 stainless steel. In this material, in addition to the  $\epsilon^*$  (hcp) hydride, a  $\gamma^*$  (fcc) hydride was observed upon charging. These hydrides, however, were more stable and decomposed at a slower rate than that in the Fe18Cr12Ni alloy. Similar patterns of cracking was also observed, and the evolution of transgranular cracking again correlated well with the kinetics of  $\epsilon - \alpha'$  transformation during aging. The pattern of cracking in both materials correlated well with that produced by corrosion fatigue, and suggested that transgranular fatigue crack growth might be associated with cracking through a hydride phase at the crack tip. Transmission electron microscopy studies are in progress to quantify the microstructure. Measurements of hydrogen concentration in the charged material are also in progress.

2. To clarify the mechanisms for corrosion fatigue crack growth, a direct comparison was made of the morphology of fracture surface of the Fe18Cr12Ni alloy that had been produced by corrosion fatigue in 3.5% NaCl solutions (at pH 2 to 12) and in hydrogen at room temperature. The results show that the fracture surface morphology was identical in these environments. The fracture paths consisted of intergranular cracking along the austenite boundaries, twin boundary separation, and quasi-cleavage of transformed martensite. The austenite and twin boundaries appeared to be preferred paths for cracking. The amount of intergranular (IG) and twin boundary (TB) separation depended upon solution pH; the amount at pH 12 was essentially equal to that observed in hydrogen at 100 kPa. The areal fractions of IG/TB separation were correlated with the corrosion fatigue crack growth rates, and their dependence on solution pH was interpreted in terms of its influence on hydrogen fugacity. These observations confirmed hydrogen embrittlement (rather than anodic dissolution) as the micromechanism for corrosion fatigue crack growth in the aqueous environments.

A quantitative assessment of the role of microstructure on hydrogen assisted fatigue crack growth in the Fe18Cr12Ni alloy has been made through the use of a superposition model. The hydrogen assisted rate of crack growth

through  $\alpha'$ -martensite was estimated to be only twice as high as that in vacuum ( $6.45 \times 10^{-12}$  versus  $3.25 \times 10^{-12}$  m/cycle), while the rate through the austenite grain and twin boundaries was more than ten-times faster ( $4.84 \times 10^{-12}$  m/cycle). These findings strongly suggest that strain-induced  $\alpha'$ -martensite formation is not a principal contributor to the environmental enhancement of corrosion fatigue crack growth, and is not necessary for hydrogen embrittlement of the metastable austenitic stainless steels. A paper summarizing these results has been accepted for publication in *Scripta Metallurgica et Materialia* (see Attachment 2).

Research under this program has clarified the micromechanism and rate controlling process for corrosion fatigue crack growth in the metastable austenitic stainless steels. The results clearly showed hydrogen embrittlement to be the mechanism for corrosion fatigue crack growth in deaerated 3.5% NaCl solutions. The crack growth response is controlled by the rate of electrochemical reactions with the bared steel surfaces at the crack tip. Austenite and twin boundaries have been shown to be the preferred paths for cracking and principal contributors to the enhancement in growth rates. The amount of intergranular (IG) and twin boundary (TB) separation depended upon solution pH; the amount at pH 12 was essentially equal to that observed in hydrogen at 100 kPa. The areal fractions of IG/TB separation are correlated with the corrosion fatigue crack growth rates, and their dependence on solution pH is interpreted in terms of its influence on hydrogen fugacity. Results from companion hydrogen charging experiments suggested that a hydride phase may be formed at the crack tip. Transgranular crack growth during corrosion fatigue was attributed to cracking through this hydride phase, and the presence of  $\alpha'$ -martensite on the fracture surface reflected post cracking decomposition of this hydride. A brief overall summary was presented at the Second International Conference on Corrosion and Deformation Interactions (CDI2), held in Fontainebleau, France last October, and will appear in the Conference Proceedings (see Attachment 3).

3. As a logical extension of this mechanistic understanding, an effort was made to examine the approaches and methodologies used in service life predictions and reliability analyses. It became clear that the development of mechanistically based probability models is essential for addressing the questions of durability and reliability of systems, such as nuclear reactors and those for high-level radio-active waste containment. The models must quantify the functional dependence of the material degradation processes on external variables (typically those associated with service conditions) and internal variables (those associated with local environment and microstructure). Model development requires the full integration of mechanistic understanding and probability analysis, and probability considerations cannot be an ex post factor addition. A paper (see Attachment 4), which describes and illustrates this approach, will appear in *Engineering Fracture Mechanics* early in 1993. This approach has been incorporated into a NSF/MRG program on environmental and stochastic aspects of crack growth in nickel-base alloys at high temperatures, and a FAA sponsored program on corrosion and corrosion fatigue of aluminum alloys as a part of its Aging Airplanes Program.

