A Progress Report
NASA-LaRC Grant NAG-1-745


NASA-UVA LIGHT AEROSPACE ALLOY AND STRUCTURES TECHNOLOGY PROGRAM (LA^3ST)

Submitted to:
National Aeronautics and Space Administration
Langley Research Center
Hampton, Virginia 23665

Attention:
Mr. Richard J. Siebels
Grants Officer, MS 126

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

Submitted by:
Richard P. Gangloff
Professor

Report No. UVA/528266/MS91/108
June 30, 1991

DEPARTMENT OF MATERIALS SCIENCE

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.
A Progress Report

January 1, 1991 to June 30, 1991

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

Report No. UVA/528266/MS91/108
June 30, 1991
NASA-UVA LIGHT AEROSPACE ALLOY AND STRUCTURES TECHNOLOGY PROGRAM

LA²ST

Program Director:
Richard P. Gangloff

Co-principal Investigators:
Richard P. Gangloff
John K. Haviland
Carl T. Herakovich
Walter D. Pilkey
Marek-Jerzy Pindera
John R. Scully
Edgar A. Starke, Jr.
Glenn E. Stoner
Earl A. Thornton
Franklin E. Wawner, Jr.
John A. Wert

NASA-LaRC Grant Monitor:
Dennis L. Dicus
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NASA-UVA LIGHT AEROSPACE ALLOY AND
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EXECUTIVE SUMMARY

The NASA-UVa Light Aerospace Alloy and Structures Technology Program (LA^2ST) has achieved a high level of activity in 1991, with projects being conducted by graduate students and faculty advisors in the Departments of Materials Science, Civil Engineering, and Mechanical and Aerospace Engineering at the University of Virginia. This work is funded by the NASA-Langley Research Center under Grant NAG-1-745. Here, we report on the progress achieved between January 1 and June 30, 1991. Sections for each project reproduce the visual aids that were presented at the Second Annual LA^2ST Grant Review Meeting held at the Langley Research Center on July 9th and 10th of 1991.

The general objective of the LA^2ST Program is to conduct interdisciplinary graduate student research on the performance of next generation, light weight aerospace alloys, composites and associated thermal gradient structures in close collaboration with Langley researchers. Specific technical objectives are established for each research project. We aim to produce relevant data and basic understanding of material behavior and microstructure, new monolithic and composite alloys, advanced processing methods, new solid and fluid mechanics analyses, measurement advances and a pool of educated graduate students.

The accomplishments presented in this report are highlighted as follows:

- Four research areas are being actively investigated, including: (1) Mechanical and Environmental Degradation Mechanisms in Advanced Light Metals and Composites, (2) Aerospace Materials Science, (3) Mechanics of Materials and Composites for Aerospace Structures, and (4) Thermal Gradient Structures.

- Fifteen research projects are being conducted by 9 PhD and 3 MS graduate students, 11 faculty members, and 3 research associates from three departments in the Engineering School at UVa. Each project is planned and executed in conjunction with a specific branch and technical monitor at LaRC.
The undergraduate program, initiated with four students working at LaRC during the summer of 1990, will not be conducted in 1991 because of a lack of qualified applicants. Recruiting efforts will be intensified for 1992. Two undergraduates are assisting in LA$^2$ST research projects at UVa.

Three new graduate students were recruited into the LA$^2$ST Program.

Reporting accomplishments between January and June of 1991 include 6 journal or proceedings publications and 5 presentations at technical meetings, bringing the LA$^2$ST totals since 1986 to 32 and 39, respectively.

