Progress Report
January 1, 1995 to June 30, 1995

NASA-UVA LIGHT AEROSPACE ALLOY AND STRUCTURES TECHNOLOGY PROGRAM (LA²ST)

NASA-LaRC Grant NAG-1-745

Submitted to:
National Aeronautics and Space Administration
Langley Research Center
Hampton, Virginia 23681-0001

Attention:
Mr. Neil Price
Grants Officer
MS 126

For Review by:
Mr. Dennis L. Dicus
Grant Monitor
Metallic Materials Branch, MS 188A

Submitted by:
Richard P. Gangloff
Professor
and
Edgar A. Starke, Jr.
University Professor

Department of Materials Science and Engineering
School of Engineering and Applied Science
University of Virginia

Report No. UVA/528266/MS95/118
July 1995

SCHOOL OF
ENGINEERING & APPLIED SCIENCE
University of Virginia
Thornton Hall
Charlottesville, VA 22903
UNIVERSITY OF VIRGINIA
School of Engineering and Applied Science

The University of Virginia's School of Engineering and Applied Science has an undergraduate enrollment of approximately 1,500 students with a graduate enrollment of approximately 600. There are 160 faculty members, a majority of whom conduct research in addition to teaching.

Research is a vital part of the educational program and interests parallel academic specialties. These range from the classical engineering disciplines of Chemical, Civil, Electrical, and Mechanical and Aerospace to newer, more specialized fields of Applied Mechanics, Biomedical Engineering, Systems Engineering, Materials Science, Nuclear Engineering and Engineering Physics, Applied Mathematics and Computer Science. Within these disciplines there are well equipped laboratories for conducting highly specialized research. All departments offer the doctorate; Biomedical and Materials Science grant only graduate degrees. In addition, courses in the humanities are offered within the School.

The University of Virginia (which includes approximately 2,000 faculty and a total of full-time student enrollment of about 17,000), also offers professional degrees under the schools of Architecture, Law, Medicine, Nursing, Commerce, Business Administration, and Education. In addition, the College of Arts and Sciences houses departments of Mathematics, Physics, Chemistry and others relevant to the engineering research program. The School of Engineering and Applied Science is an integral part of this University community which provides opportunities for interdisciplinary work in pursuit of the basic goals of education, research, and public service.
NASA-UVA LIGHT AEROSPACE ALLOY
AND STRUCTURES TECHNOLOGY PROGRAM

LA³ST

Program Directors:

Edgar A. Starke, Jr.
Richard P. Gangloff

Co-principal Investigators:

Carl T. Herakovich
John R. Scully
Gary J. Shiflet
Glenn E. Stoner
John A. Wert

NASA-LaRC Grant Monitor:

Dennis L. Dicus

Report No. UVA/528266/MS95/118

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NASA-UVa LIGHT AEROSPACE ALLOY AND STRUCTURES TECHNOLOGY PROGRAM

(LA²ST)

EXECUTIVE SUMMARY

The NASA-UVa Light Aerospace Alloy and Structures Technology (LA²ST) Program was initiated in 1986 and continues with a high level of activity. Projects are being conducted by graduate students and faculty advisors in the Department of Materials Science and Engineering, as well as in the Department of Civil Engineering and Applied Mechanics, at the University of Virginia. This work is funded by the NASA-Langley Research Center under Grant NAG-1-745. Here, we report on progress achieved between January 1 and June 30, 1995.

The objective of the LA²ST Program is to conduct interdisciplinary graduate student research on the performance of next generation, light-weight aerospace alloys, composites and thermal gradient structures in collaboration with NASA-Langley researchers. Specific technical objectives are presented for each research project. We generally aim to produce relevant data and basic understanding of material mechanical response, environmental/corrosion behavior, and microstructure; new monolithic and composite alloys; advanced processing methods; new solid and fluid mechanics analyses; measurement and modeling advances; and a pool of educated graduate students for aerospace technologies.

The accomplishments presented in this report are summarized as follows.

Three research areas are being actively investigated, including: (1) Mechanical and Environmental Degradation Mechanisms in Advanced Light Metals and Composites, (2) Aerospace Materials Science, and (3) Mechanics of Materials and Composites for Light Aerospace Structures.

Eleven research projects are being conducted by 9 PhD and 3 MS level graduate students, with 7 faculty members from 2 departments in the School of Engineering and Applied Science at UVa. Each project is planned and executed in conjunction with a specific branch and technical monitor at NASA-LaRC.

Two undergraduates are conducting research in the Metallic Materials Branch at NASA-LaRC during the Summer of 1995. No undergraduates are currently participating in LA²ST research at UVa.
Collective accomplishments between January and June of 1995 include: 6 journal or proceedings publications, 1 NASA progress report, 9 presentations at national technical meetings, 1 MS thesis, and 1 PhD dissertation published. Two students graduated during this reporting period; 1 with the Masters of Science Degree in Materials Science and Engineering, and 1 with the Doctor of Philosophy Degree in Materials Science and Engineering at UVa. The LA\textsuperscript{2}ST totals since 1986 are 102 publications (60 archival journal or book publications), 20 PhD dissertations or MS theses, 109 external technical presentations, 17 NASA progress reports, and 4 NASA Contractor Reports. Since 1986, 33 graduate students, including 31 citizens of the United States, have been involved with LA\textsuperscript{2}ST research; 20 have received the MS or PhD degree. Five post-doctoral research associates have participated in LA\textsuperscript{2}ST research. A total of 13 different faculty have worked on the LA\textsuperscript{2}ST program.

Research on mechanisms of localized corrosion and environmental fracture in Al-Cu-Li-Mg-Ag alloy X2095 and compositional variations provides measurements of electrochemistry, occluded chemistry, fractography and environmental crack growth rates of UA and PA AA2095. The UA temper yields much higher crack growth rates, as well as film free and somewhat featureless fractography, consistent with an embrittlement mechanism. The PA temper yields high angle intergranular EAC type fractography with considerable corrosion product on the fracture surfaces, much slower crack growth rates, consistent with an EAC mechanism based on the anodic dissolution of T\textsubscript{1} and copper depleted zones. Cracking was observed in chromate inhibited environments, with samples polarized anodic to a critical electrochemical potential. (Project 4)

Research on hydrogen interactions with Al-Li alloys and hydrogen embrittlement of AA2090 seeks to develop a fundamental understanding of the effects of internal lattice and trapped hydrogen on the mechanical properties of selected Al-Li-Cu-X alloys. Studies were performed on AA2090, an Al-Li-Cu-Zr alloy within the specified compositional range of AA2090, and model binary alloys. Mechanical properties were evaluated by constant extension rate testing of smooth tensile specimen and J-Integral testing of compact tension samples, both precharged with hydrogen. Degradation of mechanical properties with hydrogen was correlated with thermal desorption analysis of
hydrogen trapping and fractography. Results suggest that hydrogen embrittlement is a strong contributor to stress corrosion cracking of this class of alloys. (Project 5)

Research on the deformation and fracture of high strength titanium alloys demonstrates that predissolved hydrogen (500 to 700 wppm) causes stable crack growth at 75°C. This brittle cracking initiates at a stress intensity level that is about one-third of the plane strain fracture toughness for aged Beta-21S, and progresses by microscopic processes that are different compared to ductile fracture of as-received alloy without hydrogen. Work is progressing to develop procedures for reproducible electrochemical introduction of hydrogen, and slow-rate elevated temperature fracture resistance characterization; problems are associated with each procedure. Timet should deliver cold rolled sheet and hot rolled plate of Low Cost Beta. A single microstructure of LCB sheet, relevant to High Speed Civil Transport alloy development, will be examined to characterize and understand temperature-dependent internal hydrogen embrittlement of high strength β-titanium alloys. (Project 6a)

Research on the cryogenic fracture of Al-Cu-Li-In alloys demonstrates that the fracture toughness R-curve behavior of 2090-T81 plate is essentially constant in the temperature range from 25°C to about -150°C. Initiation and growth toughnesses increase substantially at -185°C, associated with numerous large delaminations. Delaminations occurred between grains, one of which was of the brass texture and in many cases the other was of the S texture. In contrast to the temperature dependence of fracture toughness; alloy yield strength, elastic modulus, work hardening exponent, and intrinsic fracture strain each increase monotonically with decreasing temperature from 25°C to -185°C. (Project 2)

Research on the fracture toughness of several Weldalite™ alloys demonstrates that the temperature dependence of initiation fracture toughness (K_{IC}) is reasonably predicted by the critical strain controlled model of crack tip ductile fracture. For both AA2095 and AA2195, K_{IC} is predicted to decline monotonically with decreasing temperature from 135°C to -185°C. This decline is controlled by the fact that; while alloy yield strength, work hardening, and elastic modulus increase with decreasing temperature; reduction in
area to fracture (a measure of intrinsic fracture resistance) decreases with decreasing temperature and dominates the fracture toughness temperature dependence. This prediction is consistent with measured fracture toughnesses for AA2095, however, results for AA2195 exhibit scatter that clouds the comparison between the model and experiment.

**Research on the elevated temperature fracture toughness of advanced Al-Cu-Mg-Ag alloys** establishes the importance of high resolution detection of fracture initiation in thin sheet precracked specimens. This method has provided extensive temperature-dependent fracture toughness data for a range of advanced aluminum alloys. A critical plastic strain controlled model successfully predicts these temperature dependencies of initiation toughness for ingot metallurgy and spray formed precipitation hardened alloys, as well as for submicron grain-size powder metallurgy alloys. It is hypothesized that intravoid strain localization governs the intrinsic fracture resistance ($\varepsilon_i^*$) in 2000-series alloys. As strain and strain rate localize and increase between growing microvoids, the material is strain and strain rate hardened, and dislocations promote void nucleation at dispersoids by accumulating at the particle/matrix interface. As temperature increases, the strain hardening rate decreases, the strain rate sensitivity increases, and the tendency for void nucleation at dispersoids decreases. The latter two are responsible for rising $\varepsilon_i^*$ with increasing temperature in AA2519.

**Research on the precipitation hardening and microstructural stability of Al-Si-Ge-Cu alloys** investigated the strengthening mechanisms in a recently developed Al-0.55Si-2.02Ge (wt.%) alloy and significantly improved the theoretical yield strength prediction for this ternary alloy as a function of aging time. Cavity nucleation in this ternary alloy was analyzed using finite element calculations; the results of these calculations are consistent with experimental measurements of the volume fraction of voids as a function of strain and precipitate diameter.

**Research on the effect of tensile stress on nucleation and growth of precipitates in Al-Cu-Mg-Ag alloys** shows that, in solution heat treated samples, $\Omega$ and $\theta'$ precipitates are preferentially oriented parallel to the applied stress axis. This was not observed for
T6 samples which were further aged under stress. Therefore, it was concluded that nucleation of precipitates is strongly affected by stress. It was found that a threshold stress must be exceeded before this effect can be observed. (Project 9)

Research to evaluate wide-panel aluminum alloy extrusions produced accurate predictions of yield strength anisotropy in 2090 near net shape extrusions. These predictions were made using models constructed to predict strength variations due to texture, grain morphology, and precipitate orientation. (Project 7)

Research on damage evolution in polymeric composites will be reported apart from this grant review meeting and progress report. (Project 10)

Research to incorporate environmental effects into fracture mechanics fatigue life prediction codes such as NASA FLAGRO is nearing an initial stage of completion, with two MS thesis published on modeling environmental fatigue crack propagation in 7075-T6 (S-L) and Ti-6Al-4V (ELI) in aqueous NaCl solution. A Fortran 77 computer program, UVAFAS, has been written to incorporate linear superposition, interpolative, and multiple-power-law models of environmental fatigue crack growth rates. Simple linear superposition is of limited value due to the stress corrosion cracking resistance of these alloys in NaCl. Interpolative modeling provides reasonable descriptions of stress intensity, stress ratio and loading frequency effects on crack growth rates, however, the complexity of environmental fatigue and the lack of mechanistic models hinder the usefulness of this empirical approach. Corrosion fatigue crack growth rate data have been generated for 7075 and Ti-6-4 in aqueous to support these conclusions and to provide input for mechanistic modeling. (Project 11)
INTRODUCTION

Background

In 1986 the Metallic Materials Branch in the Materials Division of the NASA-Langley Research Center initiated sponsorship of graduate student engineering and scientific research in the Department of Materials Science and Engineering at the University of Virginia\(^1\). This work emphasized the mechanical and corrosion behavior of light aerospace alloys, particularly Al-Li-Cu based compositions, in aggressive aerospace environments\(^2-4\).

In the Fall of 1988, the scope of this program increased to incorporate research on the development and processing of advanced aerospace materials\(^5\). Additional funding was provided by the Metallic Materials and Mechanics of Materials Branches at NASA-LaRC. In early 1989 the program was further enhanced to include interdisciplinary work on solid mechanics and thermal structures, with funding from several Divisions within the Structures Directorate at NASA-LaRC\(^6\). The Departments of Civil Engineering (Applied Mechanics Program) and Mechanical and Aerospace Engineering participated in this expanded program. With this growth, the NASA-UVa Light Aerospace Alloy and Structures Technology Program (or LA\(^2\)ST Program) was formed within the School of Engineering and Applied Science at UVa.

Since 1989, the LA\(^2\)ST program has operated with full participation from 6 to 13 faculty and 10 to 15 graduate students, yearly, as outlined in the last ten progress reports\(^7-18\) and five grant renewal proposals\(^19-24\). Five 2-day Grant Review Meetings have been held in July at the Langley Research Center, with over 25 faculty and graduate students from UVa participating at each meeting\(^9,11,13,15,17\). Since 1990, undergraduate engineering students have been involved in research projects at both NASA-LaRC and UVa.

In October of 1991, E.A. Starke proposed a substantial enhancement to the base LA\(^2\)ST Program\(^25,26\). The objective of this supplement was to involve UVa faculty with engineering scientists from aluminum alloy producers and airframe manufacturers in a broad research program to develop aluminum alloys and composites for elevated temperature High Speed Civil Transport applications. This research began in January of 1992 and the results are separately reported. The LA\(^2\)ST and HSCT activities were merged in 1995\(^23\).
Problem and Needs

Future aerospace structures require high performance light alloys and metal matrix composites with associated processing and fabrication techniques; new structural design methods and concepts with experimental evaluations; component reliability/durability/damage tolerance prediction procedures; and a pool of masters and doctoral level engineers and scientists. Work on advanced materials and structures must be interdisciplinary and integrated. The thermal and chemical effects of aerospace environments on light metals and composites are particularly important to material performance. Nationally, academic efforts in these areas are limited. The NASA-UVa LA²ST Program addresses these needs.

LA²ST Program

As detailed in the original proposal[6] and affirmed in the most recent renewal[24], faculty from the Departments of Materials Science and Engineering, Mechanical and Aerospace Engineering, and Civil Engineering and Applied Mechanics at UVa are participating in the LA²ST research and education program focused on high performance, light weight, aerospace alloys and structures. We aim to develop long term and interdisciplinary collaborations between graduate students, UVa faculty, and NASA-Langley researchers.

Our research efforts are producing basic understanding of materials performance, new monolithic and composite alloys, advanced processing methods, solid and fluid mechanics analyses, measurement advances, and new methods for modeling material microstructure and properties. A major product of the LA²ST program is graduate students with interdisciplinary education and research experience in materials science, mechanics and mathematics. These advances should enable various NASA technologies.

The scope of the LA²ST Program is broad. Four research areas are being investigated, including:

- Mechanical and Environmental Degradation Mechanisms in Advanced Light Metals and Composites,
- Aerospace Materials Science,
- Mechanics of Materials and Composites for Light Aerospace Structures,
Twelve research projects are currently ongoing within three of these four areas, and are reported here. These projects involve seven faculty, and twelve graduate students. Seventy-five pct of the graduate students are currently at the doctoral level (9 of 12), all but one are citizens of the United States, two are cosponsored by private industry, and two are conducting all research at the Langley Research Center. In each case the research provides the basis for the thesis or dissertation requirement of graduate studies at the University of Virginia. Each project is developed in conjunction with a specific LaRC researcher. Research is conducted at either UVa or LaRC, and under the guidance of UVa faculty and NASA staff. Participating students and faculty are closely identified with a NASA-LaRC branch.

Organization of Progress Report

This progress report first provides LA²ST Program administrative information including statistics on the productivity of faculty and graduate student participants, a history of current and graduated students, refereed or archival publications, and a list of ongoing projects with NASA and UVa advisors.

Eleven sections summarize the technical accomplishments of each research project, emphasizing the period from January 1 to June 30, 1995. Each section contains a brief narrative of objective, recent progress, conclusions and immediate milestones; coupled with a set of visual aids presented at the Sixth Annual NASA-UVa LA²ST Grant Review Meeting held at NASA-LaRC in July of 1995. The agenda of this meeting is presented in Appendix IV. Appendices I through III document grant-sponsored publications, conference participation and citations of all LA²ST Progress Reports produced since 1986.

References


SUMMARY STATISTICS

Table I documents the numbers of students and faculty who have participated in the LA²ST Program, both during this reporting period and since program inception in 1986. Academic and research accomplishments are indicated by the degrees awarded, publications and presentations. Graduate students and research associates who participated in the LA²ST Program are named in Tables II and III, respectively.

**TABLE I: LA²ST Program Statistics**

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UVA/NSF-LaRC ADVISER
R. P. Gangloff
D. L. Dicou
G. E. Stoner
W. B. Lisagor
G. E. Stoner
D. L. Dicou
F. E. Wawer
W. B. Brewer
R. E. Swanson (VPI)
D. L. Dicou
W. D. Pilkey
J. K. Haviland
D. R. Rumler
M. J. Stuart
R. P. Gangloff
W. B. Lisagor
J. C. Newman
R. P. Gangloff
C. E. Harris
J. A. Wert
D. R. Tenney
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                 | NASA-Langley    |                | W. B. Lisagor         |
| 12 | R. J. Kilmer   | 11/89           | Ph.D. 9/93       | G. E. Stoner
                 | General Motors  |                | W. B. Lisagor         |
| 13 | N. F. Coyle    | 12/89           | Ph.D.            | E. A. Thornton
                 |                 |                | J. H. Starnes, Jr.    |
| 14 | C. J. Lissenden| 9/90            | Ph.D. 6/93       | C. T. Herakovich
                 | University of Kentucky; Engineering Mechanics | | M. J. Pindera |
| 15 | C. Cooper      | 1/91            | Ph.D. 6/93       | W. D. Pilkey
                 | AMP Incorporated|                | J. K. Haviland |
| 16 | Douglas Woll   | 4/91            | Ph.D.            | M. Stuart          |
                 |                 |                | J. Stroud          |
| 17 | S. W. Smith    | 4/91            | Ph.D. 5/95       | G. E. Stoner
                 | NASA-LaRC       |                | D. L. Dicusa       |
| 18 | D. B. Gundel   | 4/91            | Ph.D. 9/94       | J. R. Scully
<pre><code>             | Wright Laboratories |            | W. B. Lisagor     |
             | US Air Force    |                | F. E. Warner      |
</code></pre>
<p>| 19 | K. McCarthy    | 5/91            | M.S. 6/93 (Nonthesis) | W. D. Pilkey |
|                 |                | M. J. Stuart       |
|                 |                | J. Stroud          |</p>
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| 20.   | M. Lyttle        | 12/91           | M.S. 12/93       |                   | Superplasticity in Al-Li-Cu Alloys | J. A. Wert  
|       |                   |                 |                  |                   |                           | T. T. Beles |
|       | NASA-LaRC        |                 |                  |                   |                           | M. J. Shuart  
|       |                   |                 |                  |                   |                           | J. Stroud |
|       | Rohm and Haas    |                 |                  |                   |                           | D. L. Dicus |
| 23.   | M. A. Rowley     | 1/92            | M.S. 12/93       | (UVa AEP Sponsored) | Viscoplasticity of Metals | E. A. Thornton  
|       |                  |                 |                  |                   |                           | J. H. Sternes, Jr. |
|       |                  |                 |                  |                   |                           | TBD |
|       | Allied Signal    |                 |                  |                   |                           | R. S. Piaucik |
|       |                  |                 |                  |                   |                           | W.B. Lisagor |
|       |                  |                 |                  |                   |                           | R.S. Piaucik |
|       |                  |                 |                  |                   |                           | W.B. Lisagor |
| 29.   | Z. Gasem         | 1/94            | Ph.D.            |                   | Time-Dependent Environmental Fatigue in 7000-Series Al Alloys | R.P. Gangloff  
|       |                  |                 |                  |                   |                           | R.S. Piaucik |
|       |                  |                 |                  |                   |                           | D.L. Dicus |
|       |                  |                 |                  |                   |                           | D.L. Dicus |
| 32.   | R. D. Schroeder, III | 6/93       | Ph.D.            |                   | Damage Evolution in Polymeric Composites | C.T. Herakovich  
<p>|       |                  |                 |                  |                   |                           | C.E. Harris |</p>
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<td>2.</td>
<td>Farshad Hizadeh</td>
<td>7/89 to 12/91</td>
<td>Deformation of Metal Matrix Composites</td>
<td>C. T. Herakovich and Marek-Jerzy Pindera</td>
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<td>5.</td>
<td>B. Skrotzki</td>
<td>1/95</td>
<td>Mechanical Property Anisotropy</td>
<td>E. A. Starke, Jr. and G. J. Shiflet</td>
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The following papers are based on research conducted under LA3ST Program support, and are published in the referred or archival literature.


COMPLETED PROJECTS:  (1986 to present reporting period)

1. DAMAGE LOCALIZATION MECHANISMS IN CORROSION FATIGUE OF ALUMINUM-LITHIUM ALLOYS
   Faculty Investigator: R.P. Gangloff
   Graduate Student: Robert S. Piascik
   Degree: PhD
   UVa Department: Materials Science and Engineering (MS&E)
   NASA-LaRC Contact: D. L. Dicus (Metallic Materials)
   Start Date: June, 1986
   Completion Date: November, 1989
   Employment: NASA-Langley Research Center

2. AN INVESTIGATION OF THE LOCALIZED CORROSION AND STRESS CORROSION CRACKING BEHAVIOR OF ALLOY 2090 (Al-Li-Cu)
   Faculty Investigator: Glenn E. Stoner
   Graduate Student: James P. Moran
   Degree: PhD
   UVa Department: MS&E
   NASA-LaRC Contact: W.B. Lisagor (Metallic Materials)
   Start Date: September, 1988
   Completion Date: December, 1989
   Co-Sponsor: ALCOA
   Employment: ALCOA Laboratories

3. MECHANISMS OF LOCALIZED CORROSION IN AL-LI-CU ALLOY 2090
   Faculty Investigator: G.E. Stoner
   Graduate Student: R.G. Buchheit
   Degree: PhD
   UVa Department: MS&E
   NASA-LaRC Contact: D.L. Dicus (Metallic Materials)
   Start Date: June, 1987
   Completion Date: December, 1990
   Co-sponsor: Alcoa
   Employment: Sandia National Laboratories
4. DEFORMATION AND FRACTURE OF ALUMINUM-LITHIUM ALLOYS: THE EFFECT OF DISSOLVED HYDROGEN
   Faculty Investigator: R.E. Swanson (VPI)
   Graduate Student: Frederic C. Rivet
   Degree: MS
   VPI Department: Materials Engineering
   NASA-LaRC Contact: D.L. Dicus (Metallic Materials)
   Start Date: September, 1988
   Completion Date: December, 1990
   Employment: Not determined

5. INVESTIGATION OF THE REACTION KINETICS BETWEEN SiC FIBERS AND SELECTIVELY ALLOYED TITANIUM MATRIX COMPOSITES AND DETERMINATION OF THEIR MECHANICAL PROPERTIES
   Faculty Investigator: F.E. Wawner
   Graduate Student: Douglas B. Gundel
   Degree: MS
   UVa Department: MS&E
   NASA-LaRC Contact: D.L. Dicus and W.B. Brewer (Metallic Materials)
   Start Date: January, 1989
   Completion Date: December, 1990
   Employment: Graduate School, University of Virginia; PhD candidate on LA²ST Program; Department of Materials Science

6. DESIGN OF CRYOGENIC TANKS FOR SPACE VEHICLES
   Faculty Investigators: W.D. Pilkey and J.K. Haviland
   Graduate Student: Charles Copper
   Degree: MS
   UVa Department: Mechanical and Aerospace Engineering (MAE)
   NASA-LaRC Contact: D.R. Rummler (Structural Mechanics Division), R.C. Davis and M.J. Shuart (Aircraft Structures)
   Start Date: April, 1989
   Completion Date: December, 1990
   Employment: Graduate School, University of Virginia; PhD candidate on NASA-Headquarters sponsored program; Department of Mechanical and Aerospace Engineering
7. ELEVATED TEMPERATURE FRACTURE OF AN ADVANCED RAPIDLY SOLIDIFIED, POWDER METALLURGY ALUMINUM ALLOY
   Faculty Investigator: R.P. Gangloff
   Graduate Student: William C. Porr, Jr.
   Degree: PhD
   UVa Department: MS&E
   NASA-LaRC Contact: C.E. Harris (Mechanics of Materials)
   Start Date: January, 1988
   Completion Date: June, 1992
   Employment: David Taylor Naval Ship R&D Center

8. QUANTITATIVE CHARACTERIZATION OF THE SPATIAL DISTRIBUTION OF PARTICLES IN MATERIALS: APPLICATION TO MATERIALS PROCESSING
   Faculty Investigator: John A. Wert
   Graduate Student: Joseph Parse
   Degree: PhD
   UVa Department: MS&E
   NASA-LaRC Contact: D.R. Tenney (Materials Division)
   Start Date: September, 1988
   Completion Date: June, 1992
   Employment: Private Consultant

9. ENVIRONMENTAL FATIGUE CRACK GROWTH AND CRACKING MECHANISMS IN Al-Li-Cu Alloy 2090
   Faculty Investigator: R.P. Gangloff
   Graduate Student: Donald C. Slavik
   Degree: PhD
   UVa Department: MS&E
   NASA-LaRC Contact: D.L. Dicus (Metallic Materials)
   Start Date: September, 1989
   Completion Date: June, 1993
   Employment: Knolls Atomic Power Laboratory

10. INELASTIC DEFORMATION OF METAL MATRIX COMPOSITES UNDER BIAXIAL LOADING
    Faculty Investigators: Carl T. Herakovich and Marek-Jerzy Pindera
    Graduate Student: Mr. Clifford J. Lissenden
    Degree: PhD
    UVa Department: Civil Engineering and the Applied Mechanics Program
    NASA-LaRC Contact: W.S. Johnson (Mechanics of Materials)
    Start Date: September, 1990
    Completion Date: June, 1993
    Employment: University of Kentucky, Department of Engineering Mechanics
11. EFFECT OF TEMPERATURE ON THE RESPONSE OF METALLIC SHELL STRUCTURES
   Faculty Investigators: W.D. Pilkey and J.K. Haviland
   Graduate Student: Karen McCarthy
   Degree: MS (non-thesis)
   Graduate Student: Theodore Johnson (NASA Minority Grantee)
   Degree: PhD
   Employment: NASA-LaRC
   Graduate Student: Charles Copper
   Degree: PhD
   Employment: AMP Incorporated
   UVa Department: MAE
   NASA-LaRC Contact: M.J. Shuart and Jeffrey Stroud (Aircraft Structures)
   Start Date: April, 1991
   Completion Date: May, 1993

12. EFFECTS OF Zn ADDITIONS ON THE PRECIPITATION AND STRESS CORROSION CRACKING BEHAVIOR OF ALLOY 8090
   Faculty Investigator: Glenn E. Stoner
   Graduate Student: Raymond J. Kilmer
   Degree: PhD
   Department: MS&E
   NASA-LaRC Contact: W.B. Lisagor (Metallic Materials)
   Start Date: September, 1989
   Completion Date: September, 1993
   Cosponsor: Alcoa
   Employment: General Motors

13. PROCESSING AND SUPERPLASTIC PROPERTIES OF WELDALITETM SHEET
   Faculty Investigator: John A. Wert
   Graduate Student: Mark Lyttle
   Degree: MS
   Department: MS&E
   NASA-LaRC Contact: T.T. Bales (Metallic Materials)
   Start Date: September, 1991
   Completion Date: December, 1993
   Employment: Graduate School, University of Virginia; PhD Candidate in Materials Science and Engineering
14. METASTABLE PITTING OF Al ALLOYS AND CRITERIA FOR THE TRANSITION TO STABLE PITTING
   Faculty Investigators: John R. Scully and J.L. Hudson
   Graduate Student: Sheldon T. Pride
   Degree: PhD
   Department: Chemical Engineering
   NASA-LaRC Contact: D.L. Dicus (Metallic Materials)
   Start Date: September, 1991
   Completion Date: May, 1994
   Cosponsor: NASA Graduate Student Researchers Program;
   Under Represented Minority Emphasis
   Employment: Rohm and Haas Chemical Company

15. THE EFFECT OF THERMAL EXPOSURE ON THE MECHANICAL PROPERTIES OF Ti-1100/SCS-6 COMPOSITES
   Faculty Investigator: F.E. Wawner
   Graduate Student: Douglas B. Gundel
   Degree: PhD
   UVa Department: MS&E
   NASA-LaRC Contact: D.L. Dicus and W.B. Brewer (Metallic Materials)
   Start Date: April, 1991
   Completion Date: June, 1994

16. ENVIRONMENTAL EFFECTS IN FATIGUE LIFE PREDICTION: MODELING ENVIRONMENTAL CRACK PROPAGATION IN LIGHT AEROSPACE ALLOYS
   Faculty Investigator: R. P. Gangloff
   Graduate Student: Mark E. Mason
   Degree: MS
   UVa Department: MS&E
   NASA-LaRC Contact: R. S. Piascik (Mechanics of Materials)
   Start Date: January, 1992
   Completion Date: November, 1994
   Employment: Allied-Signal; Hopewell, VA
17. ENVIRONMENTAL EFFECTS IN FATIGUE LIFE PREDICTION: MODELING ENVIRONMENTAL CRACK PROPAGATION IN LIGHT AEROSPACE ALLOYS
   Faculty Investigator: R. P. Gangloff
   Graduate Student: Edward Richey, III
   Degree: MS
   UVa Department: MANE
   NASA-LaRC Contact: R. S. Piascik (Mechanics of Materials)
   Start Date: September, 1993
   Completion Date: May, 1995
   Employment: Graduate School, University of Virginia; PhD Candidate in Materials Science and Engineering

18. HYDROGEN INTERACTIONS IN ALUMINUM-LITHIUM 2090 AND SELECTED MODEL ALLOYS
   Faculty Investigator: John R. Scully
   Graduate Student: Stephen W. Smith; PhD Candidate
   UVa Department: MS&E
   NASA-LaRC Contact: W.B. Lisagor and D.L. Dicus (Metallic Materials)
   Start Date: April, 1991
   Anticipated Completion Date: May, 1995
   Cosponsor: Virginia CIT
   Employment: NASA-LaRC
ADMINISTRATIVE PROGRESS

Faculty Participation

Professor G.J. Shiflet jointed the LA\textsuperscript{2}ST Program during this reporting period and will be collaborating with Professor Starke.