To assist one of the DoE Offices in the planning of research needed to address problems in "Aging Energy Production and Distribution Systems", R. P. Wei participated in a DoE workshop on this subject, held at Rice University in Houston, Texas in October 1992. A position paper was prepared and presented.

This paper defined the needs and proposed an approach for integrating mechanistic understanding and probability analysis in addressing the problems of durability and reliability assessments. The approach is to be applied to appropriate failure processes, and is designed to address the contributions of environmental and materials variables quantitatively. This paper (see Attachment 5) will appear in the Proceedings of the Workshop, which is to be published as a single issue of the Applied Mechanics Review.

Because of budgetary constraints and of the desire of DoE/BES to bring this program to a close at the end of the current grant period in 1993, the planned effort on bicrystals of Fe18Cr12Ni alloy was discontinued.

Presentations and Publications (cumulative; DoE/BES related items are denoted by an asterisk)

"The Kinetics Aspects of Hydrogen Embrittlement", R. P. Wei, 2nd National Aero-space Plane (NASP) Workshop: Hydrogen-Materials Interactions, Scottsdale, AZ, June 2, 1988.

\*"Bare Surface Reactions and Crack Growth in Steels: A New Perspective", R. P. Wei, Gordon Research Conference on Corrosion, New London, NH, July 21, 1988.

"Deformation and Subcritical Crack Growth Under Static Loading", Hui Yin, Ming Gao and R. P. Wei, 1988 TMS Fall Meeting, Chicago, IL, September 27, 1988.

"Stress Corrosion Cracking of High-Strength Steels in Aqueous Environments", H. C. Chu, Ming Gao and R. P. Wei, 1988 TMS Fall Meeting, Chicago, IL, September 28, 1988.

\*"Chemically-Induced Crack Length Effects in Corrosion Fatigue of AISI 304 Stainless Steel", D. E. Allison, A. Alavi, M. Gao and R. P. Wei, 1988 TMS Fall Meeting, Chicago, IL, September 28, 1988.

\*"In Situ Fracture Techniques for Studying Transient Reactions With Bare Steel Surfaces", R. P. Wei and A. Alavi, 174th Fall Meeting of Electrochemical Society, Chicago, IL, October 10, 1988.

"Electrochemical Considerations of Crack Growth in Ferrous Alloys, R. P. Wei, Seventh International Conference on Fracture (ICF7), Houston, TX, March 20-24, 1989.

"Corrosion Fatigue Crack Growth and Reactions With Bare Steel Surfaces", R. P. Wei, CORROSION/89, New Orleans, LA, April 17-21, 1989.

"Identification of Hydride Phases in Thermally Charged Titanium Aluminides", Ming Gao, Bart Boodey and Robert P. Wei, 3rd NASP Workshop: Hydrogen-Materials Interactions, Scottsdale, AZ, May 31-June 2, 1989.

"Phase Transformation and Moisture Induced Crack Growth in Yttria Stabilized Zirconia", R. P. Wei, International Conference on Mechanistic Aspects of Fracture, Irsee, W. Germany, June 19-23, 1989.