Research on environmental fatigue of advanced aluminum alloys and metal matrix composites shows that intrinsic rates of fatigue crack propagation in unrecrystallized sheet of Al-Li alloy 2090 are similarly and strongly enhanced by moist air and aqueous NaCl relative to vacuum. Vintage III sheet and plate (peak aged) exhibit similar fatigue behavior for moist air, however, cracking in the plate is more rapid than expected compared to the behavior of older 2090. These and literature results reflect environmental, texture and grain/subgrain morphology effects on the proportions of slip plane, cleavage and subboundary cracking. (Program 1)

Research on localized corrosion and stress corrosion cracking of Al-Li-Cu-X alloys in aqueous solutions employed electrochemical studies of the primary phases in alloy 2090 and results from time-to-failure testing to further establish a criteria for rapid SCC by anodic dissolution. Failure requires the specimen to be at an electrode potential above $E_{br}$ for $T_1$ and below $E_{br}$ for the matrix phase. Within this regime, $T_1$ dissolution is believed to be the primary mode of crack growth. Similar experiments have been initiated on three compositional variations of Weldalite (alloy X2095) in collaboration with Reynolds Metals. (Program 5)

Research on the environmental sensitivity of Al-Li-X alloys demonstrated that high stress corrosion cracking resistance is developed in 8090 with several Zn additions up to 1 wt%, and after relatively longer aging times at 160°C. Unfortunately, fracture toughness is degraded by increased aging time due to the formation of precipitates at grain and subgrain boundaries. Zn additions to alloy 8090 promote precipitation of the S' phase during artificial aging. This work is cosponsored by the Alcoa Technical Center. (Program 6)
Research on hydrogen interactions with Al-Li alloys was initiated at UVa with the goal of unambiguously demonstrating the deleterious effect of dissolved hydrogen on mechanical properties, independent of complications due to aqueous environmental and microstructural embrittlement. This work will focus on microstructural aspects of hydrogen trapping, as a means of explaining and mitigating embrittlement. (Program 7)

Research on the fracture behavior of Al-Cu-Li-In alloys demonstrated that the initiation and growth fracture toughnesses of Vintage III alloy 2090 and 2090 + In increase with decreasing temperature to cryogenic levels for both plane strain and plane stress conditions. High angle boundary delamination increases at cryogenic temperatures and contributes to this effect, independent of whether the fracture mode is transgranular slip plane cracking (2090) or intersubgranular (2090 + In). Subboundary precipitation occurs in the In bearing composition after short aging times at 160°C and degrades fracture toughness. (Program 3)

Research on the fracture toughness of Weldalite™ 049 was initiated to examine the temperature dependencies of $K_{IC}$ and tearing resistance for two model microstructures (either T1 or 6' plus T1) produced in a separate study at UVa. The alloy was characterized and specimens were prepared for fracture experiments in July. (Program 4)

Research on elevated temperature fracture of PM Al-Fe-Si-V alloy 8009 demonstrated that intermediate temperature embrittlement is not caused by the moist air environment. At the temperature (175°C) where cracking resistance is minimal, equal low $K_{IC}$ values and tearing modules are observed for loading in moist air and dynamic high vacuum. Creep crack growth occurs at similar rates in both air and vacuum. The strong and deleterious effect of high stress ratio on fatigue crack propagation is similarly observed for air and vacuum, based on collaborative experiments with Mechanics of Materials researchers. Work was initiated to conduct a detailed fractographic study of the complex fracture surfaces in 8009 (high magnification, stereo pairs and matching crack surfaces). (Program 2)

Research on Ti alloy matrix-SiC fiber reinforced composites has concluded the analysis of the elevated temperature reaction kinetics of several fibers and various titanium matrices, including Ti-1100 and Beta-21S. Work on thermal cycling of the Ti-1100/SCS-6 composite demonstrated that oxide related surface cracking and oxygen penetration into the titanium matrix reduced the uniaxial tensile properties of the material. (Program 8)
Research on quantifying the spatial distribution and homogeneity of microstructure applied the previously developed Dirichlet tessellation technique to the problem of characterizing the oxide particle distribution in high temperature aluminum alloy sheet produced by PM methods. For processing similar to that used by LaRC, the results demonstrate that the hot working temperature and amount of forging reduction have less influence on the oxide particle distribution than total hot working strain.

(Program 9)

Research on the yielding of SCS-6/Ti-15-3 metal matrix composite under biaxial loading has produced test results for [0\textdegree] and [\pm45\textdegree] SiC/Ti tubes. Good correlation between experimental data and the predictions of micromechanical modeling theory was obtained for loading prior to the onset of fiber-matrix debonding.