Graduate Student Recruitment

The LA\textsuperscript{2}ST Program has encountered no problems in recruiting the best graduate students entering the participating Departments at UVa, and in sufficient numbers to achieve our education and research objectives. Professor Scully recently recruited Mr. Keith Eklund, currently completing his MS degree in Materials Science at the Pennsylvania State University. His LA\textsuperscript{2}ST research will focus on mechanisms of stress corrosion crack initiation and growth in advanced ingot metallurgy aluminum alloys.

Undergraduate Research Participation

In April of 1990, the LA\textsuperscript{2}ST Program was increased in scope to include undergraduate engineering students. Four students worked at NASA-LaRC during the Summer of 1990, none were recruited for the 1991 program, and seven were successfully recruited to work at NASA-LaRC during the Summer of 1992. Each student was a rising senior in an engineering or science major closely related to aerospace materials and mechanics. Represented universities have included Harvard, Georgia Institute of Technology, Virginia Polytechnic Institute, Duke, the University of Missouri, California Polytechnical Institute, and North Carolina State University.

Professor Glenn E. Stoner assumed responsibility for the 1993 Summer Undergraduate Program. During the past reporting period, he recruited two students from the Pennsylvania State University and North Carolina State University. These students are working in the Metallic Materials Branch at NASA-LaRC for the Summer of 1995.
CURRENT PROJECTS

MECHANICAL AND ENVIRONMENTAL DEGRADATION MECHANISMS IN ADVANCED LIGHT METALS AND COMPOSITES

1. TIME-TEMPERATURE DEPENDENT FRACTURE IN ADVANCED WROUGHT INGOT METALLURGY, AND SPRAY DEPOSITED ALUMINUM ALLOYS
   Faculty Investigator: R.P. Gangloff
   Graduate Student: Michael J. Haynes; PhD (direct) candidate
   UVa Department: MS&E
   NASA-LaRC Contact: A. P. Reynolds (Metallic Materials)
   Start Date: September, 1992
   Completion Date: September, 1996
   Project #1

2. CRYOGENIC TEMPERATURE EFFECTS ON THE DEFORMATION AND FRACTURE OF Al-Li-Cu-In ALLOYS
   Faculty Investigator: R.P. Gangloff
   Graduate Student: John A. Wagner; PhD candidate and NASA-LaRC employee
   UVa Department: MS&E
   NASA-LaRC Contacts: W.B. Lisagor (Metallic Materials) and J.C. Newman (Mechanics of Materials)
   Start Date: June, 1987
   Anticipated Completion Date: December, 1995
   Project #2

3. EFFECTS OF AGING AND TEMPERATURE ON THE DUCTILE FRACTURE OF AA2095 AND AA2195
   Faculty Investigator: R.P. Gangloff
   Graduate Student: Cynthia L. Lach; MS candidate and NASA-LaRC employee
   UVa Department: MS&E
   NASA-LaRC Contacts: W.B. Lisagor (Metallic Materials)
   Start Date: August, 1990
   Anticipated Completion Date: December, 1995
   Project #3
4. MECHANISMS OF LOCALIZED CORROSION IN 2090 AND X2095
   Faculty Investigator: G.E. Stoner
   Graduate Student: Douglas Wall; PhD candidate
   UVa Department: MS&E
   NASA-LaRC Contact: M.S. Domack (Metallic Materials)
   Start Date: April, 1991
   Completion Date: December, 1995
   Cosponsor: Reynolds Metals Company (A. Cho)
   Project #4

5. HYDROGEN INTERACTIONS IN ALUMINUM-LITHIUM 2090 AND SELECTED
   MODEL ALLOYS
   Faculty Investigator: John R. Scully
   Graduate Student: Stephen W. Smith; PhD Candidate
   UVa Department: MS&E
   NASA-LaRC Contact: W.B. Lisagor and D.L. Dicus (Metallic Materials)
   Start Date: April, 1991
   Anticipated Completion Date: May, 1995
   Cosponsor: Virginia CIT
   Project #5

6a. MECHANISMS OF DEFORMATION AND FRACTURE IN HIGH STRENGTH
    TITANIUM ALLOYS: EFFECTS OF TEMPERATURE AND DISSOLVED
    HYDROGEN
    Faculty Investigators: R. P. Gangloff
    Graduate Student: Sean P. Hayes; PhD Candidate
    UVa Department: MS&E
    NASA-LaRC Contact: To be determined (Metallic Materials)
    Start Date: September, 1994
    Completion Date: September, 1997
    Project #6a

6b. MECHANISMS OF DEFORMATION AND FRACTURE IN HIGH STRENGTH
    TITANIUM ALLOYS: EFFECTS OF TEMPERATURE AND MICROSTRUCTURE
    Faculty Investigators: E. A. Starke, Jr.
    Graduate Student: Susan M. Kazanjian, MS Candidate
    UVa Department: MS&E
    NASA-LaRC Contact: To be determined (Metallic Materials)
    Start Date: December, 1994
    Completion Date: To be determined
    Project #6b
AEROSPACE MATERIALS SCIENCE

7. EVALUATION OF WIDE-PANEL ALUMINUM ALLOY EXTRUSIONS
   Faculty Investigator: John A. Wert
   Graduate Student: Mark T. Lyttle, Ph.D. Candidate
   UVa Department: Materials Science and Engineering
   NASA-LaRC Contact: T. T. Bales (Metallic Materials)
   Start Date: January, 1994
   Completion Date: September, 1996
   Project #7

8. Al-Si-Ge-Cu ALLOY DEVELOPMENT
   Faculty Investigator: E.A. Starke, Jr.
   Graduate Student: H.J. Koenigsmann, Ph.D. Candidate
   UVa Department: Materials Science and Engineering
   NASA-LaRC Contact: W.B. Lisagor
   Start Date: September, 1993
   Completion Date: To be determined
   Project #8.

9. EFFECTS OF TEXTURE AND PRECIPITATES ON MECHANICAL PROPERTY ANISOTROPY OF Al-Cu-Mg-X ALLOYS
   Faculty Investigators: E.A. Starke, Jr. and G.J. Shiflet
   Graduate Student: None
   Post Doctoral Research Associate: B. Skrotzki
   UVa Department: Materials Science and Engineering
   NASA-LaRC Contact: W.B. Lisagor
   Start Date: January, 1995
   Completion Date: To be determined
   Project #9.

MECHANICS OF MATERIALS FOR LIGHT AEROSPACE STRUCTURES

10. DAMAGE EVOLUTION IN POLYMERIC COMPOSITES
    Faculty Investigator: C. T. Herakovich
    Graduate Student: R. D. Schroedter III, MS Candidate
    UVa Department: Civil Engineering & Applied Mechanics
    NASA-LaRC Contact: C. E. Harris & T. Gates
    Start Date: September 1993
    Completion Date: December, 1995
    Project #10
11. ENVIRONMENTAL EFFECTS IN FATIGUE LIFE PREDICTION: MODELING ENVIRONMENTAL CRACK PROPAGATION IN LIGHT AEROSPACE ALLOYS

Faculty Investigator: R.P. Gangloff
Graduate Students: Zuhair Gasem; PhD Candidate
                  Edward Richey III; MS Candidate (Mechanical and Aerospace Engineering)

UVa Department: MS&E
NASA-LaRC Contact: R.S. Piascik (Mechanics of Materials)
Start Date: January, 1992
Anticipated Completion Date: June, 1995 (Richey)
                            December, 1996 (Gasem)

Project #11

THERMAL GRADIENT STRUCTURES

None
Research progress, recorded during the period from January 1, 1995 to June 30, 1995, is summarized for each project in the following sections. The standard format includes the program objective, recent progress, conclusions, and immediate milestones; coupled with a set of visual aids given in graduate student presentations at the Seventh Annual NASA-UVa LA²ST Grant Review Meeting held at NASA-LaRC in July of 1995. The agenda of this meeting is presented in Appendix IV.

Project #1  Time-Temperature Dependent Fracture in Advanced Wrought Ingot Metallurgy and Spray Deposited Aluminum Alloys

Michael J. Haynes and Richard P. Gangloff

Objective

The objective of this study is to quantitatively characterize plane strain initiation ($K_{Ic}$) and plane stress growth toughnesses of advanced Al-Cu-Mg-Ag alloys as a function of temperature. The effect of temperature on $K_{Ic}$ is micromechanically modeled, employing temperature dependent measurements of elastic modulus, intrinsic fracture strain, yield strength, and work hardening, coupled with an estimate of the critical microstructural distance relevant to dimpled rupture. Metallurgical aspects of elevated temperature microvoid fracture; including second phase particle distributions, shear localization during coalescence, and dynamic recovery; are identified and incorporated into explanations of time-temperature dependent fracture processes.

Recent Findings

Direct current potential difference crack length monitoring, coupled with J-integral fracture mechanics, successfully characterizes initiation and growth toughness of thin sheet compact tension [C(T)] specimens. Rising load experiments on AA2650-T6, for C(T) thicknesses of 3.2 mm and 6.0 mm, demonstrate that $K_{Ic}$ from DCPD detected initiation is nearly identical for the two thicknesses ($K_{Ic} = 28.0$ MPa$m$). In contrast measured fracture
toughness ($K_{JC}$) from a 0.2 mm offset blunting line definition of fracture initiation (ASTM E813) is 30% higher for the 3.2 mm thick C(T) specimen. The 0.2 mm offset in $\Delta a$ corresponds to a significant amount of process zone damage and a substantial loss in plane strain constraint. The R-curve rises rapidly in the low $\Delta a$ regime, resulting in the inflated $K_{JC}$. For the 6.0 mm thick specimen the plane strain constraint is maintained and $K_{JC}$ approaches $K_{JCI}$.

Ambient and elevated temperature initiation ($K_{JCI}$) and growth ($K_{J}^{2mm}$) fracture toughnesses have been characterized for a commercial aluminum alloy (AA2024), a low Fe and Ni version of AA2618 (AA2650), and three Ag bearing, 2519-type alloys (AA2519+Mg+Ag, C416, and spray formed N203). AA2519+Mg+Ag and C416 display superior initiation and growth fracture toughness at ambient temperature. Both alloys maintain their initiation toughness to elevated temperatures, and C416 displays the highest $K_{JCI}$ and $K_{J}^{2mm}$ at elevated temperatures. Spray formed N203, with a 1 to 5 $\mu$m grain size, does not display the elevated temperature ductility and fracture toughness degradations seen in submicron grain-size aluminum alloys such as AA8009 and high purity cryogenically milled aluminum.

The critical plastic strain-controlled model successfully predicts the temperature dependency of initiation fracture toughness for a wide variety of advanced aluminum alloys, including AA2519+Mg+Ag, AA2618, AA2095, AA2195, spray formed N203, AA8009, and cryogenically milled aluminum. Predictions can be reasonably based on smooth bar tensile properties and an estimate of the reduction in smooth bar strain to failure ($\varepsilon^p$) to a triaxial-stress-state constraint corresponding to the crack tip. Temperature insensitive $K_{JCI}$ is predicted and observed for precipitation hardened 2000 series alloys, while a degradation of $K_{JCI}$ is correctly modelled in submicron grain size alloys. The temperature dependencies of $K_{JCI}$ are traceable to the interplay between thermally sensitive process zone fracture resistance ($\varepsilon^p$) and the temperature-dependent crack tip strain field (dependent on $\sigma_{yt}$, $E$, and $N$). Both components are necessary to successfully predict temperature insensitive initiation toughness in precipitation hardened alloys, where $\varepsilon^p$ rises with temperature and $\sigma_{yt}$, $E$, and $N$ decline.

The critical distance ($L^*$), calculated from the model by equating measured and predicted fracture toughness at ambient temperature, correlates with the nearest neighbor particle spacing in a volume for a variety of aluminum and steel alloys. The data are divided into two correlations, based on whether the alloy contains a unimodal or bimodal size distribution of void
nucleating particles. For a bimodal distribution of particles, the linear fit suggests that the critical fracture initiation event occurs over roughly one particle spacing. For a unimodal distribution, a linear fit suggests that the critical fracture event occurs over 4 to 5 particle spacings. These results are preliminary.

Strain localization between growing microvoids ("Intravoid Strain Localization" or ISL) is hypothesized to have a large influence on the intrinsic fracture resistance (alloy ductility) since subsequent void sheeting truncates primary void growth, which would otherwise dissipate a large amount of strain energy prior to coalescence by impingement. The progression or abatement of ISL depends on the competition between intravoid flow hardening (from strain and strain rate hardening) and intravoid flow softening (partly from void nucleation at dispersoids within an ISL band). As temperature increases, the work hardening exponent (N from $\sigma = K\varepsilon^n$) declines, the strain rate hardening exponent (m from $\sigma = K\dot{\varepsilon}^m$) increases, and the propensity for void nucleation at dispersoids decreases. Competition between these three variables (in promoting or retarding ISL) results in reduced ISL and void sheeting at elevated temperatures, and an associated rise in $\dot{\varepsilon}_f^*$. 

**Milestones**

Two manuscripts, prepared for submittal to *Metallurgical Transactions A* and an ASTM *Special Technical Publication* (STP), have been written and are currently in the revision process. These papers should be completed by September. Another paper, on characterization of initiation toughness and J-$\Delta$a behavior will be written for submittal to the ASTM *Journal of Testing and Evaluation*. Mr. Haynes successfully completed the research proposal for his PhD program.

In the next reporting period, we will study intravoid strain localization in AA2519+Mg+Ag and spray formed N203. Stress relaxation tests will be employed to characterize strain rate hardening over a wide range of strain rate and temperature. Smooth and notched tensile bars will be interrupted at various damage levels (as detected by DCPD monitoring) and sectioned for microstructural observation of the microvoid failure processes. Ultra-sensitive density measurements will be employed to quantify void damage.
Presentation Captions

1. Title.

2. Project objectives.

3. Can one accurately characterize initiation ($K_{IC}$) and growth ($K_J^{2mm}$) fracture toughnesses using specimens of reduced width and thickness?

4. Chemical compositions of precipitation hardened alloys (AA2519+Mg+Ag, C416, N203, AA2650, AA2024) characterized for initiation and growth fracture toughness.

5. Photograph illustrating the experimental set up for fracture toughness testing of thin sheet C(T) specimens with buckling restraints. Load, crack length from electrical potential and crack mouth opening displacement are digitally recorded for input to the calculation of $J$ versus $\Delta a$.

6. Plane strain character of crack initiation in a thin sheet C(T) specimen and the subsequent transition to plane stress fracture as the crack extends.

7. $K_J$ versus $\Delta a$ for two thicknesses of C(T) specimens of AA2650 at 25°C, showing: (1) the sensitivity of the offset blunting line (ASTM E813) measurement of initiation toughness ($K_{IC}$) to C(T) specimen thickness, and (2) the insensitivity of direct current potential difference (DCPD) detected initiation toughness to specimen thickness. Each C(T) specimen was machined from the central portion of thick plate AA2650.

8. $K_{IC}$ versus temperature for a variety of precipitation hardened alloys (AA2024, AA2650, AA2519+Mg+Ag, C416, and N203).

9. Summary bar chart of ambient temperature growth toughness ($K_J^{2mm}$) for precipitation hardened alloys AA2650, N203, AA2024, C416, AA2519+Mg+Ag, and AA2519+Mg, displaying the superior growth toughness of the 2519-type ingot metallurgy alloys (AA2519+Mg, AA2519+Mg+Ag, and C416).

10. Summary bar chart of ambient and elevated temperature $K_J^{2mm}$ for precipitation hardened alloys AA2519+Mg, AA2650, AA2519+Mg+Ag, and C416, displaying the superior elevated temperature $K_J^{2mm}$ for Ag-bearing C416.

11. Can one quantitatively predict the temperature dependence of $K_{IC}$ in ingot metallurgy, spray formed, and submicron grain size powder metallurgy aluminum alloys?

12. Chemical compositions of aluminum alloys that were micromechanically modelled to predict initiation toughness.
13. Schematic representation of the critical plastic strain-controlled model depicting three model elements: (1) the effective plastic strain field ahead of a crack tip ($\varepsilon^p$), (2) the intrinsic fracture resistance relevant to a high stress state triaxiality ($\varepsilon^*_f$), and (3) the fracture criterion of $\varepsilon^p$ exceeding $\varepsilon^*_f$ over a critical microstructural distance ($l^*$). By combining these three elements, $K_{JCI}$ is predicted as a composite property dependent on yield strength, modulus, work hardening, constituent particle spacing, and fracture ductility (reduction in area). Each of these latter material properties depends on temperature.

14. Temperature-dependent tensile properties ($\sigma_y$ and %RA) of AA2519+Mg+Ag and spray formed N203. AA2519+Mg+Ag possesses superior yield strength and ductility.

15. Temperature dependent work hardening exponent (N) for spray formed N203 and AA2519+Mg+Ag, showing superior work hardening capacity of lower strength N203.

16. Micromechanical model predictions of temperature dependent $K_{JCI}$ for AA2519+Mg+Ag compared to experimental measurements. Two sets of predictions were produced, based on reduction in area measurements of smooth and notched round tensile bars.

17. Micromechanical model predictions of temperature dependent $K_{JCI}$ for spray formed N203 and AA2618 compared to experimental measurements.

18. Micromechanical model predictions of cryogenic to elevated temperature $K_{JCI}$ for AA2095 and AA2195 compared to experimental measurements.

19. Elevated temperature dependence of normalized intrinsic fracture strain for AA2519+Mg+Ag, cryogenically milled aluminum, and AA8009.

20. Micromechanical model predictions of temperature dependent $K_{JCI}$ for AA8009 and cryogenically milled aluminum, compared to experimental measurements.

21. Correlation between $l^*$ and the nearest neighbor spacing of void nucleating constituents in a volume for aluminum alloys and steels, subdivided into unimodal and bimodal particle size distribution classes.

22. Hypothesis: the intrinsic fracture resistance of 2000 series aluminum alloys increases with increasing temperature due to a decreasing tendency for Intravoid Strain Localization (ISL) and a decreased tendency for the associated plastic shear instability between growing microvoids.

23. Experimental evidence of retarded ISL behavior at elevated temperature, obtained from crack tip profiles of AA2519+Mg+Ag.

24. List of continuum and microstructural variables governing ISL.
25. Schematic representation of ISL between two growing microvoids (nucleated at constituent particles), illustrating important features such as: (1) stress and strain rate amplification within the band, and (2) flow hardening in the band due to local strain and strain rate hardening as well as flow softening due to void nucleation at submicron dispersoids.

26. Fractographs displaying the role of constituent particle distribution in influencing ISL behavior.

27. Explanation of retarded void sheeting and enhanced %RA at elevated temperature in AA2519+Mg+Ag. At ambient temperature, intravoid flow hardening due to strain and strain rate hardening is insufficient to overcome intravoid flow softening (partly from void nucleation at dispersoids). ISL and void sheeting is prevalent. At elevated temperatures, intravoid flow hardening is substantial and sufficient to delay the onset of ISL and void sheeting, due to enhanced strain rate hardening and reduced void nucleation at dispersoids. Fracture ductility increases due to enhanced stable growth of primary microvoids.

28. Correlation between strain rate sensitivity, m, and $\varepsilon^*_f$ for AA2519+Mg+Ag and AA2618. m was calculated from creep data for AA2219 and AA2618, while $\varepsilon^*_f$ was calculated from %RA.

29. Conclusions.
Elevated Temperature Fracture of Advanced Aluminum Alloys

Michael J. Haynes and Richard P. Gangloff

July 17, 1995

Funded by NASA Langley Research Center
W.B. Lisagor, Project Monitor
Objectives of Elevated Temperature Fracture Studies

- Characterize the initiation and growth fracture toughness of advanced aluminum alloys as a function of temperature.
- Micromechanically model initiation fracture toughness \((K_{\text{Icl}})\) as a function of temperature.
- Identify metallurgical and continuum aspects of temperature dependent microvoid fracture.
CAN ONE ACCURATELY CHARACTERIZE INITIATION ($K_{JIC}$) AND GROWTH ($K_{J2mm}$) FRACTURE TOUGHNESSES USING SPECIMENS OF REDUCED WIDTH AND THICKNESS?
### Chemical Compositions of Characterized Alloys (Wt%)

<table>
<thead>
<tr>
<th>Alloy Designation</th>
<th>Cu</th>
<th>Mg</th>
<th>Ag</th>
<th>Mn</th>
<th>Zr</th>
<th>V</th>
<th>Fe</th>
<th>Si</th>
<th>Ti (Ni)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA2519-T87+Mg+Ag</td>
<td>5.75</td>
<td>0.52</td>
<td>0.50</td>
<td>0.3</td>
<td>.16</td>
<td>.09</td>
<td>.05</td>
<td>0.04</td>
<td>---</td>
</tr>
<tr>
<td>C416-T8</td>
<td>5.40</td>
<td>0.50</td>
<td>0.50</td>
<td>0.3</td>
<td>.13</td>
<td>---</td>
<td>.06</td>
<td>.04</td>
<td>---</td>
</tr>
<tr>
<td>N203-T6</td>
<td>4.90</td>
<td>0.50</td>
<td>0.40</td>
<td>0.50</td>
<td>0.40</td>
<td>0.22</td>
<td>---</td>
<td>---</td>
<td>0.19</td>
</tr>
<tr>
<td>AA2650-T6</td>
<td>2.71</td>
<td>1.64</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>0.20</td>
<td>0.21</td>
<td>(.21)</td>
</tr>
<tr>
<td>AA2024-T3</td>
<td>4.4</td>
<td>1.5</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
</tbody>
</table>

**AA2519+Mg+Ag, C416:** Ingot metallurgy sheet (0.125" and 0.090" thick), Ω precipitate strengthened

**N203:** Spray formed (SF), precipitation hardened extrusion (1")- Ω strengthened

**AA2650:** Ingot metallurgy plate (0.250" thick), S' and θ' precipitate strengthened

**AA2024:** Ingot metallurgy sheet (0.125" thick), Naturally Aged (θ' precipitates)

**Thickness is 0.125" for C(T) specimens of AA2519+Mg+Ag, N203, AA2650, and AA2024**

**Thickness is 0.090" for C(T) specimens of C416**
C(T) Specimen Detail for Thin Sheet J-Δa Determination
Low magnification SEM fractograph of a 2519-T87 (+Mg+Ag) fracture surface produced at 25°C showing the plane strain flat fracture and transition to plane stress cracking. The shear lip - flat fracture interface is indicated by arrows, with the fatigue precrack just visible parallel to the bottom edge of the photo.
AA2650-T6

\( \frac{d\delta}{dt} = 0.26 \text{ \(\mu\)m/sec} \)

25°C

\( K_{\text{JC}} = 39.2 \text{ MPa}\sqrt{\text{m}} \)

\( K_{\text{JIC}} = 30.1 \text{ MPa}\sqrt{\text{m}} / K_q = 30.5 \text{ MPa}\sqrt{\text{m}} \)

\( K_{\text{JICl}} = \{ \nabla \ 27.8 \text{ MPa}\sqrt{\text{m}} \)

\( \triangle \ 28.1 \text{ MPa}\sqrt{\text{m}} \)

- ▼ 3.2 mm plate
- ▲ 6.0 mm plate (slidegrooved)
- 0.2 mm offset blunting line

\( \Delta a \) (mm)
Fracture Toughness, $K_{JIC}$ (MPa$\sqrt{m}$) vs Temperature (°C)

- **Al-Cu-Mg-Ag Alloys**
  - ▲ AA2519+Mg+Ag
  - ■ C416
  - ▼ Spray Formed N203

- **Al-Cu-Mg Alloys**
  - □ AA2024
  - ○ AA2650

The equation for $K_{JIC}$ is given as:

$$K_{JIC} = \sqrt{\frac{J_{IC}}{1-\nu^2}}$$
CAN ONE QUANTITATIVELY PREDICT THE TEMPERATURE DEPENDENCE OF $K_{IC}$ IN INGOT METALLURGY, SPRAY FORMED, AND SUBMICRON GRAIN SIZE (PM) ALLOYS?
### Chemical Compositions of Ingot Metallurgy, Spray Formed, Ultra-Fine Grain Size, and Metal Matrix Composite Alloys for Modelling

<table>
<thead>
<tr>
<th>Alloy Designation</th>
<th>Alloy Class</th>
<th>Product Form</th>
<th>Nominal Alloy Composition (weight %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA2095-T6</td>
<td>I/M</td>
<td>Plate</td>
<td>Al-4.6Cu-1.5Li-0.35Ag-0.4Mg-0.15Zr</td>
</tr>
<tr>
<td>AA2195-T6</td>
<td>I/M</td>
<td>Plate</td>
<td>Al-4.0Cu-1.0Li-0.35Ag-0.4Mg-0.15Zr</td>
</tr>
<tr>
<td>AA2618-T851</td>
<td>I/M</td>
<td>Plate</td>
<td>Al-2.6Cu-1.6Mg-1.0Fe-1.2Ni-0.2Si</td>
</tr>
<tr>
<td>AA2519-T87(+Mg+Ag)</td>
<td>I/M</td>
<td>Sheet</td>
<td>Al-5.75Cu-0.5Mg-0.5Ag-0.3Mn-0.15Zr-0.1V</td>
</tr>
<tr>
<td>N203-T6</td>
<td>SF</td>
<td>Extrusion</td>
<td>Al-5.0Cu-0.5Mg-0.5Mn-0.4Zr-0.4Ag-0.2Ti-0.2V</td>
</tr>
<tr>
<td>CM Al</td>
<td>SM/GS</td>
<td>Extrusion</td>
<td>Al + 2.5 vol% Al₂O₃</td>
</tr>
<tr>
<td>AA8009</td>
<td>SM/GS</td>
<td>Extrusion</td>
<td>Al-8.5Fe-1.3V-1.7Si</td>
</tr>
<tr>
<td>2009/SiC/20p-T6</td>
<td>MMC</td>
<td>Plate</td>
<td>Al-3.6Cu-1.3Mg + 19.5 vol% SiC</td>
</tr>
</tbody>
</table>
Critical Plastic Strain Controlled Model

\[ K_{JICi} = \sqrt{\frac{\sigma_{ys} E I^*}{(1-v^2) d_n \left( \frac{\bar{\varepsilon}_f^*}{C1} \right) \frac{1}{C2}}} \]

\[ \bar{\varepsilon}_f^* = \frac{1}{\gamma} \left( -\ln(1-\%RA) \right) \]
Spray Formed N203

AA2519+Mg+Ag

\[ \sigma = K \varepsilon^N \]
AA2519-T87 (+Mg +Ag)
3.2 mm, LT Sheet

Least Squares Fit to Measurements

- Measured
- Predicted $\varepsilon_f$ from Notched Bar RA ($r_n=1.5$)
- Predicted $\varepsilon_f$ from Smooth Bar RA ($r_s=6.5$)

$L^* = 20.5 \mu m$
**N203 Extrusion**
- Measurement
- Model Prediction \( (r = 7, l^* = 20.3 \, \mu m) \)

**AA2618-T851 Plate**
- Measurement
- Model Prediction \( (r = 7, l^* = 14.8 \, \mu m) \)
Least Squares Regression of Predictions

AA2195-T8, 3.8mm Sheet

AA2095-T8, 3.8mm Sheet

Temperature (°C)

K_{JIC} (MPa√m)

- Predicted (r =7, l^* =29.6 μm)
- Predicted (r =7, l^* =21.9 μm)

Measured

Δ Measured
RS/PM AA8009 Extrusion
- Measured
- Predicted ($r = 7$, $l^* = 16.8 \, \mu m$)

Cryogenically Milled Al Extrusion
- Measured
- Predicted ($r = 7$, $l^* = 8.0 \, \mu m$)

$K_{JIC}$ (MPa$\sqrt{m}$)

Temperature (°C)
Nearest Neighbor Spacing in a Volume, \( \Delta_3 \) (\( \mu \text{m} \))

Critical Microstructural Distance, \( l^* \) (\( \mu \text{m} \))

**Bimodal Particle Size**
- AA2618-T851
- AA2095-T6
- N203-T6
- AA2519-T87(+Mg+Ag)
- Fe-C-Ni-Cr-Mn (+Ni & Si)

**Unimodal Particle Size**
- AA2195-T8
- AA2009/SiC/20p-T6
- HP9-4-20#1 Steels
- 4340-Type Steels
- Fe-C-Ni-Cr-Mn (+Ni or Si)
HYPOTHESIS

The intrinsic fracture resistance of 2000 series aluminum alloys increases with increasing temperature due to a decreasing tendency for Intravoid Strain Localization (ISL) and a decreased tendency for the associated plastic shear instability between growing microvoids.
Crack tip profiles from midplane of C(T) specimens fractured at: (a) $25^\circ$C and (b) $150^\circ$C; load-line displacement rate $= 0.26 \, \mu m/sec$. Arrows indicate void sheeting.
Microstructural Variables

1. Spatial Distribution of Primary or Large Voids
2. Primary Void Shape (Projected Area Normal to Stress)
3. Void Nucleation at Small Second Phase Particles (Dispersoids)
4. Slip Mode

Continuum Variables

1. Global Stress State Triaxiality
2. Strain Hardening
3. Strain Rate Hardening

(1) Strongly Temperature Dependent
(2) May be Temperature Dependent Depending on Alloy System
Globally Applied
Effective Stress, Triaxiality, Effective Strain, Effective Strain Rate
\( \bar{\sigma}_{gl}, \sigma_m/\bar{\sigma}_{gl}, \bar{\varepsilon}_{gl}, d\bar{\varepsilon}_{gl}/dt \)

\( \sigma_{o: gl}, n_{gl}, m_{gl} \)

\( \dot{\sigma}_L \) and \( d\dot{\varepsilon}_L/dt \)

Intravoid Flow Properties:
\( \sigma_{o: L}, m_L, n_L \)

2.0 \( \mu \)m

10 \( \mu \)m
Role of Undissolved $\Theta$ Constituent in 158

25°C

2519-T87(+Mg+Ag)
High $V_r$ of Undissolved $\Theta$

C416-T8
Low $V_r$ of Undissolved $\Theta$

150°C
Void sheeting is retarded (and $\tilde{\varepsilon}_f^*$ increases) at elevated temperatures because:

**At ambient temperature:**

- Intravoid flow softening is substantial, due to void initiation and growth at submicrometer dispersoids.