- \*"Environmental and Microstructural Considerations of Corrosion Fatigue Crack Growth", R. P. Wei, FRACTURE '89, Johannesburg, South Africa, June 29, 1989.
- "Corrosion Fatigue Crack Growth and Electrochemical Reactions for A X-70 Linepipe Steel in Carbonate-Bicarbonate Solution, R. P. Wei, FRACTURE '89, Johannesburg, South Africa, June 29, 1989.
- \*"Hydrogen Embrittlement and Environmentally Assisted Crack Growth", R. P. Wei, Fourth International Conference on The Effects of Hydrogen on Material Behavior, Jackson Lake Lodge, WY, September 14, 1989.
- \*"Bare Surface Reactions and Mechanisms for Corrosion Fatigue Crack Growth", R. P. Wei, DOE/BES/DMS Contractors' Meeting, Oakland, CA, September 21, 1989.
- "Environmentally Assisted Fatigue Crack Growth in an Alumina Reinforced Aluminum Alloy", R. P. Wei, ALCAN Meeting, Chicago, IL, September 28, 1989.
- "Identification of New Precipitates in Peak Aged Al-Mg-Si (6061-T6) Aluminum Alloy", M. Gao and R. P. Wei, 1989 TMS Fall Meeting, Indianapolis, IN, October 2, 1989.
- \*"Crack Paths and Corrosion Fatigue Crack Growth in 304 Stainless Steels", Ming Gao, Shuchun Chen and R. P. Wei, 1989 TMS Fall Meeting, Indianapolis, IN, October 2, 1989.
- "Environmentally Assisted Crack Growth and Predictions of Service Life", R. P. Wei, ONR Workshop on Life Predictions, National Institute of Standards and Technology, Gaithersburg, MD, October 27, 1989.
- "Mechanistic Considerations of Corrosion Fatigue of Steels", R. P. Wei, International Conference on Evaluation of Materials Performance in Severe Environments (EVALMAT 89), November 20, 1989.
- \*"Bare Surface Reactions and Mechanicsms for Corrosion Fatigue Crack Growth in Steels", R. P. Wei, Nippon Steel Corporation, R & D Laboratories II, Sagamihara, Japan, November 24, 1989.
- \*"Rate Controlling Processes and Corrosion Fatigue Crack Growth", Robert P. Wei, Materials Science Colloquia, University of Virginia, Charlottesville, VA, April 2, 1990.
- "Phase Transformation and Sustained-load Crack Growth in Yttria Stabilized Zirconia", Hui Yin and R. P. Wei, American Ceramic Society, Dallas, TX, April 24, 1990.
- \*"Bare Surface Reactions and SCC and CF Crack Growth in Ferrous Alloys: A New Approach to Understanding", R. P. Wei, Seminar at the University of Erlangen, Erlangen, Germany, July 27, 1990.
- \*"Scratching Electrode Technique -- What is Being Measured", Ming Gao, DOE/BES Contractors' Meeting, Minneapolis, MN, September 13, 1990.
- \*"Bare Surface Reactions and SCC and CF Crack Growth in Ferrous Alloys: A New Approach to Understanding", R. P. Wei, Keynote lecture for the 1990 Annual

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"Phase Transformation and Sustained-load Crack Growth in Yttria Stabilized Zirconia", R. P. Wei, Industrial Technology Research Institute, Materials Research Laboratories, Taiwan, ROC, September 14, 1990.

"Environmentally Assisted Crack Growth in Ceramic Particle Reinforced Metal Matrix Composites", R. P. Wei, Alcan Meeting, Kingston, Ontario, Canada, September 26, 1990.

"Misfit Strains and Mechanism for the Precipitation of Hydrides in Thermally Charged Alpha-2 Titanium Aluminides", Ming Gao, J. Bart Boodey and Robert P. Wei, TMS/ASM Fall Meeting, Detroit, MI, October 8, 1990.

"Hydrogen Solubility and Hydride Formation in a Thermally Charged Gamma Based Titanium Aluminide", J. Barton Boodey, Ming Gao and Robert P. Wei, TMS/ASM Fall Meeting, Detroit, MI, October 8, 1990.

"Environmentally Assisted Fatigue Crack Growth in Aluminum and Titanium Alloys", Robert P. Wei, Institut fur Werkstoffwissenschaften, Erlangen, Germany, February 5, 1991.

\*"Hydrogen Embrittlement and Environmentally Assisted Crack Growth in High Strength Steels", Robert P. Wei, GKSS-Forschungszentrum Geesthacht GmbH, Geesthacht, Germany, February 22, 1991.

\*"Electrochemical Reactions With Bare Surfaces and Corrosion Fatigue Crack Growth in Steels", Robert P. Wei, MPI-Stuttgart, Germany, February 25, 1991.

\*"Electrochemical Reactions With Bare Surfaces and SCC and CF Crack Growth in Ferrous Alloys", Robert P. Wei, GKSS- Forschungszentrum Geesthacht GmbH, Geesthacht, Germany, March 14, 1991.

"Environmentally Assisted Fatigue Crack Growth in Aluminum and Titanium Alloys", Robert P. Wei, GKSS-Forschungszentrum Geesthacht GmbH, Geesthacht, Germany, April 17, 1991.

"Environmentally Assisted Cracking of Ceramics and Intermetallics", Robert P. Wei, GKSS-Forschungszentrum Geesthacht GmbH, Geesthacht, Germany, April 24, 1991.