(Program 11)

Research on cryogenic tankage and shell structure analytical modeling demonstrated that superplastically formed corrugated hat-section stringers could replace integrally machined stringers in a large tank structure. Using equivalent beam properties, for both stringers and frames, the allowable compression loads on a large diameter tank were determined and shown to be adequate. A new program was initiated on the design of shell structures subject to thermal loads. Benchmark tests for the SPAR elements used in the COMET program were compared with exact solutions and with elements for other finite element programs.

(Program 12)

Research on the thermoviscoplastic response of high temperature alloys has used the Bodner-Partom constitutive model, implemented in a finite element program, to show that nickel based superalloy panels may display initially higher yield stresses due to strain rate effects. As transient temperatures approach elevated levels, the analyses show that yield stress and stiffness rapidly degrade, and pronounced plastic deformation occurs. Experimental studies of locally heated panels are in progress to characterize the thermal-structural behavior of initially imperfect panels and to provide data for validation of constitutive models and finite element analyses.

(Program 13)
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 at the University of Virginia. This work emphasized the mechanical and corrosion behavior of light aerospace alloys, particularly Al-Li-Cu based compositions, in aggressive aerospace environments. Results are documented in three progress reports.

In the Fall of 1988 this program was increased in scope to incorporate materials science research at UVa on the development and processing of advanced aerospace materials. Funding was provided by the Metallic Materials and Mechanics of Materials Branches. 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. The Department of Civil Engineering and the Department of Mechanical and Aerospace Engineering at UVa participated in this expanded program. With this growth, the NASA-UVa Light Aerospace Alloy and Structures Technology Program (LA²ST) was initiated within the School of Engineering and Applied Science at UVa.

The first progress report for the LA²ST program was published in August of 1989. Research efforts in solid mechanics were in a state of infancy and were not represented at that time. Since then, graduate students have been recruited into the structural mechanics programs and several new projects have been initiated. Since July of 1989, the LA²ST program has operated with full participation from all faculty and graduate students, as outlined in the last three progress reports and two grant renewal proposals. The first two-day Grant Review Meeting was held in June of 1990 at the Langley Research Center, with over 20 faculty and graduate students from UVa and 1 faculty and graduate student from VPI participating.

The Second Annual NASA-UVa LA²ST Program Meeting was held at LaRC in July of 1991. This report summarizes the presentations at this meeting and emphasizes the
progress of LA$^2$ST research during the period from January 1st to June 30, 1991.

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 fully integrated. The thermal and chemical effects of aerospace environments are particularly important to material performance. Nationally, academic efforts in these areas are limited. The NASA-UVa Light Aerospace Alloy and Structures Technology Program addresses these needs.

LA$^2$ST Program

As detailed in the original proposal$^{[6]}$ and affirmed in the most recent renewal document$^{[12]}$, faculty from the Departments of Materials Science, Mechanical and Aerospace Engineering, and Civil Engineering at UVa are participating in the LA$^2$ST 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, and measurement advances. A major product of the LA$^2$ST 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 LA$^2$ST 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 Aerospace Structures,

Thermal Gradient Structures.

Fifteen specific research projects are ongoing within these four areas and are reported here. These projects involve eleven faculty, three research associates and twelve graduate students. The majority of the graduate students are at the doctoral level (9 of 12) and all are citizens of the United States. 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.

A primary goal of the LA²ST Program is to foster interdisciplinary research. To this end, many of the research projects share a common focus on the same next generation light alloys and composites, and on light and reusable aerospace structures which will be subjected to aggressive terrestrial and space environments. Emphasis is placed on both cryogenic and elevated temperature conditions, with severe thermal gradients typical of tankage structures.

Organization of Progress Report

This progress report first provides LA²ST Program administrative information including statistics on the productivity of faculty and student participants, a history of current and graduated students, refereed archival publications and a list of ongoing projects with NASA and UVa advisors. Twelve sections summarize the technical accomplishments of each research project, emphasizing the period from January 1st to June 30th of 1991. Each report contains an abstract and the slides/viewgraphs from the presentation at the Second LA²ST Program Review Meeting. Appendices document grant sponsored publications, conference participation, abstracts of technical papers published during this reporting period, and citations of all LA²ST Progress Reports.
References


SUMMARY STATISTICS

Table I documents the numbers of students and faculty who participated in the LA$^2$ST Program, both during this reporting period and since the program inception in 1986. Academic and research accomplishments are indicated by the degrees awarded, publications and presentations. Specific graduate students and research associates who participated in the LA$^2$ST Program are named in Tables II and III, respectively.