- Strain and strain rate hardening are low; thus intravoid flow hardening is relatively low.

- Flow hardening is insufficient to delay the onset of ISL. Relative to elevated temperatures, void sheeting is prevalent and the extent of stable void growth and $\tilde{\varepsilon}_f^*$ are low.

**As temperature is increased:**

- Intravoid flow softening is reduced, due to dislocation bypassing of dispersoids by climb.

- Strain hardening decreases, but strain rate hardening increases; intravoid flow hardening remains constant or increases.

- Intravoid flow hardening is substantial enough to delay the onset of ISL. Void sheeting is reduced, the extent of stable void growth increases and $\tilde{\varepsilon}_f^*$ increases.

**NEED:** Quantify the roles of $m$, $N$, and intravoid flow softening on the onset of strain localized coalescence.
Correlations between strain rate sensitivity, \( m \), and \( \bar{\varepsilon}_f^* \) for 2519-T87 (+Mg+Ag) and 2618-T851. \( \bar{\varepsilon}_f^* \) values are derived from smooth and notched tensile specimen reduction in areas by employing Eq. [6]; \( r_n = 1.53 \) for notched bars and \( r_s = 6.5 \) for smooth bars.
Conclusions

- Initiation ($K_{JIC}$) and growth ($K_{J2mm}$) are characterized accurately by a $J$-integral fracture mechanics method and direct current potential difference crack length monitoring, with potential for small-specimen experiments.

- The offset blunting line definition of initiation toughness from ASTM E813 overestimates the initiation toughness of thin sheet aluminum alloys (despite satisfying the thickness criterion), due to a sharply rising R-curve associated with a loss in plane strain constraint. The DCPD method detects fracture initiation closer to the fatigue crack tip and appears to be insensitive to specimen thickness, at least down to a 1/8" gage.

- At ambient temperature, the 2519-type alloy sheets display superior plane stress toughness ($K_{J2mm}$) over 2024-T3, 2650-T6, and spray formed N203-T6.

- At elevated temperatures, C416-T8 shows the best plane stress toughness ($K_{J2mm}$) compared to 2519-T87(+Mg+Ag), 2519-T87(+Mg), and 2650-T6.

- The magnitude and temperature dependence of $K_{JIC}$ is accurately predicted for a variety of advanced aluminum alloys by a strain-based micromechanical model of microvoid fracture.

- Initiation fracture toughness is a composite property, composed of temperature dependent intrinsic alloy flow ($\sigma_{ys}$, $E$, $N$) and fracture ($\epsilon^*_f$, %RA) properties.

- $l^*$ correlates to the nearest neighbor spacing of void nucleating constituents in a volume for aluminum alloys and steels.

- The intrinsic fracture strain, $\epsilon^*_f$, is strongly influenced by ISL (strain localization between primary voids nucleated at constituents). The prevalence of ISL at ambient versus elevated temperatures depends directly on the propensity for void nucleation at dispersoids and is inversely related to the levels of strain and strain rate hardening at these temperatures.
Objective

The objective of this Ph.D. research is to characterize and optimize the crack initiation and growth fracture resistance Al-Li-Cu (alloy 2090) and Al-Li-Cu-In (alloy 2090+In) for cryogenic tank applications. The program aims to understand microscopic fracture events; as influenced by ambient to cryogenic temperatures, stress state and microstructure. The specific goal is to determine the mechanisms associated with delamination and transgranular shear (TGS) fracture.

Status

The current work in this project has been focusing on the details of the mechanisms associated with fracture of 2090-T81 at intermediate temperatures in the range 25°C to -185°C and on the microtexture of fracture. Previous work has focused on the fracture behavior at the two extreme temperatures. To gain a better understanding of the mechanisms of fracture tensile, notched tensile and toughness tests were conducted at intermediate temperatures. In addition, microtexture studies were conducted using the Electron Backscattered Pattern (EBSP) technique.

Recent Results

Fracture toughness R-curve behavior was determined at intermediate temperatures using 12.0 mm thick side-grooved compact tension (CT) specimens in the LT orientation. The largest increase in toughness, as determined by R-curve behavior, was observed to occur from -155°C to -185°C. Associated with the increase in toughness at -185°C was an increase in the occurrence of large delaminations. In addition, long range transgranular shear in which numerous grains are traversed by a single shear crack, as observed at ambient temperature, tends to “break-up” as the test temperature decreases.

Transverse uniaxial tensile specimens and longitudinal notched tensile specimens were machined from the plate midplane and tested in the temperature range 25°C to -185°C. There was
a fairly linear increase in uniaxial yield strength with a decrease in temperature typical of 2090 in the T81 condition. There also was an increase in the work hardening exponent, n, with a decrease in temperature. The largest change in n was observed to occur at the lowest temperature and is probably related to more homogenous deformation at -185°C. Similarly, effective plastic strain to failure ($e_p$) increased with decreasing temperature.

Grain orientation of individual (sub)grains was determined using the EBSP technique. A typical microtexture analysis around the area adjacent to a delamination involved obtaining an EBSP pattern across a 300 μm linear region. EBSP {111} pole figures around delaminations at the midplane were similar to the X-ray pole figure. Results of EBSP analysis indicate that delaminations typically occur along high angle boundaries (27° to 40°). In addition, it was observed that one grain adjacent a given delamination was within 15° of the ideal brass orientation. In some cases, the second grain had an orientation close to the S orientation.

**Milestones**

In the next six months, a fourth publication will be completed and published. Significant progress will be made toward the completion of the PhD dissertation.

**Presentation Graphics Captions**

1. Title.
2. Problem statement and program objective.
3. Current program emphasis.
4. Backscattered SEM images of the through thickness microstructure of 2090 in the near peak aged condition.
5. R-curve fracture toughness behavior increases with decreasing temperature with the largest increase between -155°C and -185°C.
6. $K_{I_{crit}}$ was determined from the intersection the power law regression line of the $J$-$\Delta a$ data and the 0.1 mm offset of the blunting line. In the temperature range 25° to -155°C, $K_{I_{crit}}$ remains almost constant, but increases between -155°C and -185°C.
7. Both yield strength and work hardening exponent increased with decreasing temperature typical of 2090-T81 plate.

8. Effective plastic strain to failure increased with decreasing temperature presumably due to more homogeneous deformation at the cryogenic temperature.

9. Increase in fracture toughness at -185°C was associated with an increase in delamination fracture.

10. Higher magnification of delaminations associated with fracture at -185°C.

11. Schematic of instrumentation employed for EBSP studies of boundary misorientation and microtexture analysis.

12. L-S section of a compact tension specimen tested at -185°C showing typical delaminations examined using the EBSP technique.

13. (Sub)grain boundary misorientation indicating that delaminations occur at high angle boundaries with a misorientation of approximately 27° to 40°.

14. X-ray {111} pole figure at the midplane of 2090-T81 plate indicating a strong deformation brass texture.

15. EBSP {111} pole figure at the midplane of 2090-T81 plate around a delamination showing good agreement with the X-ray pole figure.

16. Backscattered images of interrupted notch tensile test showing delaminations occurred at boundaries with significant amounts of precipitation.

17. Summary.
FRACTURE OF Al-Li ALLOYS 2090 AND 2090+In AT CRYOGENIC TEMPERATURES

FRACTURE AND MICROTEXTURE OF Al-Li ALLOY 2090

John A. Wagner and Richard P. Gangloff
LA²ST Program Review
NASA Langley Research Center
July 17-18, 1995
PROGRAM PROBLEM STATEMENT AND OBJECTIVE

Fracture of 2090 and 2090+In Alloys

Problem

- No systematic investigation conducted to determine the interactive effects of:
  - Temperature
  - Delamination
  - Indium addition
  - Microstructure

  on the fracture of 2090-based alloys.

Objective

- Determine the influences of microstructure, stress state and temperature on the occurrence of various fracture modes in Al-Li-Cu-X alloys. Understand the mechanism(s) associated with delamination and transgranular shear fracture.
FRACTURE OF 2090 AND 2090+In ALLOYS

Current Program Emphasis

• Understand the fracture mode transition of 2090-T81 at various temperatures in the range 25°C to -185°C.

• Examine boundary misorientation and microtexture associated with delamination and transgranular shear fracture.
Backscattered Electron Micrograph Showing the Through-Thickness Microstructure of 2090-T81
R-curves of 12mm thick specimens at -185°C to 25°C.
Fracture Toughness, $K_{JIc_i}$, of 12mm Thick 2090-T81 as a Function of Temperature
Yield Strength and Work Hardening Coefficient of 2090-T81 as a Function of Temperature
Effective Plastic Strain To Failure of 2090-T81
As A Function Of Temperature ($\sigma_m/\sigma=1.03$)
Fracture surface cross section of 2090-T81 as a function of temperature.
Optical L-S Section of the Fracture Surface of a 2090-T81 Specimen Tested at -185°C
Schematic Representation of the Setup Used for the Electron Backscattered Pattern (EBSP) Technique
OPTICAL MICROGRAPH IN POLARIZED LIGHT OF AN L-S SECTION OF SAMPLE TESTED AT -185°C
MISORIENTATION ANGLE AS A FUNCTION OF THE (SUB)GRAIN NUMBER, ALONG ND
X-ray \{111\} Pole Figure at T/2 for 2090-T81
EBSP \{111\} Pole Figure of a Region Around Delamination
BACK SCATTERED IMAGES OF THE MIDPLANE OF INTERRUPTED NOTCH TENSILE TEST AT -185°C
FRACTURE AND MICROTEXTURE OF Al-Li ALLOY 2090

Summary

• Fracture toughness R-curve behavior increased with decreasing temperature with the largest increase between -155°C to -185°C.

• Fracture at -185°C was associated with large delaminations which occurred at high angle boundaries (27° - 40°).

• High angle boundaries where delaminations occurred were in some cases bounded by one grain of the brass texture and one of the S texture.
**Objective**

The objective of this research is to characterize the effect of cryogenic to mildly elevated temperature on the tensile deformation and fracture toughness of an emerging composition of Weldalite™-type alloys. We will determine quantitative stress versus strain data, and initiation and growth fracture toughnesses, as well as the associated microscopic fracture mechanisms, and conduct micromechanical modeling in order to understand temperature-dependent fracture.

**Status**

Two Weldalite™ compositions were selected for study to examine the limits of the Cu and Li levels for the alloy registered as AA2095. Specifically, a high Cu-Li alloy (Al-4.64Cu-1.53Li-0.34Ag-0.37Mg-0.17Zr; wt%) at the upper extreme of the AA2095 specification, and a low copper alloy (Al-4.04Cu-1.00Li-0.37Ag-0.36Mg-0.15Zr; wt%) at the low end of the AA2095 composition specification, and in the middle of the AA2195 specification, were chosen for evaluation. Uniaxial tensile stress-strain relationships and J-Integral based crack initiation and growth fracture toughnesses were determined as a function of temperature for AA2195 (aged for 30 hours at 143°C). Selected tests were conducted on AA2095 aged for 20 hours at 143°C for comparison purposes.

The following experiments were conducted on the alloy variants during this reporting period: ICP chemical analysis at various locations through the thickness of the plate, an extended aging study, precision modulus tests at -180°C and 25°C, and uniaxial tensile and fracture toughness tests from -185°C to 135°C.

**Recent Results**

The results of the chemical analyses of all of the alloys suggest a significant level of Cu segregation, as indicated by the consistently low Cu content at the mid-plane (t/2) location. The
least amount of Cu segregation with respect to the mid-plane was observed for X2095 (AA2095), while the greatest was observed for the X2095 (AA2195) alloy.

The high Cu-Li alloy variant (AA2095) did not overage at 143°C in 768 hours, while the low Cu-Li variant (AA2195) began overaging between 100 and 198 hours.

The modulus of AA2195 (aged for 30 hours at 143°C) increases by approximately 6% from ambient to cryogenic temperatures (-185°C). Increased yield strength and work hardening are observed for each alloy with decreasing temperature to cryogenic levels. For a given yield strength, the fracture toughness values were significantly lower for the high Cu-Li alloy variant at both test temperatures. This result was supported by the presence of a large volume fraction of undissolved particles in the high Cu-Li variant because its composition exceeds solid solubility at the solution treatment temperature that was employed, as discussed in previous progress reports.

**Milestones**

Fracture surfaces will be examined by SEM to detect temperature dependent changes in the details of the microvoid-based fracture process.

The critical plastic strain-controlled model developed by Ritchie and coworkers will be employed to predict the temperature dependence of $K_{IC}$ based on measured tensile properties for the initial aging study that was conducted on both alloys.

**Presentation Graphics Captions**

1. Title.

2. Objective.

3. Approach.

4. The table displays the registered compositional limits of Weldalite™ 049 alloys. The original lean and rich chemistries were chosen to reflect the upper and lower limits of the X2095 alloy. The lean variant (4 wt%Cu-1.0 wt%Li) also falls in the middle of the AA2195 specification.

5 & 6. Previous work has shown that AA2195 had variations, through the plate thickness, with respect to texture and microstructure. Alloy AA2095 exhibited only texture variations.
through the thickness. Thus, standard inductively coupled plasma (ICP) chemical analysis was conducted on samples, in the form of shavings, which were dry milled from various locations through the thickness (t/8, t/4, t/2, 3t/4 and 7t/8) of the 12.7 mm plates. The values plotted in this figure represent the averages of triplicate analyses. The data appear well behaved and exhibit very little scatter.

The upper and lower limits the registered composition limits of AA2095 and AA2195 are shown as horizontal lines. The results of the chemical analyses suggest a significant level of Cu segregation, as indicated by the consistently low Cu content at the mid-plane (t/2) location. The least amount of Cu segregation with respect to the mid-plane was observed for AA2095, while the greatest was observed for AA2195 alloy. AA2095 has an average through-the-thickness (bulk) chemistry of 4.64wt%Cu which is outside of the registered composition specification (3.9-4.6wt%). For AA2195, the bulk chemistry is 4.04wt%Cu which falls within the specification of (3.7-4.3wt%); however the midplane chemistry is 3.58wt%Cu, which is outside of the composition specification.

An aging study was conducted at 143°C for the two X2095 alloy variants to characterize more completely the age hardening response of this alloy. Originally, the aging study was conducted up to 100 hours, as depicted by the open symbols in the figure, however, neither alloy overaged in that time frame. Results of an extended aging study (24, 72, 100, 198, 334, 408, 502, and 768 hours) are shown as filled symbols in the figure. The high Cu-AA2095 with high Cu-to-Li was stable with respect to hardness throughout the 768 hour time frame. No evidence of overaging was observed, as a hardness of 92.0 +/- 0.4 HRB was maintained from 100 to 768 hours. AA2195 with low Cu-to-Li began to overage between 100 and 200 hours, which was followed by a hardness plateau through 502 hours, before significantly overaging. The overall hardness results of the extended aging study were approximately 2 Rockwell B scale (HRB) points lower than the original study; however, incremental hardness changes with corresponding time changes for 24, 72 and 100 hours, resulted in the same relative change in hardness for the two heat treat runs.

Tensile results from the initial aging study resulted in a selection of 20 and 30 hour ages at 143°C for AA2095 and AA2195, respectively. This peak-aging "-T8" condition attempted to optimize the strength and fracture of toughness of these alloy variants. Another factor in selecting this aging condition was that the tensile properties of the two variants were quite similar from ambient to cryogenic test temperatures.

Tensile precision modulus tests were conducted on the X2095 (AA2195) alloy variant aged for 30 hours at 143°C. Specimens were machined from the mid-plane of the plate in the longitudinal orientation. The tests were conducted in accordance to ASTM E111, the Standard Test Method for Young's Modulus, Tangent Modulus, and Chord Modulus at 25°C and -185°C. An average modulus was determined from three runs per specimen at each test condition. The modulus of AA2195 (aged for 30 hours at 143°C) increased by approximately 6% from ambient to cryogenic temperatures (-185°C). The critical
alignment criteria was met for all of the tests while the coefficient of variation was twice as good as expected for aluminum alloys.

11& 12. Uniaxial tensile deformation behavior of AA2195-T8 was determined as a function of temperature (-185, -140, -125, -75, -25, 25, 107, and 135°C). Selected tests were conducted on AA2095-T8 for comparison purposes. Yield strength and ultimate strengths increased with decreasing temperature.

13& 14. The effect of temperature on the percent elongation (25.4 mm gage) is shown for both alloy variants. The percent elongation of AA2095 decreases to a minimum as temperatures decrease to around 20°C. The increase in percent elongation, about the minimum, appears to be approximately the same magnitude for both high and low temperatures. The minimum elongation for AA2195 is not as clear due to the wide scatter in data. However, the percent elongation minimum for AA2195 is shifted slightly to the left of AA2095's minimum, to approximately -25 to -125°C. AA2195 is more sensitive to changes in temperature as the percent elongation decreases more rapidly than for AA2095.

15. Work hardening exponents are observed to increase for each alloy with decreasing temperature to cryogenic levels. An absence of work hardening (as calculated between the yield strength and the ultimate strength) was observed at 135°C for both alloys, and at 107°C for AA2195 where the yield strength equaled the ultimate strength.

16. The intrinsic fracture resistance (reduction of area) of each alloy decreases with decreasing temperature to cryogenic levels, with a particularly strong sensitivity noted for the low Cu-low Li composition, AA2195-T8.

17. Tearing modulus, which is used to assess the materials resistance to stable crack growth after crack initiation was determined for both alloys as a function of temperature. Tearing modulus decreased with decreasing temperature for both alloys. The largest increase in $T_R$ occurred for AA2195 for the temperature interval from 25°C to 135°C. $T_R$ increased from 11.3 at 25°C to 24.0 at 135°C.

18. Initiation fracture toughness, $K_{IC}$, decreases slightly with decreasing temperature for AA2095. The wide scatter in the AA2195 fracture data makes it difficult to determine if a slight increase in $K_{IC}$ occurs at cryogenic temperatures. For all temperatures, AA2195 has superior fracture toughness compared to AA2095.

19. For AA2095, as yield strength increases, a decrease in $K_{IC}$ is observed. The reverse trend is suggested for AA2195.

20. The tensile and microstructural data for AA2095 and AA2195 were input into the critical plastic strain-controlled model, presented by Haynes in Project #1, to predict the...
temperature dependence of $K_{JC}$ for these alloys. The experimental $K_{JC}$ results for AA2195 and AA2095 are compared to the model predictions in this figure. The model accurately predicted the effect of temperature on $K_{JC}$ for AA2095. Due to the wide scatter in $K_{JC}$ experimental data and the smaller "constituent" particle size for AA2195, it is more difficult to assess the accuracy of the model predictions. Given the well-behaved temperature dependencies of modulus, yield strength, work hardening and intrinsic fracture resistance for this alloy, however, it is reasonable to speculate that the predicted mildly declining initiation toughness with decreasing temperature is accurate. Experiments are required to test this hypothesis.


22. Conclusions.
EFFECTS OF AGING AND TEMPERATURE ON THE DUCTILE FRACTURE OF AA2095 AND AA2195

C. L. Lach and Richard P. Gangloff
LA² ST Program Review
NASA Langley Research Center
July 17-18, 1995
OBJECTIVE

To characterize the effects of aging, temperature, and alloy composition on the deformation and fracture behavior of AA2095 and AA2195 from elevated to cryogenic temperatures.
APPROACH

2095-T3
2195-T3
{X2095-Vintage 1990}
6% Stretch
12.5 mm plate

Material Characterization:
- Chemistry (ICP)
- Optical Microscopy
- PPT Image Analysis
- Texture Analysis

Aging Study:
- Preliminary Study
  0-100 hours at 143°C
- Extended Study
  0-600 hours at 143°C

Tensile Tests: at 25°C and -185°C
- 2095 (10, 20 & 72 hours at 143°C)
- 2195 (10, 20, 30 & 72 hours at 143°C)

Fracture Toughness J-Integral Tests: at 25°C and -185°C
- 2095 (10, 20 & 72 hours at 143°C)
- 2195 (10, 20, 30 & 72 hours at 143°C)

Aged Alloys to -T8:
- 2095-T8
  (20 hours at 143°C)
- 2195-T8
  (30 hours at 143°C)

Tensile Tests: at -185°C to 135°C
- 2095-T8
- 2195-T8

Fracture Toughness J-Integral Tests: at -185°C to 135°C
- 2095-T8
- 2195-T8
## CHEMICAL COMPOSITIONS

Registered Composition limits of Weldalite™049 Alloys

<table>
<thead>
<tr>
<th>ELEMENTS</th>
<th>AA2094</th>
<th>AA2095</th>
<th>AA2195</th>
<th>Lean Variant</th>
<th>Rich Variant</th>
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<td>Cu</td>
<td>4.4-5.2</td>
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CHEMICAL ANALYSIS

Through-thickness chemistry of AA2095

Weight Percent Copper, wt%

Thickness, t

4.60
4.75
5.00

3.90
3.75
3.50

t/8  t/4  t/2  3t/4  7t/8  BULK
CHEMICAL ANALYSIS

Through-thickness chemistry of AA2195

Weight Percent Copper, wt%
MECHANICAL PROPERTIES

Age hardening response of AA2095 and AA2195 at 143°C

Hardness, HRB

Time, hours
MECHANICAL PROPERTIES

Constant aging condition selected for -T8 property characterization

<table>
<thead>
<tr>
<th>Alloy</th>
<th>143°C Age, hours</th>
<th>Temp., °C</th>
<th>YS, MPa</th>
<th>ULT, MPa</th>
<th>% elongation 25.4mm gage</th>
<th>Strain hardening exponent, $\eta$</th>
<th>E, GPa</th>
<th>RA, %</th>
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<td>AA2195-T8</td>
<td>30</td>
<td>25</td>
<td>577</td>
<td>602</td>
<td>11.1</td>
<td>0.046</td>
<td>72.4</td>
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<td>25</td>
<td>572</td>
<td>600</td>
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<td>AA2195-T8</td>
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<td>745</td>
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<td>0.067</td>
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</table>
MECHANICAL PROPERTIES

Precision modulus of AA2195-T8 at 25°C

Young's Modulus, GPa

Test Number

Run 1  Run 2  Run 3
MECHANICAL PROPERTIES

Precision modulus of AA2195-T8 at -185°C

Young's Modulus, GPa

Run 1  Run 2  Run 3

Test Number

81.00
80.75
80.50
80.25
80.00
80.45
80.30
80.38

- 4H
- 4I
MECHANICAL PROPERTIES

Effect of temperature on ultimate strength of AA2095-T8 and AA2195-T8

Ultimate Strength, MPa

Temperature, °C
MECHANICAL PROPERTIES

Effect of temperature on yield strength of AA2095-T8 and AA2195-T8

Yield Strength, MPa

Temperature, °C

-200  -100   0      100     200

AA2095-T8
AA2195-T8
MECHANICAL PROPERTIES

Effect of temperature on % elongation of AA2095-T8

% Elongation (25.4 mm gage)

Temperature, °C
MECHANICAL PROPERTIES

Effect of temperature on % elongation of AA2195-T8

% Elongation (25.4 mm gage)

Temperature, °C
MECHANICAL PROPERTIES

Effect of temperature on strain hardening exponent, $\eta$, of AA2095-T8 and AA2195-T8

Strain hardening exponent, $\eta$

Temperature, °C
MECHANICAL PROPERTIES

Effect of temperature on reduction of area
of AA2095-T8 and AA2195-T8

% Reduction
of Area

Temperature, °C
MECHANICAL PROPERTIES

Effect of temperature on tearing modulus of AA2095-T8 and AA2195-T8

Tearing Modulus ($T_R$)

Temperature, °C

AA2095

AA2195
MECHANICAL PROPERTIES

Effect of temperature on fracture toughness of AA2095-T8 and AA2195-T8

Fracture Toughness, \( K_{\text{jic}} \), MPa·m\(^{1/2}\)

Temperature, °C
MECHANICAL PROPERTIES

Effect of chemistry and temperature on fracture toughness of AA2095-T8 and AA2195-T8

Fracture Toughness, $K_{\text{jic}}$, MPa$\cdot$m$^{1/2}$

Yield Strength, MPa
Assessment of the fracture toughness $K_{J_{CI}}$ for AA2195-T8 and AA2095-T8 3.8 mm sheets. The graph represents the measured and predicted values of $K_{J_{CI}}$ as a function of temperature. The least squares regression of predictions is shown for both materials.

- **AA2195-T8, 3.8 mm Sheet**
  - Measured ▲
  - Predicted ($r=7, l^* = 29.6 \mu m$) ▼

- **AA2095-T8, 3.8 mm Sheet**
  - Measured ◇
  - Predicted ($r=7, l^* = 21.9 \mu m$) □

Temperature (°C) range from -200°C to 150°C.
CONCLUSIONS

- A significant level of Cu segregation through-the-thickness occurred for both alloys.

- AA2095 did not overage at 143°C in 768 hours while AA2195 began overaging between 100-200 hours.

- The modulus of AA2195-T8 increased by 6% from ambient to cryogenic (-185°C) temperatures.

- Tensile and microstructural data were sufficient to predict the temperature dependence of the plane strain initiation fracture toughness of these alloys using a critical plastic strain-controlled model.
FUTURE WORK

- Characterize the effect of temperature on the microvoid-based fracture process.

- Predict the temperature dependence of plane strain initiation fracture toughness, as a function of aging at 143°C, for AA2095 and AA2195.
Project Objective:

The objectives of this research project are to define the conditions sufficient to cause cracking in Al-Li-Cu alloys, to identify microstructural features which may contribute to anodic dissolution based cracking in PA tempers; to describe the critical solution chemistry changes associated with cracking under alternate immersion conditions in chloride solution; to develop an in-situ technique for quantifying EC propagation rates; and to investigate the influence of material temper, sample orientation, stress intensity level, applied external electrochemical potential and bulk aqueous environment on the environmental cracking of Al-Li-Cu alloys.

Current Status:

The experimental phase of this research project is approaching completion and efforts are beginning to focus on data analysis, compilation of a dissertation and publication of findings.

Recent Findings:

Progress this reporting period has been concentrated in the areas of solution chemistry analysis and crack propagation rate studies.

Solution analysis

Initial experiments in this area were intended to simulate the chemistry which develops in an isolated pit during the dry cycle of alternate immersion testing in aqueous NaCl. Exposure of large material surface areas to low solution volumes resulted in the solutions with alkaline pH's (8-11) and significant dissolved lithium concentrations. This data is in agreement with the literature for research performed on other Al-Li-Cu alloys. However, the extrapolation of these data to propagating environmental cracks is not suitable due to the extreme depth to height ratios of cracks and the subsequent separation of anodic and cathodic reactions.
Recently samples having more restricted geometries have been exposed to alternate immersion in NaCl solution with real-time monitoring of pH as well as post-test pH determination via freezing and sampling with broad range pH paper. Additionally, post test analysis has been attempted on DCB specimens of Al-Li-Cu alloys. All data indicates that deep cracks/crevices are acidic in nature with a pH in the range of 2 to 4.

Spatially resolved solution chemistry analysis of DCB specimens has been attempted using a frozen ball sampling technique. In this procedure 12 µl droplets are deposited on a frozen DCB fracture surface after the sample has undergone alternate immersion exposure. The droplets freeze into the ice layer of the specimen thus sampling the solution chemistry. The droplets are removed, melted and analyzed using capillary electrophoresis. Although the sampling efficiency is sufficient to allow identification of Al and Li ions the technique lacks the reproducibility required to gain spatial knowledge of ion concentrations.

A new technique has been developed which relies on physical isolation of a "patch" of frozen solution followed by sampling. Initial results indicate that this system may have sufficient reproducibility and sampling efficiency to allow new insights into the occluded chemistry which develops during alternate immersion testing of Al-Li-Cu alloys.

**Environmental Cracking**

The technique for growing and monitoring in-situ environmental cracks in Al-Li-Cu alloys has been refined and used to investigate the influence of several experimental variables on crack rates and fracture surface morphology.

The base-line experiment which is used as a control consists of SL-PA 2095 exposed to 0.1M NaCl + 0.1M Na₂CrO₄ solution, polarized to -0.547 V_{SCE} and stressed such that the crack tip stress intensity falls between 10 and 20 MPa·m⁰.². Under these conditions the crack growth rates are approximately 10⁻⁵mm/sec. The environmental fracture surfaces have significant tortuosity, evidence of ductile features and are covered with extensive corrosion products.

Ramping the applied potential in the cathodic direction once the test has initiated does not result in cessation of cracking until after net cathodic currents have been measured. Smooth specimens exhibit a critical EAC potential below which cracking will not occur. This recent data suggests that the macroscopic experimental conditions for crack propagation are different than
for initiation. It is likely that IR drop down the crack has caused a loss of “communication” between the potentiostat and crack front.

Low stress intensity levels have been investigated in an attempt to define a $K_{\text{IEAC}}$ for the system being studied. However, to this point no lower bound of applied stress intensity has been clearly defined at which environmental cracks will not propagate. Low stress intensity levels ($K_{\text{APPL}} < 6 \text{ MPa-m}^{1/2}$) have resulted in a change in fracture morphology from the control experiment. Crack fracture surfaces appear flat with loss of ductile features. This observation has not been interpreted at this time.

Material temper has been shown to have an enormous effect on propagation rate. Aging 2095 from 4 to 20 hours at 160°C results in a 15X change in crack propagation rates. Additionally the fracture morphology for the UA samples is relatively featureless, is sparingly decorated with corrosion products and has been shown to have matching features on opposite sample halves. These observations indicate the possibility of an embrittlement mechanism dominating the cracking of the UA materials.

Testing in a bulk environment of NaCl without the presence of an inhibiting specie does not result in environmental cracking for an applied potential of $-0.7 \text{ V}_{\text{SCE}}$ (anodic to $E_{\text{OC}}$). Fractography indicates that overload failure ensues at the end of the fatigue precrack for a large portion of the central crack front. However, along the edges of the sample extensive attack is evident stemming from the end of the fatigue crack. Even though this attack was resolvable via DCPD measurements during the test, it appears to be gross pitting caused by polarization near the pitting potential.