"Phase Transformation and Moisture Assisted Crack Growth in Yttria Stabilized Zirconia", Robert P. Wei, Institut fur Werkstoffmechanik, Fraunhofer, Germany, May 3, 1991.

\*"Environmentally Assisted Crack Growth in Metals and Ceramics", Robert P. Wei, Chinese American Academic and Professional Society, New York City, May 26, 1991.

\*"Hydrogen Embrittlement and Environmentally Assisted Crack Growth in High Strength Steels", Robert P. Wei, Zentralinstitut fur Festkorperphysik and Werkstoffforschung, Dresden, Germany, June 27, 1991.

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- \*"Corrosion Fatigue Crack Growth in a High Purity Fe18Cr12Ni Austenitic Alloy", M. Gao, S. F. Chen and R. P. Wei, DOE Contractors Meeting, Brookhaven National Laboratory, Upton, Long Island, NY, September 20, 1991.
- "A Probabilistic Approach to Life Prediction for Corrosion Fatigue Crack Growth", Robert P. Wei, Boeing Commercial Airplane Group Seminar, Seattle, WA, October 17, 1991.
- \*"Corrosion Fatigue and Electrochemical Reactions in a High Purity Fe18Cr12Ni Austenitic Steel", M. Gao, Shyuan-Fang Chen and Robert P. Wei, TMS Fall Meeting, Cincinnati, OH, October 21, 1991.
- \*"Crack Paths, Alpha prime-Martensitic Transformation and pH Effect in a High-Purity Fe18Cr12Ni Austenitic Alloy During Corrosion Fatigue", M. Gao, Shyuan-Fang Chen and Robert P. Wei, TMS Fall Meeting, Cincinnati, OH, October 21, 1991.
- "A Probabilistic Approach to Life Prediction for Corrosion Fatigue Crack Growth", D. G. Harlow and R. P. Wei, 3rd NACE International Region Management Committee Symposium on Life Prediction of Corroding Structures, Kauai, Hawaii, November 8, 1991.
- "A Mechanistically Based Probability Approach to Life Prediction for Corrosion and Corrosion Fatigue of Airframe Materials", R. P. Wei, FAA/NASA Workshop on Corrosion, ALCOA, PA, November 23, 1991.
- "A Mechanistically Based Probability Approach to Life Prediction for Corrosion and Corrosion Fatigue of Airframe Materials", Robert P. Wei and D. Gary Harlow, International Workshop on Structural Integrity of Aging Airplanes, Atlanta, GA, April 1, 1992.
- \*"Statistical versus Mechanistic Approaches to Life Prediction", R. Wei, Gordon Research Conference on Corrosion, New London, NH, July 24, 1992.
- "A Mechanistically Based Probability Approach to Life Prediction for Corrosion and Corrosion Fatigue of Airframe Materials", Robert P. Wei and D. Gary Harlow, Seminar at Exxon, NJ, August 4, 1992.
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- \*"Micromechanism for Corrosion Fatigue Crack Growth in Metastable Austenitic Stainless Steels", Robert P. Wei and Ming Gao, International Conference on Corrosion-Deformation Interactions, Fontainebleau, France, October 6, 1992.
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D. Gary Harlow and Robert P. Wei, "A Dominant Flaw Probability Model for Corrosion and Corrosion Fatigue", Proceedings of the 12th International Corrosion Congress, 19-24 September 1993, Houston, TX.

#### Personnel

During the past 12 months, the following personnel were associated with this grant in addition to Dr. Robert P. Wei, Principal Investigator: Dr. Ming Gao and Messrs. Shuchun Chen and Shyuan-Fang Chen. Dr. Gao is a Senior Research Scientist from the People's Republic of China and is participating in the program on a part-time basis. He is responsible for the mechanical and microstructural aspects of the program. He assists Dr. Wei in addressing the problems of corrosion fatigue mechanisms and modeling of corrosion fatigue crack growth. Mr. S. C. Chen is a Ph.D. candidate in Materials Science and Engineering, and is studying the microstructural aspects of corrosion fatigue crack growth, including hydrogen induced phase transformation and subsequent decomposition during aging at room temperature. He is planning to complete his Ph.D. program by August 31, 1993.

Dr. Wei continued to serve as Chairman of the Department of Mechanical Engineering and Mechanics during 1992. In this capacity, he had administrative responsibility for a department with 34 full-time faculty, and about 370 undergraduate and 100 graduate students.

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