**TABLE I: LA$^2$ST Program Statistics**

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<td>11.</td>
<td>C. L. Lach</td>
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<td>17</td>
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The following papers were based on research conducted under LA2ST Program support, and were refereed and published in the archival literature.


COMPLETED PROJECTS

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
   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: Materials Science
   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: Materials Science
   NASA-LaRC Contact: D.L. Dicus (Metallic Materials)
   Start Date: June, 1987
   Completion Date: December, 1990
   Cosponsor: 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 at VPI
   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: Materials Science
   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: PhD
   UVa Department: Mechanical and Aerospace Engineering
   NASA-LaRC Contact: Drs. 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
ADMINISTRATIVE PROGRESS

Faculty Participation

Professor Edgar A. Starke, Jr., Dean of the School of Engineering and Applied Science, has initiated a project on novel high strength aluminum alloys. Professor J.K. Haviland has retired from the Department of Mechanical and Aerospace Engineering, but will continue to interact with Professor Pilkey on LA²ST research.

Brochure

A brochure was prepared in March of 1991 to advertise the LA²ST program. Our aim is to nationally distribute this information to Universities and industries in the aerospace materials and mechanics communities. Two hundred copies have been circulated to date. All layout, printing and mailing costs were paid by the School of Engineering and Applied Science, and by the Materials Science Department at UVa.

Student Recruitment

The LA²ST Program continues to recruit the best graduate students entering the participating Departments, and in sufficient numbers to achieve our education and research objectives (Table III). Three new graduate students; Mr. Douglas Wall (PhD, Materials Science), Mr. Stephen Smith (PhD, Materials Science) and Ms Karen McCarthy (MS, Mechanical and Aerospace Engineering); were recruited into the LA²ST Program during the Spring of 1991. Professor Wert is interviewing students to fill a single opening for his program on superplasticity.

In April of 1990, the LA²ST Program was increased in scope to include four undergraduate engineering students working on summer projects at NASA-LaRC. During the summer of 1990, four students, recruited from North Carolina State and California Polytechnic State University, worked in the Metallic Materials (3 students) and Mechanics of Materials Branches (1 student) at the Langley Research Center. In conjunction with this expansion, two UVa undergraduates are currently working on ongoing LA²ST projects in the Mechanical and Aerospace Engineering Department under Professor Thornton’s supervision.
These students are focusing on aerospace design issues.

During this reporting period, Professor Gangloff attempted to recruit four undergraduate students nationwide for LaRC employment during the summer of 1991; this effort failed and LA$^2$ST summer students were not hired. The newly developed program brochure was mailed in early April to over 100 materials science, mechanical, aerospace and civil engineering departments. Additionally, ten department chairmen and senior faculty were contacted with the aim of recruiting high quality undergraduates. While eight students responded, none met the program qualifications (viz., rising senior or some materials/mechanics course work, 3.0 or better grade point average, 10 to 12 week summer availability). Many of the students who responded would not be eligible for graduate studies in engineering at UVa.

Efforts to recruit undergraduates for the summer of 1992 will be intensified. The brochure will be mailed in December of 1991 and selected departments will be contacted to identify the best rising seniors. Each UVa participant in LA$^2$ST is encouraged to identify qualified undergraduates, nationwide. NASA-LaRC staff can similarly assist in the initial recruiting process. Experience to date indicates that the undergraduate program is important and must be exploited. The entrance requirements are not unreasonable, provided that our recruiting activities are broad based.

Complementary Programs at UVa

The School of Engineering and Applied Science at UVa has targeted materials and structures research for aerospace applications as an important area for broad future growth. The LA$^2$ST Program is an element of this thrust. Several additional programs are of benefit to LA$^2$ST work.