**Future Work:**

During the next month the experimental stage of this project will be completed. Emphasis will be on calibration and implementation of a technique for gaining improved knowledge on the solution chemistries associated with propagating environmental cracks in specimens alternately exposed to NaCl solution and ambient air. The following months (9/95 through 12/95) will be used for completion of a dissertation and publication of findings.
Explanatory Text for Viewgraphs:

1. Title slide.

2. Objectives.

3. Microstructural features considered for anodic dissolution based cracking of PA Al-Li-Cu alloys. TEM micrograph shows boundary in PA 2095 decorated with T1 particles. Lower graph is data on Cu depletion taken from Kumai. Our model for a boundary is a high density of T1 phase surrounded by a region of diminished copper content.

4. This slide lists the materials used to simulate the electrochemical behavior of relevant boundary phases. The T1 phase was cast as a bulk ingot by Rudy Bucheit (Sandia Labs). Al-Cu binaries are used to map out the electrochemical behavior of Al as a function of Cu content. The experimental techniques used to investigate the electrochemical behavior are also listed. In general, attempts were made to determine electrochemical potentials which resulted in a change in material corrosion current. These values were then compared to potentials which result in a change in material environmental cracking response for the same environment.

5. These are the relevant data in a NaCl/Na2CrO4 environment for the simulated T1 phase (upper left), Al with varying Cu content (upper right), TTF of PA 2095 as a function of applied potential (lower left), and critical potentials associated with an abrupt change in TTF for various Al-Cu-X alloys (lower right).

6. An overview of occluded chemistry analysis is presented.

7. A list of the techniques used to improve understanding of localized chemistry during propagation of environmental cracks.

8. Experimental set-up used to simulate a long propagating environmental crack and resulting data from Al exposure to NaCl environment.

9. One of the techniques used in an attempt to gain spatial knowledge of crack chemistries associated with DCB specimens under Al conditions.

10. An example of the data obtained using the technique summarized in slide #9. Although sampling efficiency is adequate, reproducibility remains a problem with this technique.

11. Improved device for obtaining local solution chemistry samples. This rig relies on physical isolation of solution followed by sampling and analysis. Initial testing indicates a high sampling efficiency and reasonable reproducibility.

12. Summary of solution analysis findings.
13. Overview of approach and variables examined in environmental cracking study.

14. Control experiment for environmental cracking study. Conditions for this test were derived from electrochemical characterization of model boundary features and results of TTF testing of smooth bar specimens under constant immersion conditions.

15. Results of testing in NaCl without an inhibiting specie present. Although crack extension is indicated by DCPD data, SEM fractography indicates that gross pitting and not EAC caused the change in the potential signal.

16-17. Effect of changing the applied external potential on the crack growth rate and resulting environmental cracking fractography. Although the potential is lowered cathodic to the material critical EAC potential the crack continues to propagate. Optical microscopy and SEM indicate that this crack developed a very deep front. This is most likely due to the loss of external electrochemical driving force. It is possible that the crack sides may have even been cathodically protected to some extent. As the overpotential was shifted in the cathodic direction the fracture surface morphology changed from an appearance consistent with the control experiment (slide#14) to a relatively featureless surface. A mechanistic interpretation of this phenomena has not been developed at this time.

18. This slide shows the crack length vs. time data typical of a test carried out at an elevated stress intensity (K > 24 MPa-m$^{1/2}$). Crack bursting events are indicated by DCPD as well as corrosion current data. These bursts may be due to either an embrittlement phenomena or ligament failure lagging the crack front. It is interesting to note that at these stress intensities the crack front is under plain stress conditions.

19-20. The effect of low stress intensity (K< 6 MPa-m$^{1/2}$) on environmental cracking is shown in these two slides. Experimentation at low K levels was carried out in order to determine a $K_{IEAC}$ value for these experimental conditions; however, no limit was conclusively determined below which a crack would not propagate. Similar to the case for reduced electrochemical driving force, reduced stress intensity results in a change of fracture morphology from that associated with the control (slide#14) to a relatively featureless surface.

21-23. These slides examine the cracking behavior of 2095 and 2090 as a function of thermomechanical processing. A strong correlation exists between crack growth rates and material temper. UA materials have been observed to crack at rates between 15 and 1000 times faster than PA materials. Additionally, differences in fracture morphology suggests that cracking of PA materials in accompanied by extensive materials dissolution while cracking of UA materials appears to be due to brittle failure.

24-25. A UA 2095 sample was tested in the LT orientation in order to determine if the high crack velocities associated with this temper were a high angle boundary phenomenon. Crack growth rate data indicates several orders of magnitude change in velocity with changing orientation. Post test failure of the specimen resulted in cracking along a direction
perpendicular to the fatigue crack plane. Fractography suggests the embrittlement of high angle boundaries.

26. This slide shows the misorientation of material along opposite sides of an environmental crack grown under control conditions (slide#14). Kikuchi patterns were generated and indexed in order to determine misorientations. Cracking under these conditions follows high angle grain boundaries.

27. General summary and conclusions of presented research.
A Study of Environmentally Assisted Cracking in Al-Li-Cu Alloys 2090 and 2095

F.D. Wall
G.E. Stoner

Department of Materials Science and Engineering
University of Virginia
Charlottesville, Virginia 22903

NASA - LaRC Contact : Marsha Domac

Additional Support:
Reynolds Metals Corporation
Waters Corporation
Objectives

Determine if the grain boundaries can provide a pathway for anodic dissolution based environmental cracking. What microstructural feature defines the active path?

Analyze the occluded chemistries that develop in an isolated pit and in a propagating environmental crack. Determine conditions (pH, ions present) which are necessary for crack initiation and crack propagation.

Develop a technique for growing and monitoring cracks in Al-Li-Cu alloys under constant immersion conditions. Use the technique to examine the effect of $E_{APPL}$, $K_{APPL}$, material temper and bulk aqueous environment on crack propagation rates.

Knowledge to be gained by meeting objectives

Conditions sufficient to cause in-situ environmental cracking.

Environmental factors associated with cracking during Al exposure in aqueous NaCl.

Effects of changing macroscopic variables on EC of alloy 2095.
Cu-depletion across grain boundary in Al-Li-Cu 2090
(from Kumai, Corrosion Science, V45, No. 4, p 300)
Electrochemical Behavior of Model GB Features and EAC Behavior of Tempered Alloys

Materials used to model GB features:

- $T_1$ phase ($Al_2CuLi$)
- Al with varying Cu content

Techniques used to assess behavior of GB features:

- Cast $T_1$ : potentiodynamic polarization $\rightarrow E_{BR} (E_{PIT})$
- Al-Cu : scratching electrode experiments $\rightarrow E_{RP}$

Technique for assessing EAC Behavior of Tempered Al-Li-Cu alloys:

- Constant extension / scratching electrode experiment $\rightarrow E_{CRIT-EAC}$
Occluded Chemistry Analysis

Stressed samples of Al-Li-Cu alloys generally do not fail during constant immersion in aqueous chloride environments but rapidly fail during alternate immersion testing in the same environment.

What happens to the pit chemistry when the sample is removed from the bulk aqueous environment?

The pH and ionic makeup of solution within an isolated pit has been experimentally modeled by exposure of large areas of material surface to small volumes of chloride solution.

- **pH microelectrode** → alkaline pH : 9-11
- **capillary electrophoresis** → significant [Li⁺] : mMol

Hypothesis

Under constant immersion conditions the pit chemistry is too aggressive and a sharp crack cannot be initiated/maintained.

Removal of a pitted sample to ambient air results in the development of an alkaline pH, absorption of CO₂ and the subsequent formation of a specie causing passivation of crack walls.

Next Question

Is this chemistry maintained as the crack propagates away from the initiation site? What pH and solution makeup are associated with a propagating EAC crack?
**Techniques for examining solution chemistries associated with crack propagation**

**Alternate immersion in aqueous NaCl:**
- pre-cracked DCB specimens
- model crevice

**pH measurements**
- micro-electrode mounted in center of model crevice (real-time)
- pH paper used to investigate frozen surfaces (post exposure)

**Solution analysis**
- Isolation of solution from DCB samples followed by speciation and quantification using capillary electrophoresis
Solution pH measured under alternate immersion conditions in aqueous NaCl for restricted geometry intended to model a propagating environmental crack.
Methodology:

1. Freeze sample in liquid nitrogen.
2. Remove from LN$_2$ and load to failure.
3. Place sample in tub of LN$_2$ to maintain temperature and provide N$_2$ blanket.
4. Deposit 12ul droplets of sampling solution onto specimen fracture surface.
5. Place frozen samples into vials to thaw.
6. Analyze samples using capillary electrophoresis.
Analysis of solution removed from 2095 DCB specimen following alternate immersion exposure in 0.1M NaCl.
Technique of physical isolation for obtaining spacially resolved samples of solution of known volume

Sampling efficiency of solution isolation rig for 0.05 M K₂MoO₄ +0.05 M MgSO₄ extracted from an artificial 0.060 mm crevice:

<table>
<thead>
<tr>
<th>trial#</th>
<th>Efficiency</th>
</tr>
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<tbody>
<tr>
<td>1</td>
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</tr>
<tr>
<td>2</td>
<td>60%</td>
</tr>
<tr>
<td>3</td>
<td>73%</td>
</tr>
<tr>
<td>3+4</td>
<td>95%</td>
</tr>
</tbody>
</table>
Summary of solution analysis

Model experiments indicate that the solution in an isolated pit is likely to develop an alkaline pH and a significant Li\(^+\) concentration during the dry cycle of an Al experiment in NaCl solutions. This environment may be critical in the initiation of environmental cracks.

Model crevices in Al exposures develop an acidic pH which remains acidic independent of wet or dry cycle. This condition is representative of a long environmental crack and suggests that an acidic pH dominates crack tip chemistry during propagation.

Techniques such as the frozen ball method and the solution isolation rig are promising for broadening knowledge of spatially resolved crack tip chemistries.
Examination of Variables affecting Environmental Cracking Behavior

Approach

Use knowledge of boundary feature electrochemistry and TTF behavior of statically loaded smooth bar specimens to select environmental and electrochemical conditions likely to result in crack initiation under constant immersion conditions.

Change variables to deviate from this base-line condition and examine effects on EC behavior in terms of crack propagation rates (quantitative) and crack path fractography (qualitative).

Variables examined

- **Bulk solution chemistry**: NaCl + Na₂CrO₄ vs. NaCl
- **Applied overpotential**: constant vs. ramp towards cathodic
- **Stress intensity**: low, intermediate, high (2-5, 10-20, 24-26 MPa-m¹/²)
- **Material temper**: UA vs. PA
- **Crack path**: high or low angle, SL vs. LT
**EC Behavior of PA 2095 Under Base-Line Conditions**

Temper: PA 2095 (SHT+CWQ+6%STR+30hrs@143°C)
Sample orientation: SL
Sample geometry: WOL ($a_0 = 9.11\text{mm}$; $b = 3.175\text{mm}$; $w = 36.58\text{mm}$)
Bulk environment: 0.1M NaCl + 0.1M Na$_2$CrO$_4$
Applied potential: $E_{\text{APPL,AVE}} = -0.547 \ \text{V}_{\text{SCE}}$

**Target stress intensity**: 18 MPa-m$^{1/2}$

Average crack growth rate = $10^{-5}$ mm/sec

Typical morphology for EC region under listed conditions.
Effect of testing in aqueous chloride without the presence of an inhibiting specie

Temper: 2095 (SHT+CWQ+6.5%STR+20hrs@143°C)
Sample orientation: SL
Sample geometry: WOL (a₀ = 9.11mm; b = 3.175mm; w = 36.58mm)
Bulk environment: 0.6 M NaCl (aerated)
Applied potential: E_{APPL,AVE} = -0.700 V_{SCE}
Target stress intensity: 16.5 MPa-m^{1/2}
Effect of overpotential on a propagating environmental crack

PA 2095 loaded to 18 MPa-m^{1/2} in NaCl/Na_2CrO_4

\[ E_{\text{APPL}} = -0.547 \text{ V}_{\text{SCE}} \]

\[ E_{\text{APPL}} \text{ ramped from -0.547 to -0.790 V}_{\text{SCE}} \]
Change in fractography associated with cathodic ramp of applied potential

Post test fracture
- Ductile features
- Similar to air fracture
Effect of elevated applied stress intensity on cracking behavior: \( 2R_p^* > 0.12\ b \)

**Temper**: PA 2095 (SHT+CWQ+6%STR+30hrs@143°C)

**Sample orientation**: SL

**Sample geometry**: WOL \((a_o = 9.11\ mm; b = 3.175\ mm; w = 36.58\ mm)\)

**Bulk environment**: 0.1M NaCl + 0.1M Na\(_2\)CrO\(_4\) (aerated)

**Applied potential**: \(E_{\text{APPL,AVE}} = -0.547\ \text{V}_{\text{SCE}}\)

**Target stress intensity**: 24 MPa-m\(^{1/2}\)

**Average crack velocity**: \(2 \times 10^{-5}\ \text{mm/sec}\)

Crack bursting **not** observed for samples loaded between 4 and 20 MPa-m\(^{1/2}\). Bursting events can be accounted for by either **embrittlement** at the crack tip causing local resistance to crack growth to fall below applied mechanical driving force, or by **failure of ligaments** lagging the average crack front.
Effect of reduced stress intensity on environmental cracking

Temper: PA 2095 (SHT+CWQ+6%STR+30hrs@143°C)
Sample orientation: SL
Sample geometry: WOL (a₀ = 9.11mm; b = 3.175mm; w = 36.58mm)
Bulk environment: 0.1M NaCl + 0.1M Na₂CrO₄ (aerated)
Applied potential: E_{APPL,AVE} = -0.547 V_{SCE}
Target stress intensity: 4 MPa-m¹/²

average da/dt
7x10⁻⁶ mm/sec

1 2 3

[1] 1x10⁻⁵
[2] 1x10⁻⁶
[3] 5x10⁻⁶
Effect of low $K$ on crack fractography
Effect of thermomechanical treatment on environmental cracking propagation rates

Sample orientation : SL
Sample geometry : WOL (a₀ = 9.11mm; b = 3.175mm; w = 36.58mm)
Bulk environment : 0.1M NaCl + 0.1M Na₂CrO₄ (aerated)
Applied Potential : \( E_{\text{APPL}} < E_{\text{CRIT-EAC}} \) (-0.515...-0.547 V \text{SCE})

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Stretch</th>
<th>Aging [t(hrs)@T(°C)]</th>
<th>( da/dt ) (mm/sec)</th>
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<tbody>
<tr>
<td>2095</td>
<td>6%</td>
<td>20 @ 143</td>
<td>1x10⁻⁵</td>
</tr>
<tr>
<td>2095</td>
<td>6.5%</td>
<td>30 @ 143</td>
<td>1x10⁻⁵</td>
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<tr>
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<td>0%</td>
<td>5 @ 143</td>
<td>5x10⁻⁴</td>
</tr>
<tr>
<td>2095</td>
<td>0%</td>
<td>14 @ 143</td>
<td>8x10⁻⁴</td>
</tr>
<tr>
<td>2095</td>
<td>0%</td>
<td>25 @ 143</td>
<td>3x10⁻⁴</td>
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<tr>
<td>2090</td>
<td>3.5%</td>
<td>14 @ 160</td>
<td>1x10⁻⁵</td>
</tr>
<tr>
<td>2090</td>
<td>0%</td>
<td>3 @ 160</td>
<td>2x10⁻³</td>
</tr>
</tbody>
</table>

Many of the 0% stretch UA alloys exhibited growth rates greater than 1x10⁻³ mm/sec for significant periods. The listed numbers indicate the longest sustained continuous growth rates.
Effect of aging time on crack propagation rates

2095 (20 hrs @ 160°C)

2095 (4 hrs @ 160°C)
Comparison of EC behavior for anodically polarized 2095-D (UA) and 2095-B (PA) in NaCl/Na$_2$CrO$_4$

2095-B-PA: Copious corrosion products, heavily attacked fracture surface.

2095-D-UA: Less corrosion evident compared to PA, matching fracture surfaces indicative of brittle failure.
Effect of sample orientation on EC behavior of UA 2095 (SHT+CWQ+0%STR+5 hrs@ 143°C)

Temper UA 2095 (SHT+CWQ+0%STR+5 hrs@143°C)
Sample orientation: SL / LT
Sample geometry: WOL (a_o = 9.11 mm; b = 3.175 mm; w = 36.58 mm)
Bulk environment: 0.1M NaCl + 0.1M Na_2CrO_4 (aerated)
Applied potential: E_{APPL,AVE} = -0.520 V_{SCE}
Effect of sample orientation on EC behavior of UA 2095 (SHT+CWQ+0%STR+5 hrs@ 143°C)
Crack path for PA 2095
AD based cracking along boundaries in Al-Li-Cu alloys
* The $T_1$ phase and Cu depleted Al may contribute to defining an active pathway.
* The electrochemical behavior of Cu depleted Al appears to define the critical EAC potentials under the conditions investigated.

Occluded solution chemistry analysis
* Models for isolated pits suggest an alkaline environment and passivating specie are necessary for EC initiation under Al conditions in aqueous NaCl.
* Models for deep cracks indicate the development of an acidic pH during crack propagation.
* Techniques for spacially resolving crack tip chemistries are being developed.

Crack propagation studies
* Conditions have been defined under which environmental cracks can be studied in-situ for Al-Li-Cu alloys in constant immersion.
* Cracking has not been observed under constant immersion in NaCl solution without the presence of an inhibiting specie.
* Propagating environmental cracks did not arrest when the externally applied potential was reduced below $E_{\text{CRIT-EAC}}$.
* Reduction of crack driving forces such as applied potential and stress intensity resulted in a change in crack morphology.
* EC behavior is a strong function of material temper. UA tempers of 2090 and 2095 exhibit accelerated growth rates and flat, matching fracture surfaces with little corrosion product build-up.
* Cracking occurs along high angle grain boundaries for the conditions investigated.
Research Objectives:

The goal of this project was to develop a fundamental understanding of the effects of internal lattice and trapped hydrogen on the mechanical properties of selected Al-Li-Cu-X alloys. The program focused on unrecrystallized alloy 2090, an Al-Li-Cu-Zr alloy within the specified compositional range of AA2090 which has been recrystallized to varying grain sizes, and complimentary model alloys. We proposed to: (a) distinguish HEAC from aqueous dissolution controlled EAC by using a novel precharging procedure, (b) correlate hydrogen induced EAC with mobile and trapped hydrogen concentrations, (c) identify significant trap sites and hydride phases (if any) through utilization of model alloys and phases. Following identification of relevant hydrogen trap sites, the emphasis during the last 2 quarters has centered on comparing hydrogen concentrations produced from the novel precharging procedure to hydrogen levels produced during conventional aqueous alternate immersion exposure and straining electrode testing. Additionally, the role of dynamic straining in promoting hydrogen uptake and repartitioning was investigated during the last reporting period. This program ended in the spring of 1995

Current Status

This project has been completed and a Ph.D. degree has been awarded to Mr. Steven W. Smith. Dr. Smith has accepted an NRC Post-Doctoral Research Position with R. S. Piascik at the NASA- Langley Research Center.

Project Findings

A novel method was successfully utilized to unambiguously separate the competitive roles of anodic dissolution and absorbed hydrogen in an Al-Li-Cu-Zr alloy (AA 2090) that is susceptible to environmentally assisted cracking. Specifically, a nickel-coating procedure was utilized to cathodically pre-charge alloys with hydrogen without damage due to corrosion. Pre-
charged samples were tested in air, exploiting the strong hydrogen permeation barrier of the native Al oxide film to retain internal hydrogen. Aqueous stress corrosion was thereby completely decoupled from hydrogen embrittlement phenomena. The decoupled effects of internal hydrogen on environmental fracture were evaluated by studying commercial unrecrystallized AA2090 sheet as well as a model recrystallized Al-Li-Cu-Zr alloy with a composition within the specified range of AA 2090. The model recrystallized alloy was fabricated in three different grain sizes and tested in the underaged (UA) and peak aged (PA) tempers. Unrecrystallized AA2090 in the LT and TL orientations was studied in the T3, UA, and PA tempers. The anisotropic nature of the AA2090 microstructure and the range of recrystallized grain sizes relative to expected plastic zone dimensions were exploited in an attempt to examine hydrogen embrittlement, if operative, on either (a) sub-grain boundaries, (b) transgranular slip planes, or (c) high angle grain boundaries. Continuous extension rate testing (CERT) of smooth tensile specimens and J-integral resistance curve testing of pre-cracked specimens at slow actuator displacement rates were employed to assess the effects of internally dissolved and trapped hydrogen on ductility and fracture initiation toughness.

Hydrogen operated in two primary ways to induce embrittlement under the limited conditions investigated. Hydrogen promoted high angle grain boundary crack initiation when such boundaries presented a favorably oriented hydrogen assisted cracking path and stress concentration was sufficient. This occurred in J-integral fracture toughness testing of the fine grain recrystallized material (UA and PA) and to a lessor extent in commercial AA2090 in the TL orientation (UA and PA). Hydrogen also promoted the localization of shear deformation into less numerous, narrower bands. This was observed in CERT test results on unrecrystallized AA 2090 particularly in the T3 and UA tempers. A decrease in $K_I$ for unrecrystallized AA 2090 was also observed for material in the T3 condition that was linked to the presence of finer, more numerous microvoids on transgranular fracture initiation surfaces. In no cases was intersubgranular cracking observed even though this cracking path would have been favorable in both the LT and TL orientations of unrecrystallized AA2090.

Embrittlement trends were correlated with hydrogen-metal defect interactions using thermal desorption spectroscopy (TDS). Trapping analysis was conducted after performing the same hydrogen charging procedure as utilized for CERT and fracture toughness testing. Six
metallurgical trapping states have been identified. Lower binding energy hydrogen trap sites included solid solution lithium and δ'. Higher energy states were correlated with dislocations, T₁, T₂ or T₈ particles, and high angle boundaries. Trapping at subgrain boundaries was not observed when comparing unrecrystallized to recrystallized materials.

The tendency for high angle grain boundaries (containing T₁, T₂ and/or T₈ particles) to provide a favorable hydrogen assisted cracking path correlated with the observation that high angle boundaries and T particle are strong hydrogen trap sites. Hydrogen trapping at dislocations is consistent with the notion of hydrogen enhanced slip localization. This effect is due to trapped hydrogen as a dislocation atmosphere lowering the interaction energy between dislocations; thereby lowering the shear stress required to sweep dislocations through a lattice containing other dislocations. However, modelling to date on dislocation interaction energies has only dealt with a limited number of dislocations. It is unclear whether such results would apply to a more complicated situation involving heavily deformed structures containing planar slip bands as well as shear deformation bands across multiple high angle boundaries in Al-Li-Cu alloys. The fact that subgrain boundaries were not an observed cracking path can be rationalized in the context of a low energy dislocation structure which does not trap hydrogen. However, the presence of hydrogen trapping T₁ particles on such boundaries would seem to make them an eligible cracking path. It could be argued that the decohesion forces required are significantly higher at subgrain boundaries compared to high angle boundaries.

TDS analysis has also produced direct evidence of significant hydrogen absorption during aqueous alternate immersion exposure testing and either enhanced uptake or transport of hydrogen via dislocations during straining electrode testing in aqueous full immersion or moist air. Enhanced hydrogen absorption occurs during conventional stress corrosion cracking testing of Al-Li-Cu-Zr alloys compared to the nickel coated precharging procedure. Moreover, hydrogen transport via dislocations was implicated as the cause of the internal redistribution of hydrogen from sites of lower binding energy to sites of higher binding energy.

**Future Plans**

A follow-on project emphasizing localized corrosion, and the transition to stress corrosion cracking initiation and SCC propagation will investigate two advanced aerospace Al-Li-Cu
alloys. The alloys of interest are AA 2096 and C155 which have nearly identical Cu/Li ratios. One alloy contains Ag as a precipitation promotor while the other contains Mn. These compositions present an interesting scientific opportunity in terms of linking composition and microstructure to localized corrosion, SCC and hydrogen trapping. (See additional information below for objective, approach and expected payoffs).

List of Presentation Overheads

1. Title.

2. Proposed EAC mechanisms for Al-Li-Cu-Zr alloys existing in the literature.

3. Project objectives.

4. Procedures utilized for introducing pre-dissolved hydrogen in Al-Li-Cu-Zr Alloys.

5. Absorbed hydrogen concentrations measured for AA2090 after several testing conditions. SET = straining electrode test, AI = alternate immersion.

6. Mechanical properties data for the unrecrystallized AA2090 and the three variants of the recrystallized Al-Li-Cu-Zr alloy used for mechanical testing.

7. Continuous extension rate test results for smooth AA2090 specimen tested with absorbed hydrogen. All testing was performed at a strain rate of $2.5 \times 10^{-6} \text{ sec}^{-1}$.

8. J-integral results indicating the stress intensity at initiation for fatigue precracked AA2090 tested with absorbed hydrogen using the nickel coated charging procedure.

9. J-integral results indicating the stress intensity at initiation for fatigue precracked recrystallized Al-Li-Cu-Zr tested with absorbed hydrogen using the nickel coated charging procedure.

10. Summary of findings from mechanical testing of Al-Li-Cu-Zr alloys.

11. Relevant questions to be investigated developed from mechanical testing.


13. Hydrogen desorption rate vs. time data for an Al-3% Li binary alloy tested in the solution heat treated (SHT), SHT + 5 hrs. @ 160°C, and SHT + 25 hrs. @ 160°C tempers.
14. Hydrogen partitioning data for AA2090 and the Al-3% Li binary as a function of aging condition. This plot shows that a lower fraction of the total internal hydrogen concentration is trapped at Li in solid solution (peaks 1 and 2) with aging in accordance with the expectation that Li is depleted from solid solution with aging. Conversely, the fraction of hydrogen partitioned at T particles (peaks 5 and 6) and high angle boundaries is increased with aging.

15. Hydrogen desorption spectra and hydrogen partitioning data for AA2090-UA. Data identified as being prestrained 3% represents specimens that were strained at a constant cross head displacement of $2 \times 10^6$ sec$^{-1}$ to a total strain of 3% before being charged. An enhancement of hydrogen trapped at dislocations results.

16. Hydrogen desorption spectra and hydrogen partitioning data for a fine and large grained recrystallized Al-Li-Cu-Zr alloy. A lower fraction of hydrogen is observed on high angle boundary and T sites (peaks 5 and 6) in accordance with a lower density of grain boundary sites in the coarse grained material.

17. Summary of the trap site identification methodologies.

18. Hydrogen desorption spectra and hydrogen partitioning data for AA2090. Data identified as being strained to 3% represents a specimen that was cathodically charged then strained at a constant cross head displacement rate of $2 \times 10^6$ sec$^{-1}$ to a total strain of 3%. A fixed internal amount of hydrogen exists and some of it is shown to repartition from δ' sites (peak 3) to dislocations (peak 4).

19. Hydrogen desorption spectra for AA2090 specimens cathodically charged under a straining electrode test. The specimens were preloaded to 75% $\sigma_{ys}$ and strained at a constant cross head displacement rate of $2 \times 10^6$ sec$^{-1}$ while being polarized at -2 V$_{SCE}$ in a pH 1 HCl solution. An increase in the amount of hydrogen at dislocation traps is observed indicative of enhance hydrogen absorption during dynamic straining as well as dynamic trap site formation during straining.

20. Hydrogen desorption "difference" spectra for AA2090 specimens following fracture toughness testing. The desorption rate data from a sample cut far from the plastic zone was subtracted from the spectra of a specimen containing the fracture process zone. The results indicated that dynamic strain enhances hydrogen absorption and trapping at dislocations even in moist lab air.


22. Conclusions.

23. Conclusions (cont.)

HYDROGEN INTERACTIONS AND THEIR 
CORRELATION TO THE HYDROGEN 
EMBRITTLEMENT SUSCEPTIBILITY OF 
Al-Li-Cu-Zr ALLOYS

Stephen W. Smith 
and
John R. Scully

University of Virginia 
Department of Materials Science and Engineering 
Center for Electrochemical Sciences and Engineering 
Charlottesville, VA 22903

Sponsors: 
NASA Langley Research Center 
Virginia Center for Innovative Technology 
Alcoa Technical Center
Proposed EAC Mechanisms for Al-Li-Cu-Zr Alloys

- **Aqueous dissolution**
  - Preferential $T_1$ dissolution at boundaries [Buis 92, Buchheit 90, Colvin 86, Bavarian 89]
    - $T_1$ has been shown to be electrochemically active with respect to the matrix.
    - $T_1$ does not provide a continuous active path for dissolution.
    - $T_1$ does not precipitate exclusively at high angle grain boundaries.
    - $T_1$ is just as prevalent, if not more so, along subgrains and dislocations.

- **Hydrogen Embrittlement**
  - Pre-dissolved hydrogen has been shown to affect IG cracking [Shin 89, Shin 90]

  - Pre-dissolved hydrogen has been shown to affect TGS [Meletis 89]

  Hydrogen effects have not been adequately decoupled from AD. Evidence for hydrogen interactions are circumstantial.

- A hydride phase ($\text{AlLiH}_4$) has been observed during concurrent straining and cathodic charging [Balasubramaniam 91, 91a].
  - The microstructure used to promote the hydride formation is not characteristic of a commercial microstructure.
Objectives

• Distinguish hydrogen induced EAC from aqueous dissolution controlled EAC
  • Mechanical Testing of pre-charged specimens
    Continuous Extension Rate Testing (CERT)
    J-Integral R-Curve Testing

• Identify significant hydrogen trap sites and hydride phases (if any), present in selected Al-Li-Cu-Zr alloys
  • Characterized hydrogen partitioning as a function of microstructural features and aging condition
    Thermal Desorption Spectroscopy (TDS)

• Correlate internal hydrogen embrittlement (IHE) with mobile and trapped hydrogen concentrations and operative trap sites
1. Cathodic polarization of Ni-coated alloys

Sputter etch native Al₂O₃ under high vacuum → deposit 400Å nickel coating → cathodic polarization in buffered solution for 28 days → mechanical testing / TDS

2. Straining electrode test (SET)

Straining of flat tensile electrode at eng. strain rate of 2x10⁶ sec⁻¹ in a pH 1 HCl solution at -2V_{SCD}, for approx. 4 hrs. to a total strain of 3% → TDS (after J. Albrecht, I.M. Bernstein, A.W. Thompson, Met. Trans. 13A, 1982)

3. Alternate immersion (AI) aqueous exposure

AI in 3.5 wt.% NaCl/moist air, at 70% yield strength (UA) or 80% yield strength (PA) for 84 days → TDS / metallographic observation
Total Absorbed Hydrogen Concentrations
(Trapped + Lattice) in AA2090

As-received
Ni-coated -1 V_{sce} pH 7
SET, 3% strain -2 V_{sce} pH 1
Al 84 days 3.5 wt.% NaCl

Hydrogen Concentration (wt. ppb)

100
200
300
400
500
600
7000

UA
PA

28 days (bulk concentration)
(max. concentration)
Commercial AA2090 - unrecrystallized:

Al - 2.15Li - 2.56Cu - 0.12Zr, wt.%
4400 µm x 600 µm x 10 µm grain size, 5-10 µm subgrains

T3: solutionized and cold worked
Underaged (UA): T3 + 5 hrs. @ 160°C
Peak aged (PA): T3 + 25 hrs. @ 160°C
\[ \sigma_{YS} = 255 \text{ MPa}; \quad \sigma_{UTS} = 303 \text{ MPa} \]
\[ \sigma_{YS} = 296 \text{ MPa}; \quad \sigma_{UTS} = 476 \text{ MPa} \]
\[ \sigma_{YS} = 434 \text{ MPa}; \quad \sigma_{UTS} = 572 \text{ MPa} \]

Recrystallized Al-Li-Cu-Zr:

Al - 2.05Li - 2.27Cu - 0.09Zr, wt.%

30 µm equiaxed grains (FGR)
Underaged (UA): T3 + 5 hrs. @ 160°C
Peak aged (PA): T3 + 25 hrs. @ 160°C
\[ \sigma_{YS} = 303 \text{ MPa}; \quad \sigma_{UTS} = 421 \text{ MPa} \]
\[ \sigma_{YS} = 400 \text{ MPa}; \quad \sigma_{UTS} = 496 \text{ MPa} \]

800 x 800 x 100 µm (MGR)
Underaged (UA): T3 + 5 hrs. @ 160°C
Peak aged (PA): T3 + 25 hrs. @ 160°C
\[ \sigma_{YS} = 241 \text{ MPa}; \quad \sigma_{UTS} = 379 \text{ MPa} \]
\[ \sigma_{YS} = 400 \text{ MPa}; \quad \sigma_{UTS} = 490 \text{ MPa} \]

1200 x 1100 x 250 µm (LGR)
Underaged (UA): T3 + 5 hrs. @ 160°C
Peak aged (PA): T3 + 25 hrs. @ 160°C
\[ \sigma_{YS} = 296 \text{ MPa}; \quad \sigma_{UTS} = 393 \text{ MPa} \]
\[ \sigma_{YS} = 379 \text{ MPa}; \quad \sigma_{UTS} = 434 \text{ MPa} \]

tensile properties for L oriented flat tensile specimens tested in lab air
## CERT RESULTS

**Unrecrystallized AA2090**

<table>
<thead>
<tr>
<th>Temper</th>
<th>Orientation</th>
<th>Charging Condition</th>
<th>Reduction in Area (% loss)</th>
<th>Fracture Mode</th>
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<tbody>
<tr>
<td>T3</td>
<td>T</td>
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<td>27.98*</td>
<td>transgranular shear</td>
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<td></td>
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<td>28 days @ -1V_{SCE}</td>
<td>17.49*</td>
<td>TGS + intersubgranular</td>
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<tr>
<td>T3</td>
<td>L</td>
<td>Uncharged</td>
<td>19.97*</td>
<td>transgranular shear</td>
</tr>
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<td>28 days @-1V_{SCE}</td>
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<td>TGS + intersubgranular</td>
</tr>
<tr>
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<td>L</td>
<td>Uncharged</td>
<td>13.35*</td>
<td>transgranular shear</td>
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<td></td>
<td>28 days @-1V_{SCE}</td>
<td>11.94*</td>
<td>hydrogen affected TGS</td>
</tr>
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<td>T</td>
<td>Uncharged</td>
<td>32.27*</td>
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</tr>
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<td></td>
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<td>38.40*</td>
<td>transgranular shear</td>
</tr>
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<td>L</td>
<td>Uncharged</td>
<td>16.30*</td>
<td>transgranular shear</td>
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<tr>
<td></td>
<td></td>
<td>28 days @-1V_{SCE}</td>
<td>14.85*</td>
<td>transgranular shear</td>
</tr>
</tbody>
</table>

All specimens charged in 0.25 M Na$_2$SO$_4$ buffered to pH 7.

# - average of 3 specimens

* - single specimen tested
Effect of Hydrogen Pre-charging on the Mechanical Behavior of Unrecrystallized AA2090

All specimens pre-charged at -1 V_{sce} in pH 7 solution for 28 days
Effect of Hydrogen Pre-charging on the Mechanical Behavior of Al-Li-Cu-Zr Alloys

All specimens pre-charged at $-1V_{SCE}$ in pH 7 solution for 28 days
Summary: Mechanical Testing

- Elevated levels of pre-dissolved hydrogen have been shown to result in the HE of Al-Li-Cu-Zr alloys in the T3, UA, and PA tempers. This HE has been observed by fracture toughness and CERT testing of pre-charged samples for a limited combination of strain rate and pre-dissolved hydrogen level.

- Pre-dissolved hydrogen alters transgranular shear in Al-Li-Cu-Zr alloys. This effect is most obvious in the T3 temper for CERT and fracture toughness testing. Absorbed hydrogen has been shown to enhance slip localization.

- Pre-dissolved hydrogen enhances intergranular cracking in Al-Li-Cu-Zr when high angle grain boundaries are oriented favorably with respect to the tensile stress and a sufficiently high tensile stress is developed.

- The favorable orientation of subgrain boundaries does not result in a significant fraction of intersubgranular cracking under the combinations of strain rate and hydrogen concentrations investigated.
Questions Developed From Mechanical Testing

- How many trapping states exist in AA2090 and what microstructural features are they associated with?
- How does aging affect trapping and can this explain IHE behavior?
- Is hydrogen absorption during AI/SCC or concurrent polarization/straining dominated by dislocation transport or mechanical destabilization of passive films coupled with locally high hydrogen fugacities?
- Can a fixed concentration of internal hydrogen repartition during straining?
Trap Site Identification
AA2090 - UA, 10°C/min

- total desorption rate
- constituent peaks
- fit results

peak 5 (grain boundaries or $T_1$)
peak 4 (dislocations)
peak 3 ($\delta'$)
peak 2 ($Li_{ss}$)
peak 1 (matrix)
peak 6 (g.b. or $T_1$)
Desorption Spectra
Al - 3 wt. % Li, 10°C/min

Temperature (°C)

Desorption Rate (g H₂ / g-sec)

- Al-3 wt% Li SHT
- Al-3 wt% Li +5 hrs@160°C
- Al-3 wt% Li +25hrs@160°C
- AA2090-UA

surface Li or Li in hydrated oxide

Liₚ

δ'}
Hydrogen Partitioning for AA2090 and Al- 3 wt. % Li

Aging Condition

Fraction of Hydrogen

AA2090
- • peaks 1+2
- ■ peak 3
- ▲ peak 4
- ▼ peaks 5+6

Al-3wt.%Li
- ○ surface Li + L_{ss}
- □ δ'
Hydrogen Partitioning for AA2090-UA

- Lattice Hydrogen (peaks 1+2)
- peak 3
- peak 4
- peaks 5+6

Desorption Rate (g H₂/g-sec)

Time (sec)

Fraction of Hydrogen

AA2090-UA
charged

AA2090-UA
prestrained 3%
Al-Li-Cu-Zr recrystallized, 10°C/min

Desorption Rate (g H₂/g-sec)

Time (sec)

Hydrogen Partitioning for Al-Li-Cu-Zr

- Lattice Hydrogen (peaks 1+2)
- peak 3
- peak 4
- peaks 5+6
Summary: Trap Site Identification

- Solution heat treated and aged Al-3%Li was used to identify hydrogen trapped at: \( \text{Li}_{ss} \) and \( \delta' \) (\( \text{Al}_3\text{Li} \)).

- Variation of aging times for AA2090 aided in the identification of hydrogen associated with \( \text{Li}_{ss} \), \( \delta' \) (\( \text{Al}_3\text{Li} \)) and \( \text{T}_1(\text{Al}_2\text{CuLi}) \).

- Plastic straining of AA2090 was exploited to identify trapping at dislocations.

- Variation of recrystallized grain size followed by aging was utilized to identify trapping at high angle boundaries with \( \text{T}_1 \).

- Comparison of AA2090 and recrystallized Al-Li-Cu-Zr aided in establishing whether hydrogen was trapped at subgrain boundaries.

- Solution heat treated and aged Al-1% and Al-4%Cu were utilized to identify hydrogen trapping at: \( \text{Cu}_{ss} \), \( \text{GP}_{\text{zones}} \), \( \theta^\prime \), \( \theta' \), and \( \theta \) (\( \text{Al}_2\text{Cu} \)).

- By a process of elimination trapping state 1 was identified as interstitial hydrogen and trapping at \( \text{T}_1 \) particles was determined to be trapping state 5 or 6.
Hydrogen Partitioning for AA2090-UA

- Lattice Hydrogen (peaks 1+2)
- peak 3
- peak 4
- peaks 5+6

AA2090-UA, 10°C/min

charged -1V SCE pH 7
precharged, strained 3%

Desorption Rate (g H₂/g-sec)

Time (sec)

0.0e+0
0.2e-11
0.4e-11
0.6e-11
0.8e-11
1.0e-11
1.2e-10

Fraction of Hydrogen

0.0
0.2
0.4
0.6
0.8

AA2090-UA charged
AA2090-UA charged, strained 3%
Desorption Spectra
AA2090 SET, 10°C/min

Desorption Rate (g H₂ / g·sec)

Time (sec)

- AA2090-T3-L
- AA2090-UA-L
- AA2090-PA-L
- AA2090-UA -2Vₚₛₑ pH 1
- AA2090-UA -1Vₚₛₑ pH 7

SET at -2Vₛₑ pH 1, 2 x 10⁻⁸ sec⁻¹
Desorption Spectra, TL Oriented AA2090, 10°C/min

Spectra of Bulk Sample Subtracted from Fracture Surface

Temperature (°C)

Normalized Desorption Rate

Time (sec)

- AA2090 - T3
- AA2090 - UA
- AA2090 - UA
- AA2090 - PA

peak 1 interstitial H
peak 2 Li_{ss}
peak 3 δ'
peak 4 dislocations
peak 5 grain boundaries?
peak 6 T_{1}?
Summary: Hydrogen Transport

• Dislocations can collect trapped hydrogen from lower energy trapping states during dynamic straining.

• Hydrogen transported by dislocations can be deposited at higher energy trapping sites, such as grain boundaries, within a plastic zone.

• An increased rate of hydrogen uptake has been measured after dynamic straining in pH 1 HCl solution under cathodic polarization or in moist laboratory air. Hydrogen uptake is facilitated by dislocation transport.
• Elevated levels of absorbed hydrogen results in intergranular cracking when high angle grain boundaries are oriented favorably with the tensile stress and a sufficient stress concentration is present.

  • FGR Al-Li-Cu-Zr, UA temper - demonstrated a change in fracture mode from TGS to IG with hydrogen pre-charging ($J_i$ also decreased).

  • FGR Al-Li-Cu-Zr, PA temper - hydrogen pre-charging altered the existing IG fracture ($J_i$ also decreased).

  • MGR and LGR Al-Li-Cu-Zr - Independent of temper and absorbed hydrogen level fracture was dominated by TGS.

  • Pre-charged TL oriented AA2090 - demonstrated more IG cracking than pre-charged LT oriented AA2090 ($J_i$ decreased with hydrogen pre-charging for all tempers of TL oriented AA2090).

  • High angle grain boundaries trap hydrogen and are one of the highest energy trapping sites for hydrogen in Al-Li-Cu-Zr alloys.

Conclusions
Conclusions (cont.)

• Elevated levels of absorbed hydrogen may promote slip localization.
  
  • Dislocations are effective trapping sites for Al-Li-Cu-Zr alloys.
  
  • Enhanced deformation has been demonstrated for pre-charged AA2090 (enhanced deformation T3 > UA > PA).
  
  • Pre-charged FGR Al-Li-Cu-Zr, UA temper - possess planar slip bands on IG fracture facets.

• Hydrogen is implicated in the EAC of Al-Li-Cu-Zr alloys.
  
  • The rate of hydrogen absorption is increased for concurrent straining and charging.
  
  • Hydrogen is absorbed during Al aqueous exposure of AA2090.
  
  • The preferred cracking path during Al aqueous exposure and mechanical testing of specimens with pre-dissolved hydrogen is IG cracking.
  
  • The preferred cracking path correlates with a high energy trapping state.
Conclusions

1) Elevated levels of absorbed hydrogen results in intergranular cracking when high angle grain boundaries are oriented favorably with the tensile stress and a sufficient stress concentration is present.

2) Elevated levels of absorbed hydrogen may promote slip localization.

   Elevated levels of absorbed hydrogen have been shown to affect deformation during intergranular and transgranular shear fracture processes. Dislocations have been shown to trap and transport hydrogen.

3) Hydrogen is implicated in the EAC of Al-Li-Cu-Zr alloys.
Project #5  Metallurgical Factors Controlling Stress Corrosion Cracking in Advanced Al-Li-Cu Alloys

Keith Eklund and John R. Scully

Objective

The objective of this new project is to characterize the stress corrosion initiation and propagation behavior of two selected advanced Al-Li-Cu alloys under chromate inhibited full immersion conditions, as well as alternate immersion conditions. We aim to correlate SCC kinetics with cracking, metallurgical variables (aging condition, orientation, composition, processing) and, to a limited extent, electrochemical variables using diagnostics such as thermal desorption trapping analysis.

Approach

The following alloys will be investigated: C155 (approx: 3.1% Cu, 1.8% Li, 0.35% Mg, 0.16% Zr, 0.6% Mn, 0.35% Zn; max wt. pct.) and 2096 (approx. 3.0% Cu, 1.9% Li, 0.8% Mg, 0.18% Zr, 0.6% Ag, 0.25% Zn; max. wt. pct.). The approach to this new project will involve:

- Characterize SCC threshold stress intensities and stage II da/dt using conventional fatigue precracked compact tension specimens (inhibited full immersion) or double beam bolt loaded configurations (alternate immersion) for various aging treatments, specimen orientations, and/or processing variables.
- Use SEM fractography to identify crack initiation sites and cracking paths
- Correlate the above findings and metallurgical as well as compositional variables with thermal desorption analysis of trapping states. TDS analysis will be performed on specimens sectioned from the plastic zone of SCC specimens.
- Conduct other diagnostics (i.e.: dissolve δ' to change deformation mode)
- Compare threshold stress intensities and, possibly, growth rates between long cracks and short cracks to investigate differences: i.e. examine "chemical crack length" effects.
The proposed work will provide direct correlation between metallurgical variables (Ag content at the same Cu/Li ratio) and SCC resistance, that will lead to improved mechanistic understanding of the factors controlling SCC in advanced Al-Li-Cu alloys and serve as inputs to future alloy design.

Project 5: Localized Corrosion and Stress Corrosion Crack Initiation in Advanced Al-Li-Cu Alloys

Glenn E. Stoner and John R. Scully

Objective

The objective of this new project is to understand localized corrosion and the factors governing the transition from localized corrosion to SCC/CF initiation. We aim to define the "windows" of susceptibility to crack initiation in terms of metallurgical (composition, aging condition and strengthening precipitate development, constituent particles, and specimen orientation), electrochemical (applied potential, localized corrosion site propagation rate), chemical (macrocell and microcell composition), and mechanical (dynamic strain rate, stress state, localized corrosion site flaw dimensions) variables.

Approach

The following alloys will be investigated: C155 (approx: 3.1% Cu, 1.8% Li, 0.35% Mg, 0.16% Zr, 0.6% Mn, 0.35% Zn max wt. pct.) and 2096 (approx: 3.0% Cu, 1.9% Li, 0.8% Mg, 0.18% Zr, 0.6% Ag, 0.25% Zn max. wt. pct.). These alloys have approximately the same Cu/Li ratio but 2096 contains Ag. The approach to this new project will involve:

- Characterize conditions for localized corrosion behavior in precursor "macrocell" local chemistry environments and refined local environments using advanced electrochemical (current transient) and chemical diagnostics (CE).
- Superimpose mechanical variables such as dynamic strain rate, stress state, and/or cyclic strain to define windows of susceptibility to crack initiation using blunt notch specimens. Employ ex-situ crack initiation detection by metallographic and SEM techniques, and in-situ detection by use of a long focal length microscope. Characterize the critical
corrosion site flaw size necessary to initiate SCC. Attempt to determine threshold stress intensities and possibly growth rates from short cracks to ascertain any evidence of differences between the behaviors of short versus long cracks, i.e. a "chemical crack length" effect.

Payoff

The proposed work will provide correlations between localized corrosion and SCC initiation that will lead to improved mechanistic understanding of the factors controlling the transition to SCC. $K_{th}$ for short cracks along with critical corrosion site flaw sizes will be obtained.

Coordination

The two projects are designed to be complimentary but not redundant. For perhaps the first time a comprehensive understanding of the transition from localized corrosion to SCC initiation and propagation may be achieved.
Investigation of the same two alloys will strengthen understanding of the roles of metallurgy, processing and composition: (initiation versus propagation debate). Results from each study will provide direct input to the other. Examples include:

- Characterization of local macrocell chemistry developed in an alternate immersion double beam bolt loaded configuration will provide guidance on environments for blunt notch SCC initiation experiments.

- Determination of windows of SCC initiation susceptibility will guide follow-on conventional SCC initiation and growth test conditions.

- The electrochemical activity of T, Ω or other phases with and without partitioning of Ag may be compared and contrasted to their function as hydrogen trapping states. Clear guidance on alloy design may emerge.

In summary, a coherent comprehensive picture of localized corrosion behavior and the transition to SCC initiation and growth will be developed.
Objective

The broad objective of this research is to characterize and understand the relationships between microstructure, deformation mode, and fracture resistance of high strength titanium alloys for HSCT applications. In this project, the effects of time, temperature, and dissolved hydrogen on the precracked-fracture resistance of an advanced metastable β-titanium alloy will be characterized. The mechanisms for deformation and fracture will be physically defined and micromechanically modeled.

Current Status

The current work in this project is focusing on hydrogen-precharging and fracture toughness testing of high strength solution treated and aged (STA) Beta-21S and Low Cost Beta at temperatures ranging from 25 to 150°C. It has been observed that dissolved hydrogen decreases the plane strain initiation fracture toughness of STA Beta-21S to 30 pct of the value for the alloy without additionally added hydrogen. Fracture mode changes in concert with this reduction in toughness. These results are consistent with work by Scully and his students. Long term fracture toughness experiments with Beta-21S revealed unacceptable variability in the electrical potential signal being used to monitor crack extension during loading. Experiments were conducted to solve this problem.

Preliminary investigation of an LCB sheet provided by McDonnell Douglas has begun. Solution treated sheet was aged at 510°C for 8 hours in an argon atmosphere, and is currently being machined into four compact tension [C(T)] specimens. Two specimens will be precharged to a hydrogen concentration between 500 and 1000 wppm, while two will be tested with the as-received hydrogen concentration of 150 wppm. Charged and uncharged CT specimens will be fractured at 25 and 150°C to scope the effects of dissolved hydrogen and temperature on deformation mode and fracture resistance. A section of the sheet is being used to determine the
diffusivity of hydrogen in the solution treated condition. Metallographic techniques are being developed to characterize both the ST and STA microstructures.

A major focus of this project is on TIMETAL Low Cost Beta. Timet has recently cold rolled LCB sheet (6 pieces; 1.52 mm x 200 mm 390 mm or 0.060" x 8" x 15.3") and plate (1 piece; 10.16 mm x 152 mm x 254 mm or 0.4" x 6" x 10"). Three sheets and the plate will be solution treated in the $\alpha + \beta$ phase field, cooled to 25°C and isothermally aged at a temperature that is known to provide a balance of yield strength and fracture toughness. Three sheets will be provided in the as-cold rolled condition to heat treatment studies by Kazanjian and Starke at UVa. These materials should be delivered by August.

Recent Findings

The rising load R-curve test was used to determine the fracture toughness of hydrogen-precharged STA Beta-21S plate at 75°C with a slow load-line displacement rate of 0.22\mu m/min. This sample was charged at a current density of 0.30 mA/cm², as was a specimen previously tested at 150°C. However, the 75°C specimen surface was not masked, while the 150°C specimen had the area near the loading pin holes coated to prevent hydrogen uptake and embrittlement. As a result, hydrogen in the 150°C specimen had ample time to diffuse to the masked areas thereby lowering the hydrogen concentration immediately ahead of the precrack. Consequently, it is expected that the 75°C specimen had a hydrogen concentration between 500 and 1000 wppm while the 150°C was lower. Sections of each specimen were sent to LECO for nominal hydrogen content analysis.

The plane strain fracture toughness of the Beta-21S C(T) specimen, hydrogen-precharged and tested at 75°C, was 23.7 MPa/m based on ASTM standard E-399 and a 95% slope-intercept method. This is significantly reduced from previously determined values of 71.9, 65.9, and 53.5 MPa/m for uncharged specimens of Beta-21S at 150 and 25°C, and a hydrogen charged specimen at 150°C, respectively. Fractography of the specimen tested at 75°C showed several modes of fracture which were not observed previously. This indicates that hydrogen, or a hydrogen/temperature combination, degrades the fracture resistance of high strength STA Beta-21S.
Future Work

Work over the next six months will focus on: (1) completing preliminary fracture experiments with STA Beta-21S and LCB sheet provided by McDonnell Douglas, (2) beginning a systematic study of internal hydrogen embrittlement of a single STA condition of LCB sheet provided by TIMET, (3) studying the effect of fatigue precracked specimen thickness on resistance curve fracture toughness for uncharged and hydrogen-charged TIMET STA LCB plate, and (4) investigating the deformation and fracture modes in LCB of varying fracture test temperature and dissolved hydrogen concentration for fixed strength and microstructure.

Presentation Viewgraph Captions

1. Title.
2. Objectives
3. Compositions of TIMETAL Beta-21S and TIMETAL LCB.
4. Calibration curve relating applied electrochemical cathodic current and hydrogen uptake in STA Beta-21S exposed to aqueous $\text{H}_2\text{SO}_4 + \text{Na}_4\text{P}_2\text{O}_7$ at 90°C for 7 days. The majority of these results were obtained by Gaudett, Young, and Scully who are assisting with the charging of both Beta-21S and LCB.
5. C(T) specimen, buckling restraints, crack mouth opening displacement measurement by LVDT, and dcEPD probes used for determining elevated temperature fracture toughness of thin sheet alloys.
6. Load versus crack mouth opening displacement data for STA Beta-21S C(T) specimens at 25°C and 150°C, without precharged hydrogen, and at 75°C and 150°C with precharged hydrogen. A slow loading rate was employed such that each experiment required 20 to 50 hours to fracture. The deleterious embrittling effect of predissolved hydrogen at 75°C is apparent.
7. Plane strain initiation fracture toughness, defined by a loading slope secant offset method from ASTM Standard E-399, for the four specimens of STA Beta-21S at the various test temperatures and hydrogen-precharged conditions represented in Figure 6. Note the reduction in initiation toughness due to dissolved hydrogen.
8. Direct current electrical potential versus time for an unloaded C(T) specimen which shows signal variability on the order of 5 microvolts. This behavior is unacceptable and will hinder detection of the onset of cracking.
9. Direct current electrical potential versus time for an unloaded C(T) specimen which shows drift in the measured potential over time. This behavior is unacceptable and will hinder detection of the onset of cracking.

10. Measured load and electrical potential versus crack mouth opening displacement for a Beta-21S C(T) specimen (hydrogen precharged to about 700 wppm and loaded at 75°C and a slow constant load-line displacement rate of 0.2 μm/sec). This plot shows the excellent agreement in crack initiation load, resolved by either the first deviation from linearity in load vs displacement or the change in slope of the electrical potential vs displacement. For this and other experiments with high strength β-titanium alloys, small scale yielding is maintained; the contribution of the plastic component of the J-integral is small, enabling a simple elastic stress intensity analysis of fracture toughness.

11. Stress intensity vs crack extension R-curves for two C(T) specimens of STA Beta-21S: (1) uncharged and fractured at 25°C, and (2) hydrogen-precharged and fractured at 75°C. A slow loading rate was employed for each case. Note the strong and deleterious effect of hydrogen embrittlement. (Previous experiments represented in Figure 6 show that the R-curve for as-received STA Beta-21S is somewhat higher for loading at 150°C compared to the result here for loading at 25°C. Presumably, fracture of Beta-21S without hydrogen is similar at 75°C.)

12. Results reported by Scully and coworkers establish the reduction in initiation fracture toughness, with increasing dissolved hydrogen concentration, for precharged STA Beta-21S and STA Ti 15-3 fractured by slow loading at 25°C. The current result for precharged Beta-21S at 75°C is both consistent with the general deleterious effect of dissolved hydrogen, and shows a potentially increasingly deleterious effect of hydrogen at elevated temperature. Also note the complex fracture surface morphologies that are typical of both ductile fracture and internal hydrogen embrittlement of these high strength β-titanium alloys.

13. Scanning electron fractographs of STA Beta-21S show the change in crack surface morphology produced by dissolved hydrogen. Fracture of Beta-21S, at 150°C and without precharged hydrogen, produced a transgranular crack surface with at least two types of ductile fracture features as shown by the two fractographs on the left side of the figure. Note the fatigue precrack in the bottom portion of each fractograph. Fracture of Beta-21S, at 75°C and with precharged hydrogen, produced a mixed transgranular-intergranular surface with multiple features labelled a through f (see the two fractographs on the right side of the figure). Different fracture processes appear to be operative depending on the dissolved hydrogen concentration.

14. Higher magnification scanning electron fractographs of the hydrogen-precharged C(T) specimen of STA Beta-21S fractured at 75°C. These six areas are contained in the lower right fractograph in Figure 13. Each of these features in (a) through (f) appear to be unique to hydrogen-charged Beta-21S and internal hydrogen embrittlement, however, the cause of each feature is not understood fundamentally. For example, intergranular
cracking has been reported by others at UVa investigating the aqueous chloride stress corrosion cracking of Beta-21S, and may be traced to hydrogen trapping and decohesion of \( \beta \)-grain boundaries. These workers hypothesized a role of locally intense slip in nucleating boundary hydrogen embrittlement. The linear features in (b) and (e) may be indicative of this process. A substantial effort is required to understand the microscopic processes that control ductile fracture and internal hydrogen embrittlement in high strength \( \beta \)-titanium alloys. This work will be complicated by the fine-scale microstructures typical of cold worked and aged alloys such as LCB sheet.

15. Fracture experiments will soon be conducted on STA LCB sheet obtained from McDonnell Douglas, as a precursor to a more systematic study of LCB sheet from Timet.

16. Schematic diagram of the method employed to determine the diffusivity of hydrogen in \( \beta \)-titanium alloys.

17. Results of the measurement of the diffusivity of hydrogen at room temperature in annealed single phase (bcc) Beta C; after Scully and Young.

18. Conclusions.

19. Future work with TIMETAL LCB sheet and plate.

20. TIMETAL LCB will be employed for the next phase of the present study of internal hydrogen embrittlement, and will also be investigated by Kazanjian and Starke.
MECHANISMS OF DEFORMATION AND FRACTURE IN HIGH STRENGTH TITANIUM ALLOYS: EFFECTS OF TEMPERATURE AND HYDROGEN

Sean P. Hayes and Richard P. Gangloff

Funded by NASA Langley Research Center
Dennis Dicus, Grant Monitor
Objectives

- Understand the relationships between microstructure, deformation mode, and fracture resistance of titanium alloys for HSCT applications.

- Characterize the effects of time, temperature, and predissolved hydrogen on the fracture resistance and growth fracture resistance of an advanced metastable $\beta$-titanium alloy.

- Physically define mechanisms and micromechanically model hydrogen-enhanced deformation and fracture.
<table>
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<th>Alloy</th>
<th>Ti</th>
<th>Mo</th>
<th>Fe</th>
<th>Al</th>
<th>Nb</th>
<th>O</th>
<th>H</th>
<th>Balance</th>
<th>TIMETAL</th>
<th>LCB</th>
<th>Balance</th>
<th>TIMETAL</th>
<th>21S</th>
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<td>--</td>
<td>0.128</td>
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<td>Alloy Compositions (wt. pct.)</td>
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</tbody>
</table>
Beta 21S Calibration Curve

Beta 21S (STA)
H₂SO₄ / Na₄P₂O₇
90°C
7 days

Hydrogen Concentration (wppm)

Current Density (mA/cm²)

- • Young, Gaudette, and Scully
- ○ Hayes
C(T) Specimen Detail for Thin Sheet $J-\Delta a$ Determination
Beta 21S Plate, STA

\[ \sigma_{ys} = 1425 \text{ MPa} \]

\[ \delta = 0.2 \mu\text{m/sec} \]
BETA 21S FRACTURE TOUGHNESS RESULTS

<table>
<thead>
<tr>
<th>Test Temperature (°C)</th>
<th>Estimated Hydrogen Content (wppm)</th>
<th>Fracture Toughness ($K_{IC}$-MPa$\sqrt{m}$)</th>
<th>Hardness ($R_c$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>100</td>
<td>65.9</td>
<td>41.4</td>
</tr>
<tr>
<td>25</td>
<td>100</td>
<td>67.9</td>
<td>45.2</td>
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<tr>
<td>150</td>
<td>100</td>
<td>71.9</td>
<td>45.2</td>
</tr>
<tr>
<td>75</td>
<td>750</td>
<td>23.7</td>
<td>44.5</td>
</tr>
<tr>
<td>150</td>
<td>&lt; 750</td>
<td>53.5</td>
<td>44.8</td>
</tr>
</tbody>
</table>

$K_{IC}$ is defined by ASTM standard E-399 and a 95% slope-intercept method
Ti 15-3
Room Temperature
K = 0

Potential (microvolts)

Time (seconds)
Ti 15-3
Room Temperature
K = 0
Beta 21S (STA)
700 wppm H
75°C
δ = 0.22 μ/min

- Load (lbs)
- CMOD (mm)
- Potential (microvolts)
Beta 21S (STA)

δ = 0.22 μm/min for the 75°C test

δ is ~ 8-10x greater for the 25°C test

$K_{IC} = 65.9 \text{ MPa} \sqrt{\text{m}}$ (E-399)

$K_{JIC_i} = 50.6 \text{ MPa} \sqrt{\text{m}}$ (DCPD)

$K_{IC} = 23.7 \text{ MPa} \sqrt{\text{m}}$ (E-399)

$K_{JIC_i} = 23.6 \text{ MPa} \sqrt{\text{m}}$ (DCPD)

△ 25°C, No H precharge

■ 75°C, H precharged
1. M1 = microvoid coalescence
2. M2 = bimodal distribution of microvoids
3. P = M1 + ductile failure along planar slip bands
4. TT = transgranular tearing, fine terraces/ledges
5. IG = intergranular
6. C = cleavage

工作于 UVA 的 G. Young, L. Young, Gaudette, 和 Scully
Beta 21S (STA) Plate

100 wppm H  
150°C

700 wppm H  
75°C
CURRENT WORK - LOW COST BETA

- Determine the diffusivity of hydrogen in solution treated LCB.

- Four CT specimens of LCB sheet (McDonnell Douglas) will be tested to scope the effects of temperature and dissolved hydrogen on fracture resistance:

<table>
<thead>
<tr>
<th>STA LCB</th>
<th>25°C</th>
<th>150°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-received - 150 wppm H</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>Precharged to 500-1000 wppm H</td>
<td>X</td>
<td>X</td>
</tr>
</tbody>
</table>

- Sheet and plate of LCB (ST and STA) are being provided by TIMET.


\[ \eta = \text{erf}^{-1} \left[ 1 - \frac{2(C(x,t) - C_r)}{(C_l - C_r)} \right] \]

\[ D_H = \frac{1}{4t \left( \frac{d\eta}{dx} \right)^2} \]
Beta C (ST)
$D_H = 5.0 \times 10^{-7} \text{ cm}^2/\text{sec}$

After G. Young, UVA
CONCLUSIONS

- Dissolved hydrogen dramatically reduces the fracture toughness of high strength (STA) Beta 21S.  
  500-1000 wppm H reduces $K_{IC}$ by 50% or more

- Dissolved hydrogen causes a distinct change in fracture mode in STA Beta 21S plate. The morphologies of air and hydrogen fracture are complex.

- Problems associated with direct current potential drop (DCPD) monitoring of crack growth have been corrected.
FUTURE WORK - LOW COST BETA

- Determine the diffusivity of hydrogen in STA LCB at several temperatures.

- Establish the calibration for hydrogen charging of STA LCB in H₂SO₄ at 90°C.

- Characterize the effects of dissolved hydrogen and temperature on the fracture resistance \( (K_{TH}, K-\Delta a, da/dt(K)) \) of a single microstructure of 0.060" LCB sheet.

- Study the effect of fatigue precracked specimen thickness on the fracture toughness resistance curve in moist air and perhaps for IHE for STA LCB.
TIMET LCB MATERIAL SUPPLY

- Six LCB sheets - 0.060" x 15.25" x 8"
  - Three will be solution treated in the $\alpha + \beta$ region and subsequently aged
  - Three will be in the as-cold rolled condition

- One LCB plate - 0.4" x 6.75" x 13.5"
  - Heat treated to achieve highest possible strength with acceptable toughness
Project #7  Evaluation of Wide-Panel Aluminum Alloy Extrusions

M.T. Lyttle and J.A. Wert

Research Objectives

The overall objective of this project is to experimentally observe and model the effects of microstructure and texture on yield strength in wide panel aluminum alloy extrusions. The modeling results will enable the prediction of locations and tensile axis orientations corresponding to minimum yield strength where localized yielding could occur in service. A generalized form of these models can be applied to any combination of microstructure and texture found in other aluminum alloys.

Progress During the Reporting Period

A model has been constructed to predict the variations in yield strength due to grain orientation, grain morphology, and precipitate plane orientation for both the plastic and elastic inclusion assumptions. Using specimen texture obtained from pole figure data, an average Taylor factor can be determined for any uniaxial stress orientation in the specimen. Combining specimen texture and precipitate plane orientation, the contribution to strengthening from precipitates with a specified habit plane orientation can be calculated.

Compression testing of different regions of the 2090 extrusion in various in-plane directions in the as-received and the overaged case has been conducted. Yield strength has been measured for the skin and cap regions in the 2090 extrusion, which contain pancake- and lath-shaped grains, respectively. Variations in yield strength are observed in Figure 8 and can be compared roughly to the typical Taylor factor variations predicted in Figure 4. The cap region (lath-shaped grains) generally exhibits a lower yield strength than the skin region (pancake-shaped grains) for all orientations tested. This is consistent with the model predictions illustrated in Figure 4. Note in particular that equal transverse strength (90°) is predicted in the cap and skin locations, precisely matching experimental observations.

To critically evaluate the significance of these predicted yield strength variations, precipitate and texture effects must be eliminated from the experimental observations. To minimize the precipitate strengthening effect, portion of the 2090 skin was overaged at 160°C for
400 hours. The resulting microstructure should contain the same volume fraction of matrix and precipitate as the usual aging procedures, but the coarsened precipitates will contribute less to strengthening and anisotropy. Figure 5 shows the correlation between the yield strength prediction of the plastic inclusion model and the experimental results for the overaged skin section of a 2090 extrusion. The correlation coefficient for the plastic inclusion model is 0.96 and the standard deviation is 9 MPa. These experimental results correspond well to the model predictions.

Refinement of the compression testing apparatus and the use of larger compression specimens has allowed more accurate and consistent yield strength data to be generated.

Recent Important Findings

A model has been developed that incorporates the effects of texture, precipitate characteristics, and grain morphology in a prediction of yield anisotropy. The effect of precipitate characteristics was addressed by consideration of the precipitate as elastic and plastic inclusions in two separate models. Both of these models predict similar behavior, so further research in this area will mainly focus on refinement of the plastic inclusion model. Grain morphology is predicted to have a measurable effect on yield anisotropy and magnitude.

Accurate predictions of yield strength anisotropy of as-received and overaged skin specimens were made. However, experimental results from the cap section deviated from the predictions of the model (standard deviation = 56 MPa). The shape of the curve correlated well with the variation in yield strength (correlation coefficient = 0.92), but the magnitude of variation was not predicted by the model. The observed variation of ~150 MPa in the cap section corresponds to about a 25% variation in yield strength as a function of stress axis orientation. For typical rolling texture, anisotropy of this magnitude is not usually predicted for Taylor factor (2.7 to 3.4) or precipitate strengthening factor (0.65 to 0.81). It is encouraging that, for all measured orientations, the current model does predict the correct trends in the anisotropy, and in most cases the magnitude of the anisotropy.

Since the yield strength predictions for the cap section specimens were the least accurate and good results were obtained for the overaged specimens, further refinement of the model appears to be necessary to correctly account for lath-shaped grains and precipitate strengthening.
Next Six Month Milestones

Development of the capability to calculate average Taylor factor and the strengthening contribution of precipitates in the plastic and elastic inclusion models for a specified uniaxial stress orientation is complete. The yield strength model consisting of grain orientation and precipitate orientation data accurately predicts the general trends of yield strength variation for the alloys tested, but to generate a more complete and exact description of the yield anisotropy, the effect of basic grain morphology effects will need further refinement.

Accurate model predictions have been made for several regions in the 2090 extrusion. Quantitative metallography will be performed on the tested regions of the 2090 extrusion to see if there is a preferred orientation of precipitates and to explicitly determine the matrix and precipitate material property inputs for the model. These results will be included in the model for 2090. To assess the general applicability and to fine tune the adjustable morphological parameters of these models, extensive yield strength testing, texture measurements and microstructural characterization will be conducted on 2096 and 2195 extrusions.

Viewgraph Captions

1. Title and Outline.

2. Approach - A brief description of the plastic inclusion model which is used to predict yield strength anisotropy given texture, grain morphology, and precipitate orientation data.

3. Dependence of Taylor Factor on Grain Shape - How Taylor factor is calculated for equiaxed and pancake-shaped grains.

4. Effect of Grain Morphology on Taylor Factor - Taylor factor variation for a rolling texture of a sample containing grains with equiaxed, pancake, and lath morphology.

5. 2090 Skin Overaged Yield Strengths - A comparison of experimental and predicted yield strengths for the 2090 skin in an overaged condition.


8. 2090 Skin and Cap Yield Strengths - A comparison of experimental and predicted yield strengths for the 2090 skin and cap regions.

9. Conclusions and Future Work
Effects of Texture and Microstructure on the Anisotropic Yield Strength of Wide Panel Aluminum Alloy Extrusions

M. T. Lyttle
J. A. Wert

University of Virginia
Department of Materials Science and Engineering

Objectives

- Characterize the effects of texture and microstructure on the yield strength in wide panel aluminum alloy extrusions

- Develop models which enable the prediction of locations in the extrusion and tensile axis orientations of minimum yield strength where localized yielding could occur in service
Approach

- Examine factors that influence the yield strength anisotropy of a material and construct models to predict this variation.

- Models have been proposed which consider precipitate strengthening as either elastic or plastic inclusions. While the models are derived differently, similar results are predicted by both.

\[ \sigma = M \tau (1 - f) + N \sigma_p f \]

1. Grain Orientation - Determines average Taylor factor for a specified stress axis orientation
   - Grain Morphology - Deviation from spherical grains relaxes constraints from adjacent grains, modifying the average Taylor factor

2. Precipitate Orientation - Affects the amount of strengthening due to precipitates

- Compression tests of 2090 extrusion in the as-received and an overaged condition were conducted and validated the initial model constructions.

- Using the same modeling and experimental methods in different aging conditions and on 2096 and 2195 extrusions, the role of grain morphology and precipitate orientation in influencing yield strength anisotropy will be investigated.
**Dependence of Taylor Factor on Grain Shape**

**Spherical grains**

<table>
<thead>
<tr>
<th>Stress State</th>
<th>Strain State</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\begin{bmatrix} -\sigma &amp; 0 &amp; 0 \ 0 &amp; 0 &amp; 0 \ 0 &amp; 0 &amp; 0 \end{bmatrix}$</td>
<td>$\begin{bmatrix} -\varepsilon &amp; 0 &amp; 0 \ 0 &amp; -\varepsilon/2 &amp; 0 \ 0 &amp; 0 &amp; -\varepsilon/2 \end{bmatrix}$</td>
</tr>
</tbody>
</table>

- Spherical grains are completely constrained by their neighbors
- Strain State is defined strictly by Hooke's Law
- Five slip systems are required to achieve the prescribed strain state
- Using pole figure data, generate a set of grain representative of the specimen texture
- Calculate the Taylor factor for each of these grains and average Taylor factor can be calculated directly

**Pancake shaped grains**

<table>
<thead>
<tr>
<th>Stress State</th>
<th>Strain State</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\begin{bmatrix} \sigma &amp; 0 &amp; 0 \ 0 &amp; 0 &amp; 0 \ 0 &amp; 0 &amp; 0 \end{bmatrix}$</td>
<td>$\begin{bmatrix} -\varepsilon &amp; x &amp; 0 \ 0 &amp; -\varepsilon/2 &amp; 0 \ 0 &amp; 0 &amp; -\varepsilon/2 \end{bmatrix}$</td>
</tr>
</tbody>
</table>

- Deviation from equiaxed grains causes a relaxation in constraint from adjacent grains
- Surface tractions on large grain boundaries result in shear strains that can be accommodated by adjacent grains
- This accommodation enables fewer slip systems to achieve a modified strain state resulting in a lower Taylor factor
Effect of Grain Morphology on Taylor Factor

![Graph showing the effect of grain morphology on Taylor factor.

- **Equiaxed**
- **Pancake**
- **Lath**

Taylor Factor vs. Angle With Extrusion Direction

- **Equiaxed** curve shows a decrease in Taylor factor with increasing angle.
- **Pancake** curve shows a similar trend.
- **Lath** curve shows a decrease initially, then increases slightly.

Graph scales:
- Y-axis: Taylor Factor (3.3 to 2.9)
- X-axis: Angle With Extrusion Direction (0 to 90 degrees)
Comparison of 2090 Skin Overaged Predicted and Experimental Yield Strengths

Overaged Skin Region of 2090 Extrusion
400h @ 160°C

<table>
<thead>
<tr>
<th></th>
<th>Correlation Coefficient</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Overaged Skin Region</td>
<td>0.96</td>
<td>9 MPa</td>
</tr>
</tbody>
</table>
Example of Yield Strength Prediction
Skin of 2090 Extrusion

Taylor Factor

Precipitate Strengthening Ratio
Example of Yield Strength Prediction
Skin of 2090 Extrusion

<table>
<thead>
<tr>
<th></th>
<th>Correlation Coefficient</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Taylor Factor</td>
<td>0.02</td>
<td>18 MPa</td>
</tr>
<tr>
<td>Precipitate Strengthening</td>
<td>0.65</td>
<td>14 MPa</td>
</tr>
<tr>
<td>Yield Strength Model</td>
<td>0.69</td>
<td>13 MPa</td>
</tr>
</tbody>
</table>
Comparison of 2090 Skin and Cap Predicted and Experimental Yield Strengths

Skin Region of 2090 Extrusion

Cap Region of 2090 Extrusion

<table>
<thead>
<tr>
<th>Region</th>
<th>Correlation Coefficient</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Overaged Skin Region</td>
<td>0.96</td>
<td>9 MPa</td>
</tr>
<tr>
<td>Skin Region</td>
<td>0.69</td>
<td>13 MPa</td>
</tr>
<tr>
<td>Cap Region</td>
<td>0.92</td>
<td>56 MPa</td>
</tr>
</tbody>
</table>
Conclusions

• A model has been constructed that allows yield strength predictions based on texture, grain morphology, and precipitate plane orientation inputs.

• Accurate predictions of the trends in yield strength anisotropy in 2090 near net shape extrusions have been made.

• Refinement of compression testing techniques has allowed accurate determination of yield strength (± 5 MPa)

Future Work

• Microstructural characterization of the 2090 extrusions and compression testing at different overaging times will allow refinement of the yield strength models.

• Similar testing will be conducted on 2096 and 2195 extrusions. Using a wide range of grain shapes and precipitate morphologies should allow a greater understanding of the role each play in the strengthening process.
Project Objective

The objective of this project is to investigate the strengthening mechanisms and the void nucleation process in a recently developed Al-0.55Si-2.02Ge (wt.%) alloy. The relationship between precipitate size and critical strain for cavity nucleation during plastic deformation is of primary interest.

Current Status

The volume fraction of the SiGe precipitates in a ternary Al-0.55Si-2.02Ge (wt.%) alloy was determined as a function of aging time using Transmission Electron Microscopy (TEM), electrical conductivity measurements, and X-ray diffraction lattice parameter measurements. The (111), (200), and (220) pole figures were determined from center sections of as-quenched samples and the results were converted into an Orientation Distribution Function (ODF) in order to calculate the Taylor factor.

Finite element calculations were performed on an IBM RS/6000 computer using the Abaqus software package. Meshes containing up to 7,000 triangular six-node elements were generated using the Patran software package. The stress and strain distributions around the SiGe precipitates were calculated for different precipitate diameters using both elasticity and plasticity models.

Recent Important Findings

The correlation between TEM measurements, electrical conductivity measurements, and lattice parameter measurements shows that there is still solute in solution in the T6 condition and that precipitation in Al-0.55Si-2.02Ge (wt.%) continues to occur up to about 100 hrs at 160°C. This observation led to a significant improvement of the theoretical yield strength prediction for this ternary alloy as a function of aging time.

Finite element calculations reveal that the maximum principal stress at the precipitate-matrix interface decreases approximately linearly with increasing precipitate diameter for both
the elasticity and plasticity models. This is consistent with the experimental observation that the volume fraction of voids decreases with increasing precipitate diameter for a given strain.

**Future Work**

The direct observation of voids around the SiGe precipitates will be attempted using TEM which would allow the determination of the void nucleation and void growth rates from measurements of the areal void density and the maximum void size as a function of strain for different aging times. This separation of void nucleation and void growth might provide an explanation for the experimental observation that the tensile ductility decreases with increasing aging time while the critical strain for cavity nucleation increases.

Furthermore, the quantification of the precipitate-matrix interfacial structure will be attempted using atomic resolution TEM in order to calculate the stresses associated with ledges and steps.

**Presentation Viewgraphs**

1. Title.
2. Outline.
3. Background.
4. Project objective.
5. Experimental procedure.
6. Experimental procedure (cont.).
7. Change of the SiGe precipitate size during aging at 160°C. The average precipitate diameter increases from 5.0nm after 1 day of aging to 14.4nm after 16 days of aging.
8. TEM microstructure. The microstructure of the ternary alloy after aging for 8 days at 160°C shows SiGe precipitates. <110> Al orientation.
9. Room temperature longitudinal tensile properties as a function of aging time at 160°C. With increasing aging time, the tensile ductility decreases significantly, whereas the decrease of both the yield strength and the tensile strength is almost negligible.
10. Prediction of the total theoretical yield strength. The total theoretical yield strength is estimated by adding the contribution of the precipitates, the yield strength of pure Al, and the contributions from grain boundary and solid solution strengthening.

11. Modified Orowan equation. This equation predicts that the contribution of the precipitates to the yield strength is proportional to the square root of the precipitate volume fraction and inversely proportional to the precipitate diameter. The ratio between the Taylor factor calculated from X-ray diffraction data and the Taylor factor calculated for fcc metals with a random distribution of grain orientations was also taken into account.

12. TEM measurements of the SiGe precipitate volume fraction as a function of aging time at 160°C. This is a direct method to determine the precipitate volume fraction, but it is associated with significant experimental errors.

13. Measurements of the electrical conductivity of the ternary alloy as a function of aging time at 160°C. These measurements are associated with very small experimental errors, but it is an indirect method and Nordheim's rule was used to estimate precipitate volume fractions based on these results.

14. Measurements of the lattice parameter of the matrix as a function of aging time at 160°C. This is a third independent approach to estimate precipitate volume fractions, but is also an indirect method and Vegard's rule was used to convert the data into precipitate volume fractions.

15. Determination of the volume fraction of the SiGe precipitates as a function of aging time at 160°C. The average value of the precipitate volume fractions determined using TEM, electrical conductivity measurements, and lattice parameter measurements was used in the modified Orowan equation to estimate the contribution of the precipitates to the yield strength for each aging time.

16. Size distribution of the SiGe precipitates during aging at 160°C. The precipitate diameter used in the modified Orowan equation was based on the precipitate size distribution for each aging time shown here for the shortest and the longest aging times.

17. SEM fracture surface of the peak-aged ternary alloy after performing a room temperature tensile test in longitudinal direction. The micrographs reveal that fracture occurred as dimpled rupture.

18. Determination of the volume fraction of voids as a function of strain and precipitate diameter. The volume fraction of voids was determined through a pycnometer by measuring densities of the deformed gage section and the grip part. All measurements were performed before necking occurred. The volume fraction of voids decreases with increasing precipitate diameter at a given true strain value. The critical strain to nucleate cavities ($\varepsilon_c$) was determined from the experimental data by linear regression.
19. Energy criterion for cavity nucleation. This criterion predicts that the critical strain to
nucleate cavities is independent of the precipitate size.

20. Geometrical correction factor used in the energy criterion. This correction factor takes
into account that cavities nucleate at particle poles in the direction of the tensile axis.

21. Coarsening theory developed by Lifshitz and Wagner. This theory predicts that, when the
rate of coarsening is controlled by the diffusion of the solute species through the matrix,
the cube of the average precipitate radius increases linearly with aging time.

22. Determination of the precipitate-matrix interfacial free energy. The precipitate-matrix
interfacial free energy ($\gamma$) is a parameter in the energy criterion for cavity nucleation. This
parameter was estimated from coarsening data using the theory developed by Lifshitz and
Wagner. Note that the graph is only drawn for longer aging times when the precipitate
volume fraction stays approximately constant.

23. Evaluation of the energy criterion for cavity nucleation. The experimental results agree
approximately with the prediction of the energy criterion, but the critical strain clearly
depends on the precipitate size contrary to the prediction of this criterion.

24. Stress Criterion for cavity nucleation. This criterion predicts that the critical strain to
nucleate cavities depends linearly on the precipitate size.

25. Evaluation of the stress criterion for cavity nucleation. The experimental results agree
well with the prediction of the stress criterion if the critical stress is chosen as 1/40 of the
shear modulus of the matrix and if the numerical parameter $\alpha$ is chosen as 1/7.

26. Typical finite element mesh used for the calculations of the elastic stress and strain
distributions around two quarter precipitates. This mesh contains 6,570 triangular six-
node elements. Based on symmetry considerations, the boundary conditions were chosen
in such a way that the displacements are zero in the vertical direction along the horizontal
lower edge and in the horizontal direction along the two vertical edges. A uniform load
based on the experimental tensile data was applied in the vertical direction along the
horizontal upper edge.

27. Typical distribution of the maximum principal elastic stresses around two quarter
precipitates. This stress distribution reveals that the stresses associated with the smaller
(left) precipitate are larger than those associated with the larger (right) precipitate. This
is consistent with the experimental observation that the volume fraction of voids
decreases with increasing precipitate diameter.

28. Ramberg-Osgood relationship. This relationship describes the mechanical behavior of the
material as nonlinear at all stress levels, but, for commonly used values of the stress
exponent ($n>5$), the nonlinearity only becomes significant at stress magnitudes exceeding
the yield strength.
29. Typical finite element mesh used for the calculations of the total stress and strain distributions around a quarter precipitate. This mesh contains 3,570 triangular six-node elements. The boundary conditions and the load were chosen as described above.

30. Typical distribution of the maximum principal total stresses around a quarter precipitate. The total stresses were calculated using the deformation plasticity model based on the Ramberg-Osgood relationship described above. Only one quarter precipitate was considered since no overlap of the stress fields of the precipitates occurs.

31. Dependence of the maximum principal stress at the precipitate-matrix interface on the precipitate diameter using the elasticity and the deformation plasticity models. The stress decreases approximately linearly with increasing precipitate diameter for both models. The stresses predicted by the deformation plasticity model are always smaller than those predicted by the elasticity model as should be expected.

32. Conclusions.

33. Future work.
Al-Si-Ge Alloy Development

H.J. Koenigsmann and E.A. Starke, Jr.
Department of Materials Science and Engineering
University of Virginia

Sponsor: NASA LaRC
Outline

- Background and project objective
- Experimental procedure
- Tensile properties
  - Experimental results
  - Strengthening mechanisms
- Cavity nucleation
  - Experimental void volume fractions
  - Cavity nucleation models
  - Finite element calculations
- Conclusions and future work
Background

- Uniform distribution of very small SiGe precipitates
  - Diamond structure
  - Looped by dislocations
- High degree of hardening for a small volume fraction of precipitates
- Precipitate density in Al-Si-Ge alloys is one order of magnitude higher than in Al-Si and Al-Ge alloys
  - Si has smaller and Ge has larger atomic diameter than Al
  - Atomic size misfit can be compensated by pair formation
  - Similarities in electronic structure between Si and Ge
Project Objective

- Investigate strengthening mechanisms and void nucleation process in recently developed Al-0.55Si-2.02Ge (wt.%) 

- Study relationship between precipitate size and critical strain for cavity nucleation during plastic deformation
Experimental Procedure

- Al-0.55Si-2.02Ge (wt.%) provided by Alcoa
  - Compensation of atomic size misfit
- Homogenization
  - 30hrs at 500°C (based on DSC)
- Hot rolling
- Solution heat treatment
  - 1hr at 490°C
- Cold water quenching
- Aging
  - Up to 16 days at 160°C
Experimental Procedure (cont.)

- Tensile tests as function of aging time
- Electron microscopy
  - TEM (quantitative stereology, CBED) and SEM
- Determination of precipitate volume fractions
  - TEM micrographs, electrical conductivity, lattice parameter
- Determination of Taylor factor
  - (111), (200), (220) pole figures, ODF
- Cavity nucleation measurements
  - Void volume fraction measurements (pycnometer)
  - Ultrasonic velocity measurements (SiGe shear modulus)
Change of Precipitate Size during Aging

Al-0.55Si-2.02Ge (wt.%) aged at 160°C

Corrected for truncation and overlap.

- d=14.4 nm
- d=11.5 nm
- d=10.3 nm
- d=8.8 nm
- d=5.0 nm

Average Diameter (nm)

Aging Time (hrs)
Microstructure (TEM)

Al-0.55Si-2.02Ge (wt.%) aged 8 days at 160°C
([110]Al zone axis)
<table>
<thead>
<tr>
<th>$t_a$ (d)</th>
<th>$\sigma_{th}$ (MPa)</th>
<th>$\sigma_Y$ (MPa)</th>
<th>$\sigma_{TS}$ (MPa)</th>
<th>$\varepsilon_f$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>107</td>
<td>105</td>
<td>164</td>
<td>17</td>
</tr>
<tr>
<td>2</td>
<td>90</td>
<td>103</td>
<td>162</td>
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<td>4</td>
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<tr>
<td>8</td>
<td>75</td>
<td>100</td>
<td>155</td>
<td>9</td>
</tr>
<tr>
<td>16</td>
<td>67</td>
<td>94</td>
<td>153</td>
<td>9</td>
</tr>
</tbody>
</table>
Theoretical Yield Strength

\[ \sigma_{th} = \Delta \sigma_p + \sigma_{Al} + \Delta \sigma_b + \Delta \sigma_s \]

- Total theoretical yield strength: \( \sigma_{th} \)
- Contribution of SiGe precipitates to the yield strength: \( \Delta \sigma_p \)
  - Decreases from 81MPa (1 day) to 42MPa (16 days)
- Yield strength of pure Al: \( \sigma_{Al} \)
  - 20MPa
- Contribution from grain boundary strengthening: \( \Delta \sigma_b \)
  - 5MPa (for a grain size of 200\( \mu \)m)
- Contribution from solid solution strengthening: \( \Delta \sigma_s \)
  - 1MPa (Fleischer's approach)
Modified Orowan Equation

\[ \Delta \sigma_p = M' \mu b \sqrt{f/d} \]

- Contribution of SiGe precipitates to the yield strength: \( \Delta \sigma_p \)
- Ratio between Taylor factor calculated from X-ray diffraction data (2.86) and Taylor factor calculated for f.c.c. metals with a random distribution of grain orientations (3.06): \( M' \)
- Shear modulus of the matrix: \( \mu \)
  - 26GPa
- Burgers vector: \( b \)
  - 0.284nm
- Volume fraction of SiGe precipitates: \( f \)
- Diameter of SiGe precipitates: \( d \)
TEM Volume Fraction Measurements

Al-0.55Si-2.02Ge (wt.%) aged at 160°C

Corrected for truncation and overlap.
Electrical Conductivity Measurements

Al-0.55Si-2.02Ge (wt.%) aged at 160°C

Electrical Conductivity (MSm⁻¹) vs. Aging Time (hrs)
Lattice Parameter Measurements

Al-0.55Si-2.02Ge (wt.%) aged at 160°C

Graph showing the lattice parameter (Å) over aging time (hrs).
SiGe Volume Fraction Determination

Al-0.55Si-2.02Ge (wt.%) aged at 160°C

- Transmission Electron Microscopy
- Electrical Conductivity Measurements
- Lattice Parameter Measurements
- Average Values

Precipitate Volume Fraction vs. Aging Time (hrs)
SiGe Precipitate Size Distribution

Al-0.55Si-2.02Ge (wt.%) aged at 160°C

1 day
d=5.0nm

16 days
d=14.4nm
Fracture Surface (SEM)

Room temperature tensile test performed with Al-0.55Si-2.02Ge (wt.%) in longitudinal direction after peak-aging at 160°C
Determination of Void Volume Fraction

Al-0.55Si-2.02Ge (wt.%) aged at 160°C

- 1 day (d=5.0nm)
- 2 days (d=8.8nm)
- 4 days (d=10.3nm)
- 8 days (d=11.5nm)
- 16 days (d=14.4nm)

True Strain

Volume Fraction of Voids (x1000)

$\varepsilon_c = 0.008$
$\varepsilon_c = 0.011$
$\varepsilon_c = 0.012$
$\varepsilon_c = 0.014$
$\varepsilon_c = 0.014$
Cavity Nucleation: Energy Criterion

- $\Delta E_{\text{el}} + \Delta E_{\text{s}} \leq 0$
  - Internal elastic energy of the precipitates: $\Delta E_{\text{el}}$
  - Energy increase in forming new surfaces: $\Delta E_{\text{s}}$

- $\varepsilon_c \geq \gamma(1 - \cos \Theta)/2\mu_p b \sin^2 \Theta$
  - Critical strain for cavity nucleation: $\varepsilon_c$
  - Precipitate-matrix interfacial free energy: $\gamma$
  - Geometrical correction factor: $\Theta$
  - Shear modulus of the precipitates: $\mu_p$
  - Burgers vector: $b$
Geometrical Correction Factor

- Internal surfaces are formed from only a small fraction of the particle-matrix interface.
- Elastic energy release occurs from the cylinder bounded by the cavity caps.
- Resulting correction factor: \((1 - \cos(\Theta))/4\sin^2(\Theta)\)
Coarsening Theory (Lifshitz & Wagner)

\[ r^3 - r_o^3 = 8\gamma Dc_o V_m^2(t-t_o)/9RT \]

- Average precipitate radius: \( r \)
- Initial average precipitate radius: \( r_o \)
- Precipitate-matrix interfacial free energy: \( \gamma \)
- Diffusivity of the solute species in the matrix: \( D \)
- Equilibrium concentration of the solute species: \( c_o \)
- Molar volume of the precipitate: \( V_m \)
- Time: \( t \)
- Time when coarsening commences: \( t_o \)
- Gas constant: \( R \)
- Temperature: \( T \)
Al-0.55Si-2.02Ge (wt.%) aged at 160°C

Precipitate-Matrix Interfacial Energy

Corrected for truncation and overlap.

\[ \gamma = 423 \text{mJm}^{-2} \]

(Average Radius) \( \bar{r} \) (nm)

Aging Time (hrs)
Evaluation of Energy Criterion

- $\varepsilon_c \geq 0.008$ independent of precipitate size
  - Precipitate-matrix interfacial free energy: 423mJm$^{-2}$
    - Estimated from coarsening data
  - Geometrical correction factor: 35°
    - Estimated from finite element calculations
  - Shear modulus of the SiGe precipitates: 49GPa
    - Determined through ultrasonic velocity measurements in the ternary alloy and in pure Al
    - Burgers vector: 0.284nm

- Experimental data between 0.008 and 0.014, but not independent of precipitate size
Cavity Nucleation: Stress Criterion

- $\sigma_c = 4.2\alpha\mu b\sqrt{\rho}$, $\rho = 5.1\varepsilon_c/db$
  - Critical stress: $\sigma_c$
  - Numerical parameter: $\alpha$
  - Shear modulus of the matrix: $\mu$
  - Burgers vector: $b$
  - Local dislocation density: $\rho$
  - Critical strain: $\varepsilon_c$
  - Precipitate diameter: $d$

$\varepsilon_c \geq \frac{\sigma_c^2d}{90\alpha^2\mu^2b}$
Evaluation of Stress Criterion

Al-0.55Si-2.02Ge (wt.%) aged at 160°C

Critical Strain for Cavity Nucleation

Experimental Data

Stress Criterion ($\sigma_c = \mu / 40$, $\alpha = 1/7$)

Precipitate Diameter (nm)
Mesh for Calculation of Elastic Stresses
Elastic Stress Distribution around SiGe
Ramberg-Osgood Relationship

- \( E \varepsilon = \sigma + \alpha \left( \frac{|\sigma|}{\sigma_Y} \right)^{n-1} \sigma \)
  - Young's modulus: \( E \)
  - Strain: \( \varepsilon \)
  - Stress: \( \sigma \)
  - Yield offset: \( \alpha \), defined in such a way that, when
    \[ \sigma = \sigma_Y, \ \varepsilon = (1 + \alpha) \frac{\sigma_Y}{E} \]
  - Hardening exponent: \( n \ (n \geq 1) \)

- Nonlinearity significant for \( \sigma > \sigma_Y \) (if \( n > 5 \))
Mesh for Calculation of Total Stresses
Total Stress Distribution around SiGe
Finite Element Calculations

![Graph showing the relationship between maximum stress at the interface (MPa) and precipitate diameter (nm).](image)

- **Elasticity**
- **Deformation Plasticity**

The graph illustrates how the maximum stress at the interface decreases as the precipitate diameter increases.
Conclusions

- Precipitation in Al-0.55Si-2.02Ge (wt.%) continues to occur up to 100hrs at 160°C
- Improvement of the yield strength prediction for this ternary alloy as a function of aging time
- Volume fraction of voids determined in this ternary alloy decreases with increasing diameter of the SiGe precipitates at a given true strain value
- Critical strain to nucleate cavities increases linearly with precipitate size in agreement with the stress criterion by Brown & Stobbs
- Finite element calculations are consistent with experimentally determined void volume fractions
Future Work

- Direct observation of voids around SiGe precipitates using TEM
  - Determination of void nucleation and void growth rates from measurements of areal void density and maximum void size as a function of strain for different aging times
  - Explanation of decreasing tensile ductility but increasing critical strain for cavity nucleation with increasing aging time
- Quantification of precipitate-matrix interfacial structure using atomic resolution TEM in order to calculate stresses associated with ledges and steps
Objective

The objective of this study is to determine the effect of texture in unrecrystallized and recrystallized modified 2519 on the precipitation of the Ω and Θ' phases in the T6 and T8 conditions, and the associated relationship with anisotropy of the mechanical properties. In addition, the effect of an applied stress (both elastic and plastic) on nucleation and growth of the strengthening precipitates will be examined.

Current Status

Solution heat treated and T6 samples of the Al-Cu-Mg-Ag alloy were aged under stress to study the effect on nucleation and growth of precipitates. Solutionized binary Al - 5 wt.% Cu samples were also aged under stress to study the effect in greater detail. This alloy was used as a model system because its microstructure is less complicated than that of the quaternary alloy. The microstructure was characterized by TEM and the volume fraction, number density and size of precipitates were measured.

Recent Findings

Quantitative microstructure analysis was carried out to investigate whether preferential nucleation or growth on certain habit planes occurs when aging is done under stress. Therefore, the volume fraction was determined separately for every precipitate habit plane and the angle between the precipitate and the direction of the applied load was measured. The value of the measured angle, α, between precipitate and the stress direction is subtracted from 90°. This means that precipitates with |90° - α| = 0° are perpendicular and those with |90° - α| = 90° are parallel to the stress direction. For solution heat treated samples it was found that the higher volume fractions of Θ' are parallel and the lower volume fractions are perpendicular to the stress direction when the applied stress is 40% of the room temperature yield stress. For Ω, the values
are randomly scattered over the whole spectrum of angles when the same amount of stress was applied.

It was supposed that there might be a threshold stress which has to be exceeded before a similar effect can be observed for Ω. Therefore, samples were aged under a higher stress. After aging under a stress equivalent to the room temperature yield stress, the higher volume fractions are observed parallel to the stress axis for both type of precipitates. The threshold stress for Ω in this alloy is found to be between 119 and 142 MPa for solution heat treated samples. No comparable effect was found for samples in the T6 condition at this stress, neither for the volume fractions, nor for the number density or the size of the precipitates. Samples of the solutionized binary Al - Cu alloy were also aged under a different stress to study the observed effect in greater detail. A similar effect was observed. The Θ' phase precipitates preferentially parallel to the stress axis. The threshold stress for Θ' is found to be between 16 and 19 MPa.

The following discussion will focus on the Al-Cu system because there are more data available on precipitation of Θ' than for Ω, but may also apply to the more complicated alloy. An externally applied stress during aging can affect nucleation of precipitates or their growth (coarsening) or both. If nucleation is affected, the number of particles perpendicular to the stress axis should be dramatically different than the number parallel to the axis. Likewise, if growth is affected, the precipitate length/thickness should be different parallel and perpendicular to the applied stress axis. A large effect on nucleation was observed since most of the Θ' precipitates are parallel to the stress axis. The following discussion will also argue that the growth and coarsening behavior should be affected, as well because of the presumed growth ledges on the plates.

A model developed by Dahmen and Westmacott suggests that the smallest Θ' precipitate (critical nucleus) is 2 unit cells and has a vacancy misfit (negative). Stobbs and Purdy have experimentally shown that 2 unit cells (or smaller) indeed have a vacancy-type misfit. A 2 unit cell Θ' precipitate fits into 3 unit cells of the Al-matrix with a misfit of -4.5 % (calculated from the lattice parameters). Based on previous work (i.e. δ' in Al-Li) a negative misfit with the matrix will result in nucleation of the precipitate in the compressed region of the matrix. Therefore, it is concluded that the plate shaped Θ' nuclei should preferentially nucleate parallel to the tensile stress axis, because it is this direction that is under compression. This contradicts
the results of Hosford and Agrawal. They found a higher density of θ' precipitates perpendicular to the tensile stress axis. (Nevertheless, it has to be noted that their single published micrograph (in a [310] orientation) is not conclusive and they did not carry out any quantitative analysis.) In contrast to their findings, Eto, et al., observed preferential precipitation parallel to the stress axis which is in agreement with our observations. It has to be noted that different aging temperatures were used.

Calculations from the lattice parameters indicate that O nuclei probably have an even larger vacancy type misfit and are therefore affected in the same way. The growth and coarsening behavior is probably affected as well because of the mechanism of thickening of a precipitate plate is by the movement of ledges. This has not been observed in the T6 samples so far but might show if the aging is carried out under a higher stress which exceeds the threshold stress for this condition.

**Milestones**

DSC measurements are planned to get more information about the sequence of precipitation and what type of precipitation stage is affected by the external stress. The question arises if precursor phases of θ' form during heat-up. Preliminary results of DSC experiments show no dissolution of any phase before precipitation starts at 188°C in the Al-Cu-Mg-Ag alloy (no stress applied). This might be different if aging is done under stress. More TEM investigations need to be done to find out how the misfit is affected by stress aging. The approach first used by Ashby and Brown will be applied.

TEM work will be done on the new material (C415 and C416). Creep samples were sent by Alcoa and the microstructure will be characterized concerning grain boundary precipitation and coarsening of the precipitate phases.

**Presentation Viewgraphs**

1. Title.
2. Objective.
3. Subtitle.
4. Background: Effect of external and internal stresses associated with second phase particles.

5. Background: Reason for preferential nucleation and consequences of preferential precipitation.

6. Alloy composition and processing.

7. Precipitate phases and morphology.

8. Mechanical properties at room temperature and elevated temperature in solution heat treated and T6 condition for different orientations.

9. Parameters used for heat treatment, aging and stress aging.

10. Creep sample geometry used for stress aging experiments.

11. Experimental procedure for microstructure characterization.

12. Volume fractions of precipitates in Al-Cu-Mg-Ag measured for T6, T8, solution heat treated and stress aged and T6 and stress aged. Higher volume fractions of Ω and Θ' are present in prestrained samples than in T6. Further nucleation of Ω occurs in stress aged T6 samples than in T6 alone. Higher volume fraction of Θ' form after aging of solution heat treated samples under higher stress (SHT*) compared to lower stress.

13. Number density of precipitates in Al-Cu-Mg-Ag measured for T6, T8, solution heat treated and stress aged and T6 and stress aged. A high number density of Ω and Θ' formed after aging under stress. A high number density of Θ' formed after aging of solution heat treated samples under higher stress (SHT*) compared to lower stress.

14. Precipitate diameter and thickness in Al-Cu-Mg-Ag were measured for T6, T8, solution heat treated and stress aged and T6 and stress aged. Ω shows higher thermal stability than Θ'. Θ' starts to grow very early, but considerable growth of Ω occurs after 1000 h aging.

15. Volume fractions were measured separately for each variant in Al-Cu-Mg-Ag, and the angle α between the precipitate and the stress axis was measured. α was subtracted from 90°. |90° - α| = 0° is perpendicular to the stress axis. |90° - α| = 90° is parallel to the stress axis. In solution heat treated and stress aged samples, preferential precipitation of Θ' occurs parallel to the stress axis. No effect was observed for Ω.

16. For higher stresses, preferential precipitation of both kinds of precipitates parallel to the stress axis was observed for solution heat treated Al-Cu-Mg-Ag samples.

17. In binary Al-Cu preferential precipitation of Θ' was also observed for solution heat treated and stress aged samples.
18. Microstructure of stress aged Al-Cu in TEM. Stress direction is indicated by arrows. Almost all precipitates are parallel to the stress axis.

19. The threshold stress for preferential precipitation in solution heat treated samples was determined. No effect was observed in T6 samples.

20. Microstructure of Al-Cu in TEM. Sample was heated with applied stress to 160°C in about 70 min. Precipitates are Θ⁰ and randomly distributed.

21. DSC curves show precipitation at temperatures higher than 160°C.

22. Schematic drawing showing the observed effect on microstructure in solution heat treated samples.

23. Definition of vacancy and interstitial type of misfit. Misfit predicted and observed for Θ⁰ precipitates.


25. Due to the negative (vacancy) misfit, Θ⁰ should nucleate in the compressed region of the matrix which is parallel to the external tensile stress axis. Growth should also be affected by an external stress because the elastic accommodation strain can be minimized by growth ledge heights which have the requisite number of Θ⁰ subunits.

26. Ω precipitate in HRTEM. Growth of plate shaped precipitates occurs by moving of the growth ledges.

27. Conclusions.
Effect of Texture and Precipitates on Mechanical Property Anisotropy of Al-Cu-Mg-X Alloys

B. Skrotzki, E. A. Starke, G. J. Shiflet

LA²ST Grant Review Meeting
NASA Langley Research Center
July 17-18, 1995
Objective

Determine the effect of texture in unrecrystallized and recrystallized modified 2519 on the precipitation of the $\Omega$ and $\Theta'$ phases in T6 and T8 conditions and the associated relationship with anisotropy of mechanical properties.

In addition, the effect of an applied stress on nucleation and growth of the strengthening precipitates will be examined.
The Effect of Stress on Nucleation and Growth of Precipitates in Al-Cu-Mg-X Alloys
Background

- Externally applied stresses and internal stresses associated with second phase particles can affect both nucleation of the precipitates and their growth.

- Coherency strains
  - usually stabilize single phase field

- Externally imposed strains
  - may change stability of a phase
  - may move solvus line either into the single phase or two phase region
Background

- Normal aging conditions (no applied or residual stresses) => equal number of platelets on all habit planes
- A strong texture results in anisotropy of properties and this can be enhanced or decreased by precipitates
- Preferential precipitation could produce anisotropy of strength properties => texture strengthening effect
- Preferential nucleation
  - precipitates have large coherency strains
  - strains being produced in the matrix due to volume difference between precipitate and matrix
# Alloy System

## Chemical Composition [wt.%]

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Cu</th>
<th>Mg</th>
<th>Mn</th>
<th>Ag</th>
<th>Zr</th>
<th>V</th>
<th>Fe</th>
<th>Si</th>
<th>Al</th>
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<tr>
<td>1*</td>
<td>5.00</td>
<td></td>
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<td></td>
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<tr>
<td>2</td>
<td>5.75</td>
<td>0.52</td>
<td>0.30</td>
<td>0.49</td>
<td>0.16</td>
<td>0.09</td>
<td>0.06</td>
<td>0.05</td>
<td>bal.</td>
</tr>
</tbody>
</table>

* high purity

Alloy 1: cast at Reynolds Metals Co. and rolled to 0.25” (6.3 mm) sheet

Alloy 2: cast at Alcoa Technical Center and rolled to 0.125” (3.2 mm) sheet
Precipitate Phases

Strengthening Phases

- Alloy 1: \( \Theta'' \), \( \Theta' \), \( \Theta \)
- Alloy 2: \( \Theta'' \), \( \Theta' \), \( \Theta \), \( \Omega \), \( \Omega' \), \( \Omega'' \), \( S' \)

Morphology

- \( \Theta' \): \{100\}_Al, plate shaped, \( \text{Al}_2\text{Cu} \)
- \( \Omega \): \{111\}_Al, plate shaped, \( \text{Al}_2\text{Cu} \)
- \( S' \): \{210\}_Al, lath shaped, \( \text{Al}_2\text{CuMg} \)
# Mechanical Properties

(completely recrystallized; random texture)

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Condition</th>
<th>Orientation</th>
<th>T [°C]</th>
<th>σ_y [MPa]</th>
<th>UTS [MPa]</th>
<th>A [%]</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>SHT</td>
<td>RD</td>
<td>RT</td>
<td>120.1</td>
<td>274.5</td>
<td>30.8</td>
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<tr>
<td>1</td>
<td>SHT</td>
<td>RD</td>
<td>163</td>
<td>168.8</td>
<td>-</td>
<td>-</td>
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<tr>
<td>2</td>
<td>SHT</td>
<td>RD</td>
<td>RT</td>
<td>167.0</td>
<td>408.8</td>
<td>24.3</td>
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<tr>
<td>2</td>
<td>SHT</td>
<td>45°</td>
<td>RT</td>
<td>181.2</td>
<td>405.2</td>
<td>24.2</td>
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<tr>
<td>2</td>
<td>SHT</td>
<td>TD</td>
<td>RT</td>
<td>169.5</td>
<td>402.8</td>
<td>24.2</td>
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<tr>
<td>2</td>
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<td>RD</td>
<td>163</td>
<td>244.4</td>
<td>-</td>
<td>-</td>
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<tr>
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<td>T6</td>
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<td>RT</td>
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<td>535.4</td>
<td>12.1</td>
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<tr>
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<td>T6</td>
<td>45°</td>
<td>RT</td>
<td>471.1</td>
<td>520.5</td>
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</tr>
<tr>
<td>2</td>
<td>T6</td>
<td>TD</td>
<td>RT</td>
<td>481.4</td>
<td>526.7</td>
<td>12.0</td>
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<tr>
<td>2</td>
<td>T6</td>
<td>RD</td>
<td>163</td>
<td>366.7</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
Heat Treatment

- SHT (#1: 1h/540°C, #2: 1h/520°C)
- T6 (T8) @ 163°C
- Stress aging* @ 160°C
  - 40% of $R_{p0.2}(RT)$
    - 69 MPa for #2 (SHT)
    - 191 MPa for #2 (T6)
  - tapered samples
    - various stresses in one sample

* load was applied to SHT samples before heating
Sample Geometry

\[ \sigma = \text{const.} \quad \sigma \neq \text{const.} \]

\( t = \text{sheet thickness} \)
Microstructure Characterization

- TEM (Philips EM 400T, U = 120 kV)
- HRTEM (JEOL 4000 EX, U = 400 kV)
- Volume fraction, number density and size of precipitates
- CBED => foil thickness
Stereology

- Higher volume fractions of $\Omega$ and $\Theta'$ in prestrained samples
- Further nucleation of $\Omega$ after aging of T6 samples
- Higher volume fraction of $\Theta'$ after aging of SHT samples under higher stress
Stereology

- High number density of $\Omega$ and $\Theta'$ after SHT and T6 and aging under stress
- High number density of $\Theta'$ in SHT samples after aging under higher stress
Stereology

- $\Omega$ shows higher thermal stability than $\Theta'$
- $\Theta'$ starts to grow early
- Considerable growth of $\Omega$ occurs after 1000 h aging

![Graph showing particle diameter and thickness for Al-5.75 Cu-0.52 Mg-0.49Ag under different heat treatments and aging conditions.](image)
Stereology

- Preferential precipitation of $\Theta'$ parallel to stress axis after SHT and aging under stress

UVA Dept. of Materials Science
Stereology

- Preferential precipitation of $\Omega$ and $\Theta'$ parallel to stress axis after SHT and aging under higher stress
  $\Rightarrow$ threshold stress

**Graph:**
- Al-5.75 Cu-0.52 Mg-0.49Ag
- 100 h creep @ 160°C Tapered Sample
  $\sigma = \sigma_y$

**Legend:**
- $\Omega$ (h)  
- $\Omega$ (l)
- $\Theta'$ (h)
- $\Theta'$ (l)
Al - 5 wt.% Cu

100 h creep @ 162°C
σ = 28% σ_y = 33 MPa
Tapered Sample

Θ' (h)
Θ' (l)
Al - 5 wt.% Cu

- SHT + 100 h @ 162°C, 33.4 MPa (= 28 % R_{p0.2})

[001]_{\alpha}

UVA Dept. of Materials Science
Threshold Stress

- Critical stress for SHT samples:
  - $\Omega$: $120 < \sigma_c < 140$ MPa
  - $\Theta'$: $16 < \sigma_c < 19$ MPa

- No effect observed in T6 samples
Al - 5 wt.% Cu

- Creep sample heated with applied stress ($\sigma = 75.4$ MPa) from 20°C to 160°C in ~70 min.
Al - Cu - Mg - Ag

Curve 1: DSC
Sample Weight: 27.420 mg
AlCuMgAg repeat

Heat Flow (mW)

Temperature (°C)

Onset 187.93 °C
Peak 237.12 °C
AH 10.37 J/g
Peak 415.71 °C
Onset 430.16 °C

from 20 to 500
Ramp 15.8 °C/m
Time: 0.6 min Rate: 6.0 °C/min

Donglian Li
PERKIN-ELMER
7 Series Thermal Analysis System
Tue Jun 27 10:19:35 1995

UVA Dept. of Materials Science
Al - Cu

- Large effect on nucleation
- Precipitates are parallel to applied stress
Discussion

- Model by Dahmen and Westmacott suggests that smallest Θ' precipitate (critical nucleus) is 2 unit cells which would have vacancy misfit (negative). Stobbs and Purdy have experimentally shown that 2 unit cells or smaller have vacancy-type misfit.

- Based on previous work (e.g. δ') a negative misfit with the matrix will result in nucleation in compressed region of the matrix.
Crystallographic Model
Discussion

- Plate shaped $\Theta'$ nuclei should preferentially nucleate parallel to stress axis.
- Contradicts results (tensile stress) of Hosford and Agrawal (Met. Trans. 6A (1975) 487) but is in agreement with results of Eto, et al. (Acta Metall. 26 (1978) 499)
- During growth the growth ledge height should have the requisite number of $\Theta'$ subunits (half unit cells) to minimize the elastic accommodation strain.
Al - 5.75 Cu - 0.52 Mg - 0.49 Ag

SHT + 1000 h @ 161°C/ 69 MPa (= 40% $R_{p0.2}$)
Conclusion

- $\Omega$ and $\Theta'$ are preferentially oriented parallel to the applied stress.
- Large effect on nucleation. $\Theta'$ nuclei have negative misfit which is accommodated in compressed matrix.
- Growth ledge height minimize the elastic accommodation strain.
Progress achieved in this project will be reported elsewhere.
Project #11  Computer Modeling Environment Enhanced Fatigue Crack Propagation in Light Aerospace Alloys

Edward Richey III and Richard P. Gangloff

Objective

The objective of this research is to develop the methods and understanding needed to incorporate time and loading wave-form dependent environmental effects on fatigue crack propagation (FCP) in NASA FLAGRO. The research seeks to test the Wei and Landes linear superposition model and an interpolative model, and to suggest improvements to these models based on an improved understanding of the time dependencies which affect environmental fatigue crack propagation (EFCP) in structural alloys. In particular Ti-6Al-4V is studied in the mill annealed (MA), extra low interstitial (ELI) condition due to its complex, time-dependent environmental fatigue behavior.

Current Status

The experimental work for Ti-6Al-4V (MA, ELI) in NaCl was completed during this reporting period. Computer-automated constant $\Delta K$ and $\Delta K$ decreasing experiments were employed to determine the effect of frequency on $da/dN$. Monotonically rising load tests were conducted to determine the stress corrosion cracking (SCC) susceptibility of the alloy. These data were employed to assess computer models of EFCP rates developed in previous reporting periods. The graduate student, Mr. Edward Richey III, has completed and defended his Master's of Science thesis, and is beginning his doctoral studies in the Department of Materials Science and Engineering at UVa. A paper reporting the complete findings of this research is planned for journal publication. A computer program and documentation will be provided to Dr. R.G. Forman (NASA-Johnson Space Center) for possible incorporation into NASA-FLAGRO.

Recent Results

Monotonic loading experiments were conducted in order to determine the stress corrosion cracking susceptibility of Ti-6Al-4V (MA, ELI) in a 3.5% NaCl solution. ELI grade Ti-6Al-4V
exhibits a much greater resistance to SCC than standard grade Ti-6Al-4V. The threshold stress intensity for crack initiation in 3.5% NaCl solution (K\(_{th}\)) is between 48 and 55 MPa\(\sqrt{m}\) depending on loading rate. For moist air, K\(_{HC}\) was found to be 78 MPa\(\sqrt{m}\), slightly higher than the value for standard grade Ti-6Al-4V. ELI grade Ti-6Al-4V exhibits a much slower da/dt than the standard grade, of order 10\(^{-5}\) mm/sec or less. ELI grade Ti-6Al-4V exhibits environment assisted cracking (EAC), albeit at a very high K level.

Corrosion fatigue experiments were conducted on Ti-6Al-4V (MA,ELI) in moist air and a 3.5% NaCl solution for \(\Delta K\) values in the range of 5 to 25 MPa\(\sqrt{m}\), frequencies in the range of 0.03 to 40 Hz, and a stress ratio of 0.1. A 3.5% NaCl solution is detrimental, and enhances fatigue crack growth rate two to four fold compared to cracking in moist air. The ELI grade alloy does not exhibit the same frequency dependence as standard grade Ti-6Al-4V. The ELI grade alloy does not exhibit the frequency "crossover" effect exhibited by the standard grade. Instead, da/dN increases as frequency increases; da/dN is proportional to \(f^{0.1}\) to \(f^{0.2}\).

Nominal crack closure levels were recorded during all fatigue experiments. Crack closure levels were consistent with plasticity induced closure and the Forman Equation with closure. Crack closure levels were independent of environment. For \(\Delta K\) value below 10 MPa\(\sqrt{m}\), closure levels increased as \(\Delta K\) decreased. This could be due to roughness or oxide induced closure. For \(\Delta K\) levels above 10 MPa\(\sqrt{m}\), there is no roughness or oxide induced closure.

Both linear superposition and interpolative models were utilized to represent EFCP rates in standard and ELI grade Ti-6Al-4V in 3.5% NaCl. The linear superposition model proved effective for standard grade Ti-6Al-4V for \(\Delta K\) values where K\(_{max}\) exceeds K\(_{th}\) (23 MPa\(\sqrt{m}\)) and frequencies greater than 1 Hz. The linear superposition model is not effective for ELI grade Ti-6Al-4V due to the increased SCC resistance of this alloy. The interpolative model was effective in modeling the effect of frequency in both standard and ELI grade Ti-6Al-4V when the frequencies where data were interpolated are within the establishing data base.

The computer models developed during this research are reasonably effective in representing EFCP rates, and can be incorporated in NASA FLAGRO. The programs are available on a FORTRAN 77 program disk in executable form, along with the source code. Given the complexity of EFCP in a variety of aluminum, iron, nickel and titanium-based alloys,
this program is only a first step in developing the necessary interpolative and predictive capabilities in NASA-FLAGRO; substantial additional work is required.

Presentation Viewgraph Captions

1. Title.

2. Problem Statement. The problem is that coupling an aggressive environment with cyclic plastic deformation can significantly reduce the fatigue life of an aerospace structure; these environmental effects, however, have not been incorporated in NASA FLAGRO.

3. Objective and current work. The general objective of the research is to develop an effective method for estimating environmental effects on fatigue crack propagation for use in NASA FLAGRO.

4. Da/dN versus ΔK data for Ti-6Al-4V in moist air and a 3.5% NaCl solution for a frequency of 5 Hz, and R = 0.1. In moist air da/dN is a function only of ΔK and R. In an aggressive environment such as NaCl, da/dN is a function not only of ΔK and R, but also loading frequency and hold time.

5. Summary of equation used by the computer programs to relate da/dN to ΔK. Five different crack growth rate equations are available including the Forman Equation with and without crack closure, the Paris Equation, the Hyperbolic Sine Equation, and the Sigmoidal Equation.

6. Summary of the method proposed to interpolate trends in environmental fatigue behavior using an approach similar to that reported by Haritos et al. Trends are interpolated by relating equation constants to load-time characteristics using empirical relationships. The form of these relationships is selected based on interpolative models discussed in the literature, and experimental results generated for Ti-6Al-4V (MA,ELI).

7. Summary of equations used to interpolate crack growth rate-equation constants based on load characteristics. The load characteristics are stress ratio, hold time and frequency. The form of these relationships depends on the number of data sets fit to a crack growth rate equation, and the form of the basic crack growth rate equation.

8. Summary of the multiple power law model developed during this research. The purpose of the multiple power law model is to develop a computer program which allows the user to fit a multiple power law equation to da/dN versus ΔK data.
9. Plot of \( \frac{da}{dN} \) versus \( \Delta K \) for Ti-6Al-4V (MA,ELI) in moist air and a 1.0% NaCl solution for \( R = 0.4 \) and a frequency of 5 Hz. The alloy exhibits a complex \( \frac{da}{dN} \) versus \( \Delta K \) behavior in both moist air and aqueous chloride, as exhibited by the multiple slope transitions. The power law segments were determined using the multiple power law model developed during this research.

10. Chemical composition and mechanical properties for Ti-6AlV (MA,ELI). In all environment assisted cracking experiments, compact tensions specimens machined in the L-T orientations were utilized. A 3.5% NaCl solution served as the corrosive environment, and specimens were held at a fixed electrode potential of -500 mV_{SCE}.

11. Summary of the method utilized during the constant CMOD rate tests which were conducted to determine the stress corrosion cracking susceptibility of Ti-6Al-4V (MA,ELI). All experiments were conducted in CMOD control with crack length calculated using the DCPD method, and CMOD measured by a clip gauge. Also shown in a schematic of the environmental cell utilized during the experiments.

12. Summary of the results for the constant CMOD rate experiments conducted for Ti-6Al-4V (MA,ELI). \( K_{JC} \) was found to be 78 MPa\(\sqrt{m} \), slightly higher than \( K_C \) for standard grade Ti-6Al-4V. ELI grade Ti-6Al-4V exhibits a greater resistance to stress corrosion cracking than standard grade Ti-6Al-4V, with a \( K_{TH} \) between 48 and 55 MPa\(\sqrt{m} \) depending on loading rate. ELI grade Ti-6Al-4V also exhibits a slower \( \frac{da}{dt} \) than standard grade.

13. Plot of load versus CMOD for Ti-6Al-4V (MA,ELI) in a 3.5% NaCl solution. The specimen was loaded monotonically to a stress intensity level of 40 MPa\(\sqrt{m} \) in 65 minutes. The load was then held constant for 140 hours at this level. A load drop of 0.1 kN would have been noticeable, and corresponded to 0.2 mm of crack extensions. The load did not drop, thus \( \frac{da}{dt} \) is less than \( 5 \times 10^{-7} \) mm/sec. Shown on the graph is the literature-reported \( K_{TH} \) value for standard grade Ti-6Al-4V.

14. Plot of load versus CMOD for Ti-6Al-4V (MA,ELI) in moist air and 3.5% NaCl for various loading rates. Notice that there are small sharp decreases in the load at a constant CMOD for the NaCl experiments. The air data do not show decreases in load at a constant CMOD. Since for two of the experiments, the loading rates were identical and only the environment varies, environment assisted cracking occurs in Ti-6Al-4V (MA,ELI), but only at high stress intensities. (Note that the fatigue precrack length was longer for the experiment represented by o, compared to the two experiments with equal and shorter crack lengths, represented by □ and +.)

15. Conclusions for environmental fatigue experiments conducted on Ti-6Al-4V (MA,ELI). 3.5% NaCl is detrimental, enhancing fatigue crack growth rates two to fourfold compared to moist air. ELI grade Ti-6Al-4V does not exhibit the frequency "crossover" effect for the range of frequencies and \( \Delta K \) levels utilized during this research (0.03 to 40 Hz and 5 to 25 MPa\(\sqrt{m} \), respectively) due to the increased stress corrosion cracking resistance of
the alloy. Instead, ELI grade Ti-6Al-4V exhibits a mild frequency dependence for $\Delta K$ values between 12.5 and 25 MPa√m where $da/dN$ is proportional to $f^{0.1\text{ to } 0.2}$. The closure levels exhibited by Ti-6Al-4V (MA,ELI) are consistent with plasticity induced closure.

16. Description of the frequency "crossover" effect exhibited by $\alpha + \beta$ titanium alloys in aqueous chloride solutions. The "crossover" is a change in the ordering of $da/dN$ as a function of frequency at a transition $\Delta K$ level ($\Delta K_{\text{SCC}}$) associated with the onset of "cyclic" stress corrosion cracking. Below $\Delta K_{\text{SCC}}, da/dN$ decreases as frequency decreases perhaps due to passive film stability. Above $\Delta K_{\text{SCC}}, da/dN$ increases as frequency decreases perhaps due to hydrogen embrittlement mechanisms.

17. Da/dN versus $\Delta K$ for Ti-6Al-4V (MA,ELI) in moist air, 1.0% NaCl and 3.5% NaCl for various frequencies and a stress ratio of 0.1. Fatigue crack growth rates are enhanced in the aqueous chloride compared to moist air, and are only mildly frequency dependent.

18. Plot of $da/dN$ versus frequency for Ti-6Al-4V (MA,ELI) for constant $\Delta K$ values of 12.5 and 25 MPa√m. For both constant $\Delta K$ values, $da/dN$ generally increases mildly as frequency increases for frequencies in the range 0.03 to 40 Hz. For both $\Delta K$ levels where $K_{\text{max}}$ was less than $K_{\text{TH}}$, $da/dN$ was proportional to $f^{0.1\text{ to } 0.2}$. The dashed line indicates the type of trend that would be observed if the alloy/environment system exhibited the frequency crossover effect.

19. Plot of $\Delta K_{\text{SCC}}$ versus frequency for standard and ELI grade Ti-6Al-4V. The literature data shown for standard grade Ti-6Al-4V are consistent with a $K_{\text{TH}}$ of 23 MPa√m. Since the ELI grade alloy exhibits a much higher $K_{\text{TH}}$ value, the EFCP data for standard grade Ti-6Al-4V were linearly scaled to generate the trend lines shown for the ELI grade Ti-6Al-4V. "Cyclic" stress corrosion cracking does not occur in the ELI grade Ti-6Al-4V for the range of frequencies and applied stress intensity ranges used during this study due to the increased SCC resistance. Since "cyclic" SCC did not occur, the alloy did not exhibit the frequency "crossover" effect. For example, for applied $\Delta K$ values below 25 MPa√m, "cyclic" SCC should not occur for frequencies below 200 Hz, five times faster than the fastest frequency where data were collected during this research, 40 Hz.

20. Plot of $K_{\text{cl}} / K_{\text{max}}$ versus crack length for Ti-6Al-4V (MA,ELI) in moist air and a 3.5% NaCl solution for a constant $\Delta K$ value of 15 MPa√m, stress ratio of 0.1, and frequency of 5 Hz. Crack closure levels exhibited by Ti-6Al-4V (MA,ELI) were independent of environment. The closure levels are consistent with plasticity induced closure modeling, and the associated Forman Equation with closure. For $\Delta K$ values less than 10 MPa√m, crack closure increased as $\Delta K$ decreased. This could be due to roughness or oxide induced closure. For $\Delta K$ values greater than 10 MPa√m, there is apparently no contribution from roughness or corrosion debris/oxide induced closure.

21. Summary of conclusions for the computer models developed during this research. Linear superposition is effective in modeling environment enhanced FCP rates for material/environment systems where the alloy is extremely sensitive to stress corrosion...
cracking. For standard grade Ti-6Al-4V, linear superposition is effective for $\Delta K$ values where $K_{\text{max}}$ exceeds $K_{\text{TH}}$. Linear superposition is not effective for ELI grade Ti-6Al-4V due to the increased SCC cracking resistance of the alloy. The interpolative model is effective in modeling FCP rates when the load characteristics where data are interpolated are within the establishing data base. Specifically, the effects of frequency and stress ratio are reasonably modeled in standard and ELI grade Ti-6Al-4V.

22. Linear superposition predictions of literature-reported corrosion fatigue crack growth rates in standard grade Ti-6Al-4V in 3.5% NaCl. The linear superposition model is effective for $\Delta K$ values where $K_{\text{max}}$ exceeds $K_{\text{TH}}$ (23 MPa$\sqrt{\text{m}}$) and loading frequencies greater than 1 Hz.

23. Fitted and interpolated data for standard grade Ti-6Al-4V in a 3.5% NaCl solutions using the Forman Equation with closure. The logarithmic form of the frequency dependence was selected.

24. Fitted and interpolated data for ELI grade Ti-6Al-4V in a 3.5% NaCl solution using the Forman Equation with closure. The crack growth rate equation constants were related to the frequency using the $f^\alpha$ dependence. $\alpha$ was specified as 0.1.

25. Summary of the University of Virginia Fatigue Analysis Software. The linear superposition, interpolative, and multiple power law models are each contained in the DOS executable program UVAFAS.EXE. The source code is also included on the disk. The program was written using FORTRAN 77. This program will be provided to Dr. R.G. Forman at the NASA-Johnson Space Center.

26. Summary of future work required to improve and generalize the linear superposition model. The question has been posed: Can one calculate $\frac{da}{dt}$ vs $K$ from measured $\frac{da}{dN}$ vs $\Delta K$ data; this issue will be investigated. This calculation can be completed currently using linear regression, provided that $\frac{da}{dt}$ is well represented by a simple Paris equation, and the fatigue load-wave form is a simple square function. Saff et al. advocate this approach. The problem of calculating $\frac{da}{dt}$ becomes nonlinear when other functions are necessary. It is erroneous to assume that $\frac{da}{dt}$ is necessarily independent of loading frequency, because frequency affects crack tip strain rate and crack tip strain rate may affect $\frac{da}{dt}$, for example, as predicted by film rupture modeling.

27. Schematic diagram showing typical dependencies of EFCP rate ($\frac{da}{dN}$ vs $\Delta K$) on loading frequency. For the NaCl solution case, steels often exhibit $\frac{da}{dN}$ proportional to the inverse square root of frequency above a critical stress intensity range, while aluminum and titanium alloys often exhibit $\frac{da}{dN}$ proportional to frequency raised to a power on the order of -0.1 for aluminum alloys and both +0.1 and -0.8 for titanium alloys. The exact exponent depends on frequency and $\Delta K$, as discussed with regard to the crossover effect. The simple linear superposition predicts that this power is -1.0, if a frequency independent value of $\frac{da}{dt}$ exists at a given applied $K$ level.
28. Summary of the feasibility and usefulness of calculating $\frac{da}{dt}$ from $\frac{da}{dN}$. The model, if developed with sufficient mathematical sophistication, should be able to calculate a set of $\frac{da}{dt}$ vs $K$ data from $\frac{da}{dN}$ vs $\Delta K$, however, the model will always predict $\frac{da}{dN}$ proportional to $f^{-1.0}$ if $\frac{da}{dt}$ is assumed to be frequency-independent. This result is not broadly accurate for aluminum, titanium alloys and even steels. These various frequency-dependent $\frac{da}{dN}$ data sets will produce a nonunique $\frac{da}{dt}$ vs $K$ relationship. That is, frequency-dependent $\frac{da}{dN}$ vs $\Delta K$ data may produce a unique set of frequency-dependent $\frac{da}{dt}$ vs $K$. This result may be mechanistically meaningful and should be pursued.

29. Summary of the method proposed to express $\frac{da}{dt}$, derived from $(\frac{da}{dN})_{SCC}$ vs $\Delta K$ data, as a function of frequency for use in superposition modeling.
Computer Modeling Environment Enhanced Fatigue Crack Propagation in Light Aerospace Alloys

Edward Richey, III and Richard P. Gangloff
LA2ST Program Review
NASA Langley Research Center
July 18, 1995
**Problem Statement**

- Coupling an aggressive environment with cyclic plastic deformation can reduce fatigue life of an aerospace alloy.

- Environmental effects have not been incorporated in computer life prediction codes such as NASA FLAGRO.
General Objective

Develop an effective method to estimate environmental effects on fatigue crack propagation in light aerospace alloys for use in NASA FLAGRO.

Computer Modeling Goals

- Develop program implementing Wei and Landes linear superposition model.
- Develop curve-fitting based interpolative model extending approach used in NASA FLAGRO.
- Develop computer program to fit multiple power laws to fatigue data.
- Apply models to titanium alloys.

Environment Assisted Cracking (EAC) of Ti-6Al-4V

- Literature analysis for standard grade Ti-6Al-4V.
- Determine stress corrosion susceptibility of Ti-6Al-4V in 3.5% NaCl solution.
- Determine effect of loading frequency on fatigue crack propagation in Ti-6Al-4V in 3.5% NaCl.

Output

- Program for NASA.
- Understanding of corrosion fatigue behavior of Ti-6Al-4V.
Fatigue Crack Propagation in Ti-6Al-4V (MA, ELI)

In moist air, \( \frac{da}{dN} \) is a function of \( \Delta K \) and \( R \).

In NaCl, \( \frac{da}{dN} \) is a function of \( \Delta K, R, \tau, f \).
**Crack Growth Rate Equations**

- **Forman With Closure**

  \[
  \frac{da}{dN} = \frac{C (1 - f)^n \Delta K^n \left(1 - \frac{\Delta K_{th}}{\Delta K}\right)^p}{(1 - R)^q \left(1 - \frac{\Delta K}{(1 - R) K_C}\right)^q}
  \]

- **Forman Without Closure**

  \[
  \frac{da}{dN} = \frac{C \Delta K^n (1 - R)^m \left(1 - \frac{\Delta K_{th}}{\Delta K}\right)^p}{\left(1 - R - \frac{\Delta K}{K_C}\right)^q}
  \]

- **Paris**

  \[
  \frac{da}{dN} = C \Delta K^n
  \]

- **Hyperbolic Sine (SINH)**

  \[
  \log \left(\frac{da}{dN}\right) = C_1 \sinh \left( C_2 \left[ \log (\Delta K) + C_3 \right] \right) + C_4
  \]

- **Sigmoidal**

  \[
  \frac{da}{dN} = e^{B \left(\frac{\Delta K}{\Delta K_{th}}\right)^P \left[\ln \left(\frac{\Delta K}{\Delta K_{th}}\right)\right]^Q \left[\ln \left(\frac{\Delta K_C}{\Delta K}\right)\right]^D}
  \]
Interpolative Model

○ FLAGRO is primarily accurate for fatigue in moist air.

○ Need: estimate time and load-wave form-dependent trends in fatigue behavior for corrosive environments.

○ Interpolate trends by relating equation constants to load time characteristics.

○ If η is an equation constant then: \( \eta = f(f, \tau, R) \)
  ○ \( f = \) frequency
  ○ \( \tau = \) hold time
  ○ \( R = \) stress ratio

○ Form of relationships based on interpolative models discussed in literature and results generated for Ti-6Al-4V (MA,ELI).

○ Interpolation function are empirical, not based on mechanistic understanding.
Interpolation Functions

- If one data set entered:

\[ \eta = a_1 \log \left( \frac{1}{f} \right) + 1 \]

- If two data sets entered using Forman Equation:

\[ \eta = a_1 \log \left( \frac{1}{f} \right) + 1 \times a_2 \log \left( \frac{\tau + 1}{0.1} \right) \]

- If three data sets entered using Paris, SINH, or Sigmoidal:

\[ \eta = a_1 \log \left( \frac{1}{f} \right) + 1 \times a_2 \log (1 - R) \times a_3 \log \left( \frac{\tau + 1}{0.1} \right) \]

- Log [(1/f)+1] dependence can be replaced with \( f^\alpha \), \( \alpha \) is a user specified constant defined by experiment or theory.
Multiple Power Law Model

○ Goal: Develop computer program to fit a multiple power law equation to \( \frac{da}{dN} \) vs. \( \Delta K \) data.

○ User has three methods for fitting equation to data:
  ○ Define slope offset between two successive segments.
  ○ Define number of segments.
  ○ Define desired number of segments and approximate \( \Delta K \) transitions.

○ Current methods of fitting equation are subjective.
Ti-6Al-4V (MA,ELI) in Moist Air and 1.0% NaCl, R = 0.4, f = 5 Hz

Graph showing da/dN (mm/cycle) vs. ΔK (MPa√m) for Ti-6Al-4V (MA,ELI) in Moist Air and 1.0% NaCl with R = 0.4 and f = 5 Hz.
Materials Specifications for Ti-6Al-4V (MA, ELI)

Chemical Composition (wt. %)

<table>
<thead>
<tr>
<th>Al</th>
<th>V</th>
<th>C</th>
<th>N</th>
<th>Fe</th>
<th>O</th>
<th>H</th>
<th>Y</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>6.17</td>
<td>4.33</td>
<td>0.025</td>
<td>0.011</td>
<td>0.19</td>
<td>0.12</td>
<td>0.0055</td>
<td>0.0005</td>
<td>balance</td>
</tr>
</tbody>
</table>

Mechanical Properties

<table>
<thead>
<tr>
<th>Yield Strength MPa (ksi)</th>
<th>Tensile Strength MPa (ksi)</th>
<th>Elongation Percent (%)</th>
<th>Reduction in Area (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>940 (136)</td>
<td>982 (142)</td>
<td>13</td>
<td>30.2</td>
</tr>
</tbody>
</table>

EAC Experiments

- Compact tension (CT) specimens machined in L-T orientation.
- 3.5% NaCl solution used, specimen held at fixed electrode potential of -500 mV_SCE.
Constant CMOD Rate EAC Tests

- Constant CMOD rate experiments conducted to determine stress corrosion susceptibility of Ti-6Al-4V (MA, ELJ).
- All tests conducted in CMOD control, crack length calculated by DCPD, CMOD measured with clip gage.
Results and Conclusions for Constant CMOD Rate Tests

- $K_{JIC}$ is 78 MPa$\sqrt{m}$, slightly higher than standard Ti-6Al-4V.

- $K_{TH}$ for Ti-6Al-4V (MA,ELI) is between 48 and 55 MPa$\sqrt{m}$ in 3.5% NaCl depending on loading rate.
  - $K_{TH} = 55$ MPa$\sqrt{m}$ (CMOD Rate = $3 \times 10^{-5}$ mm/sec)
  - $K_{TH} = 48$ MPa$\sqrt{m}$ (CMOD Rate = $4.7 \times 10^{-5}$ mm/sec)

- Ti-6Al-4V (MA,ELI) exhibits a greater resistance to SCC than standard grade Ti-6Al-4V (MA).

- ELI Ti-6Al-4V exhibits a slower (da/dt) than standard Ti-6Al-4V (of order $10^{-5}$ mm/sec or less).
Load increased monotonically to stress intensity level of 40 MPa√m in 65 minutes, held for 140 hours.

Load drop of 0.1 kN (25 lbs) would have been noticeable and corresponded to 0.2 mm of crack growth.

Load did not drop, thus da/dt is less than 5 x 10^{-7} mm/sec.
Results for Constant CMOD Rate Tests

There are sharp decreases in load at constant CMOD for NaCl experiments.

Air data do not show sharp decreases in load at constant CMOD.

Since loading rates were identical and only environment changed, EAC does occur in Ti-6Al-4V (MA,ELI), only at high K.
Conclusions: Environmental Fatigue Behavior of Ti-6Al-4V (MA, ELI)

- 3.5% NaCl solution is detrimental and enhances fatigue crack propagation (FCP) rates two to four fold compared to moist air.

- ELI Ti-6Al-4V does not exhibit the frequency "crossover" effect for ΔK value less than 25 MPa√m for frequencies between 0.03 and 40 Hz due to increased SCC resistance.

- ELI Ti-6Al-4V exhibits a mild frequency dependence for ΔK values 12.5 and 25 MPa√m; da/dN is proportional to \( f^{0.1 \text{ to } 0.2} \).

- Closure levels exhibited by ELI Ti-6Al-4V are consistent with plasticity induced closure, and are independent of environment.
Frequency "Crossover" in $\alpha + \beta$ Titanium Alloys

- $\alpha + \beta$ titanium alloys in aqueous chloride solutions exhibit frequency "crossover".

- "Crossover" is a change in ordering of $\text{da/dN}$ as a function of frequency at a transition $\Delta K$ level.

- Transition $\Delta K$ level ($\Delta K_{SCC}$) is associated with onset of "cyclic" SCC.

- Below $\Delta K_{SCC}$, $\text{da/dN}$ decreases as frequency decreases perhaps due to passive film stability.

- Above $\Delta K_{SCC}$, $\text{da/dN}$ increases as frequency decreases perhaps due to hydrogen embrittlement mechanisms.
Fatigue Results for Ti-6Al-4V (MA, ELI)

Ti-6Al-4V (MA, ELI)
L-T Orientation
R = 0.1

Constant $\Delta K = 15 \text{ MPa } \sqrt{\text{m}}$

Moist Air
- $5 \text{ Hz}$
- $3.5\% \text{ NaCl, } -500 \text{ mV}_{\text{SCE}}$
- $5 \text{ Hz}$

$\frac{\text{da}}{\text{dN}}$ (mm/cycle)

$\Delta K$ (MPa $\sqrt{\text{m}}$)

- Moist Air
- $5 \text{ Hz}$
- $3.5\% \text{ NaCl, } -500 \text{ mV}_{\text{SCE}}$
- $40 \text{ Hz}$
- $1.0\% \text{ NaCl, } -500 \text{ mV}_{\text{SCE}}$
- $5 \text{ Hz}$
Results do not exhibit frequency "crossover" effect.

- \( \frac{da}{dN} \) increases as frequency increases for \( 0.03 < f < 40 \) Hz.

- \( \frac{da}{dN} \) is proportional to \( f^{0.1 \ to \ 0.2} \) for each \( \Delta K \) where \( K_{\max} < K_{TH} \).
"Cyclic" SCC does not occur in ELI grade for the range of frequencies tested due to increased SCC resistance.

Since "cyclic" SCC does not occur, frequency "crossover" effect is not exhibited.

For $\Delta K$ values below 25 MPa$\sqrt{m}$, "cyclic" SCC does not occur for frequencies below 200 Hz.

Fastest frequency where data were collected during this study is 40 Hz.
Crack closure levels are independent of environment.

Closure levels are consistent with plasticity induced closure ($K_{cl}/K_{max} = 0.3$) and with Forman Equation.

Crack closure levels increase as $\Delta K$ decreases for $\Delta K$ values less than 10 MPa$\sqrt{m}$, could be due to roughness or oxide induced closure.

At $\Delta K$ values $> 10$ MPa$\sqrt{m}$, there is no roughness or oxide induced closure.
Conclusions for Computer Modeling

- Linear superposition is effective in modeling environment enhanced FCP rates for material/environment systems where the alloy is sensitive to SCC.

- Linear superposition is effective for standard Ti-6Al-4V for $\Delta K$ values where $K_{\text{max}}$ exceeds $K_{\text{TH}}$.

- Linear superposition does not work for ELI Ti-6Al-4V due to SCC resistance of alloy.

- Interpolative model effectively models FCP rates when the load characteristics are within the establishing data base.

- Interpolative model effectively models the effect of $f$ and $R$ in standard and ELI grade Ti-6Al-4V.

- Models contained in the program UVAFAS.EXE are effective in modeling environment enhanced FCP rates, and can be incorporated in NASA FLAGRO.
Superposition is effective for $\Delta K$ values where $K_{\text{max}}$ is greater than $K_{\text{TH}}$ (~23 MPa√m) and $f > 1$ Hz.

Superposition is not effective for ELI Ti-6Al-4V due to increased SCC resistance ($K_{\text{TH}}$ ~ 48 to 55 MPa√m).
Application of Interpolative Model to Standard Ti-6Al-4V

Forman Equation With Closure

Actual and fitted data for $f = 1$ Hz.

Actual and fitted data for $f = 10$ Hz.

Actual and interpolated data, $f = 5$ Hz.

Effect of frequency on $da/dN$. 
Application of Interpolative Model to ELI Ti-6Al-4V

Actual and fitted data for $f = 5 \text{ Hz}$.  
Actual and interpolated data for $f = 40 \text{ Hz}$.  
Predicted $da/dN$ for $0.01 < f < 100 \text{ Hz}$.  

Ti-6Al-4V (MA, ELI)  
$1.0\%$ NaCl, $-500 \text{ mV}_{SCE}$  
$f = 5 \text{ Hz}, R = 0.1$  

Ti-6Al-4V (MA, ELI)  
$3.5\%$ NaCl, $-500 \text{ mV}_{SCE}$  
$f = 40 \text{ Hz}, R = 0.1$  

Ti-6Al-4V (MA, ELI)  
$3.5\%$ NaCl, $-500 \text{ mV}_{SCE}$  
$R = 0.1, L-T$  

Model Predictions:  
- $0.01 \text{ Hz}$  
- $0.1 \text{ Hz}$  
- $1 \text{ Hz}$  
- $10 \text{ Hz}$  
- $100 \text{ Hz}$
University of Virginia Fatigue Analysis Software

Edward Richey, III
Allen W. Wilson
Richard P. Gangloff
Department of Materials Science and Engineering
University of Virginia
Future Work Regarding Linear Superposition Model

- Include capability to calculate \( \frac{da}{dt} \) (K) from \( \frac{da}{dN} \) versus \( \Delta K \).

- May improve model by generating a set of \( \frac{da}{dt} \) data more reflective of crack tip strain rates during fatigue.

- Solve for \( \frac{da}{dt} \) in following equation:

\[
\frac{da}{dN} = \int_{\tau}^{\tau} \frac{da}{dt} [K(t)] \, dt
\]

- Linear regression utilized if \( \frac{da}{dt} \) is represented by Paris Equation and \( K(t) \) is represented by square wave with \( R=0 \).

\[
\frac{da}{dt} = C \, K^n
\]

\[
\left( \frac{da}{dN} \right)_{SCC} = C \, \tau \, K_{\text{max}}^n \, (1 - \alpha)
\]

\( \tau = \) period of loading cycle\n\( \alpha = \) fraction of load cycle at \( K_{\text{max}} \)

- Problem is complicated and non-linear when other wave forms and equations are used.
Relationship between \( \frac{da}{dN} \) and Frequency

- \( \frac{da}{dN} \propto f^{-0.5} \) (Steel in NaCl)

- \( \frac{da}{dN} \propto f^{-0.1} \) (Ti in NaCl)

- Linear Superposition predicts \( \frac{da}{dN} \propto f^{-1} \)
Feasibility of Calculating $da/dt$ From $da/dN$

- Assume $da/dN \propto f^{-\alpha}$, $-1.0 < \alpha < 0.1$

- Will $da/dt$ calculated from $da/dN$ be physically meaningful and useful?
  
  - Model will predict $da/dN$ proportional to $f^{-1}$, with $da/dt$ not dependent on frequency.
  
  - Model may be effective if $da/dt$ is treated as a function of frequency.
  
  - Model not effective for material/environment systems where the dependence of $da/dN$ on frequency changes at a critical frequency or $\Delta K$ level.
**Relating $da/dt$ to Frequency**

- Dimensional analysis shows:
  \[
  \frac{da}{dN} = \frac{da}{dt} f^{-1}
  \]

- If $da/dt$ is independent of frequency:
  \[
  \frac{da}{dN} \propto f^{-1}
  \]
  as predicted by simple linear superposition model.

- If $da/dt$ is a function of frequency:
  \[
  \frac{da}{dt} \propto f^\alpha \quad 0 < \alpha < 1.5
  \]
  \[
  \frac{da}{dN} \propto \frac{da}{dt} f^{-1} \propto f^{(\alpha - 1)}
  \]

- Must define frequency dependence of $da/dt$:
  \[
  \frac{da}{dt} = C K^n f^\alpha
  \]
  
  **Film rupture modeling:**
  \[
  \frac{da}{dt} \propto f^m \quad \text{where } m \approx 0.5
  \]
APPENDIX I: GRANT PUBLICATIONS  (January 1 to June 30, 1995)


APPENDIX II: GRANT PRESENTATIONS  (January 1 to June 30, 1995)


APPENDIX III: GRANT PROGRESS REPORTS (January, 1988 to June, 1995)


APPENDIX IV: LA³ST GRANT REVIEW MEETING AGENDA

FINAL AGENDA

SIXTH ANNUAL NASA-UVa LA³ST MEETING

NASA-Langley Research Center
Hampton, Virginia

Day 1: Monday, July 17, 1995

1:00-1:15 pm  Dennis L. Dicus: Welcome. Edgar A. Starke and Richard P. Gangloff: LA³ST program status.

1:15-2:00  "Al-Si-Ge Alloy Development"; Holger Koenigsmann and E.A. Starke.

2:00-2:45  "Effects of Texture and Microstructure on the Anisotropic Yield Strength of Wide Panel Aluminum Alloy Extrusions"; Mark T. Lyttle and John A. Wert

2:45-3:00  Break

3:00-3:40  "Effect of Texture and Precipitates on Mechanical Property Anisotropy of Al-Cu-Mg-X Alloys"; Birgit Skrotzki, G.J. Shiflet and E.A. Starke.


Day 2: Tuesday, July 18, 1995


10:00-10:15  Break

10:15-11:00  "Hydrogen Interactions and Their Correlation to the Hydrogen Embrittlement Susceptibility of Al-Li-Cu-Zr Alloys"; Stephen W. Smith and J.R. Scully.

11:00-11:20  "SCC Initiation in Advanced Al-Li-Cu Alloys; J.R. Scully and G.E. Stoner.


11:50-1:15 pm  Lunch

1:15-2:15  Group discussion between UVa and LaRC participants on the health of the LA2ST Grant, the LA2ST-HSCT program merger, and directions for future NASA/UVa research.
DISTRIBUTION LIST

1-4  Mr. D. L. Dicus  
Contract Monitor  
Metallic Materials Branch, MS 188A  
NASA Langley Research Center  
Hampton, VA  23681-0001

5-6*  NASA Scientific and Technical Information Facility  
P. O. Box 8757  
Baltimore/Washington International Airport  
Baltimore, MD  21240

7  Mr. Neil Price  
Grants Officer, M/S 126  
NASA Langley Research Center  
Hampton, VA  23681-0001

8  Dr. Darrel R. Tenney  
Materials Division  
NASA Langley Research Center  
Hampton, VA  23681-0001

9  Dr. Charles E. Harris  
Mechanics of Materials Branch  
NASA Langley Research Center  
Hampton, VA  23681-0001

10  Mr. W. Barry Lisagor  
Metallic Materials Branch  
NASA Langley Research Center  
Hampton, VA  23681-0001

11  Mr. T.W. Crooker  
Code RM  
NASA Headquarters  
Washington, DC  20546

12  Dr. Robert S. Piascik  
Mechanics of Materials Branch  
NASA Langley Research Center  
Hampton, VA  23681-0001
Mr. W. Brewer  
Metallic Materials Branch, MS 188A  
NASA Langley Research Center  
Hampton, VA 23681-0001

Mr. Thomas T. Bales  
Metallic Materials Branch, MS 188A  
NASA Langley Research Center  
Hampton, VA 23681-0001

Mr. John Wagner/Ms. Cynthia Lach  
Metallic Materials Branch, MS 188A  
NASA Langley Research Center  
Hampton, VA 23681-0001

Dr. William F. Bates  
Lockheed Aeronautical Systems Co.  
86 South Cobb Drive  
Marietta, GA 30063-0648

Dr. Alex Cho  
Reynolds Metals Co.  
4th and Canal Street  
Richmond, VA 23261

Mr. E.A. Colvin  
Alcoa Technical Center  
Route 780, 7th Street Road  
Alcoa Center, PA 15069

Dr. L.M. Karabin  
Alcoa Technical Center  
Route 780, 7th Street Road  
Alcoa Center, PA 15069

Dr. Ravi Kahandal  
McDonnell Douglas Aerospace  
Mail Stop 36-90  
3855 Lakewood Blvd.  
Long Beach, CA 90846
Mr. Fred Casey
Space Transportation Systems Division
Rockwell International
Dept. 289 MC/AC56
12214 Lakewood Blvd.
Downey, CA 90241

E.A. Starke, Jr.; MS&E

R.P. Gangloff; MS&E

G. J. Shiflet; MS&E

G.E. Stoner; MS&E

J.A. Wert; MS&E

J.R. Scully; MS&E

C.T. Herakovich; CE and AM

S.S. Kerbel; Clark Hall

SEAS Preaward Administration Files

Mr. Gwyn Faile
Code ED 24
Marshall Space Flight Center
Huntsville, AL 35812

Mr. Brian McPherson
Code ED 24
Marshall Space Flight Center
Huntsville, AL 35812

Dr. William E. Quist
Boeing Aerospace and Electronics
Aerospace Group
Mail Stop GH-CJ
P.O. Box 3707
Seattle, WA 98124
37  Dr. Howard G. Nelson  
NASA-Ames Research Center  
EEM: 213-3  
Moffett Field, CA  94035

38  Dr. R.G. Forman  
Mail Code ES-5  
NASA-L.B. Johnson Space Flight Center  
Houston, TX  77058

39  Professor A.K. Noor  
Center for Computational Structures Technology  
NASA Langley Research Center  
Hampton, VA  23681-0001

40  Prof. A.K. Ghosh  
Department of Materials Science and Engineering  
University of Michigan  
2102 Dow Building  
Ann Arbor, MI  48109-2136

41  Dr. D. Ferton  
Pechiney Centre de Recherches  
De Voreppe  
B.P. 27 -- 38340 Voreppe  
FRANCE

42  Dr. John Papazian  
Grumman Aerospace & Electronics  
Mail Stop A02-026  
Bethpage, NY  11714-3582

43  Dr. Richard Lederich  
McDonnell Douglas Aircraft Company  
Mail Stop 111-1041  
P.O. Box 516  
St. Louis, MO  36166

44  Dr. Alan Hopkins  
Senior Research Metallurgist  
Research Institute  
The University of Dayton  
300 College Park  
Dayton, OH  45469-0172
Dr. Frances Hurwitz  
Ceramic Composites  
NASA Lewis Research Center  
21000 Brookpark Road  
Cleveland, OH 44135

Dr. Malcolm Ozelton  
Manager, Metallic & Ceramic Materials  
B-2 Division  
Northrop Corporation  
8900 E. Washington Blvd.  
T241/GK  
Pico Rivera, CA 90660-3737

Dr. S. Sampath  
Technical Center  
Federal Aviation Administration  
Atlantic City International Airport,  
NJ 08405

Dr. James Staley  
Alcoa Laboratories  
Alcoa Center, PA 15069

Dr. Jeffrey Waldman  
Code 6063  
Naval Air Development Center  
Warminster, PA 18974

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