The Board of Visitors at UVa awarded SEAS an Academic Enhancement Program Grant in the area of Light Thermal Structures. The aim is to use University funds to seed the establishment of a world-class center of excellence which incorporates several SEAS Departments. This program is lead by Professor Thornton and should directly benefit NASA.

The Light Metals Center has existed within the Department of Materials Science at
UVa for the past several years under the leadership of Professor H.G.F. Wilsdorf. A Virginia Center for Innovative Technology Development Center in Electrochemical Science and Engineering was established in 1988 with Professor G.E. Stoner as Director. Professors Pilkey, Thornton and Gangloff are conducting research under NASA-Headquarters Grant sponsorship to examine "Advanced Concepts for Metallic Cryo-thermal Space Structures"[2,3]. Research within this program is complementing LA²ST studies.

References


CURRENT PROJECTS

MECHANICAL AND ENVIRONMENTAL DEGRADATION MECHANISMS IN ADVANCED LIGHT METALS AND COMPOSITES

1. ENVIRONMENT-ENHANCED FATIGUE OF ADVANCED ALUMINUM ALLOYS AND METAL MATRIX COMPOSITES
   Faculty Investigator: R.P. Gangloff
   Graduate Student: Donald Slavik; PhD Candidate
   UVa Department: Materials Science
   NASA-LaRC Contact: D.L. Dicus (Metallic Materials)
   Start Date: September, 1989
   Anticipated Completion Date: December, 1992
   Program # 1

2. ELEVATED TEMPERATURE FRACTURE OF AN ADVANCED RAPIDLY SOLIDIFIED, POWDER METALLURGY ALUMINUM ALLOY
   Faculty Investigator: R.P. Gangloff
   Graduate Student: William C. Porr, Jr.; PhD candidate
   UVa Department: Materials Science
   NASA-LaRC Contact: C.E. Harris (Mechanics of Materials)
   Start Date: January, 1988
   Anticipated Completion Date: June, 1992
   Program # 2

3. ELEVATED TEMPERATURE CRACK GROWTH IN ADVANCED ALUMINUM ALLOYS
   Faculty Investigator: R.P. Gangloff
   Research Associate: Dr. Yang Leng
   Graduate Student: None
   UVa Department: Materials Science
   NASA-LaRC Contact: C.E. Harris (Mechanics of Materials)
   Start Date: March, 1989
   Completion Date: July, 1991
   Program # 2
4. 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: Materials Science  
NASA-LaRC Contacts: W.B. Lisagor (Metallic Materials)  
J.C. Newman (Mechanics of Materials)  
Start Date: June, 1987  
Anticipated Completion Date: May, 1992  
Program # 3

5. THE EFFECT OF TEMPERATURE ON THE FRACTURE TOUGHNESS OF WELDALITE™  
Faculty Investigator: R.P. Gangloff  
Graduate Student: Cynthia L. Lach; MS candidate and NASA-LaRC employee  
UVa Department: Materials Science  
NASA-LaRC Contacts: W.B. Lisagor (Metallic Materials)  
Start Date: August, 1990  
Anticipated Completion Date: May, 1992  
Program # 4

6. MEASUREMENTS AND MECHANISMS OF LOCALIZED CORROSION IN AL-LI-CU ALLOYS  
Faculty Investigator: G.E. Stoner  
Graduate Student: Douglas Wall; PhD candidate  
UVa Department: Materials Science  
NASA-LaRC Contact: D.L. Dicus (Metallic Materials)  
Start Date: April, 1991  
Completion Date: To be determined  
Cosponsor: Alcoa  
Program # 5

7. EFFECT OF ZINC ADDITIONS ON THE ENVIRONMENTAL STABILITY OF ALLOY 8090  
Faculty Investigator: Glenn E. Stoner  
Graduate Student: Raymond J. Kilmer; PhD candidate  
Department: Materials Science  
NASA-LaRC Contact: W.B. Lisagor (Metallic Materials)  
Start Date: September, 1989  
Anticipated Completion Date: December, 1992  
Cosponsor: Alcoa  
Program # 6
8. HYDROGEN INTERACTIONS IN ALUMINUM-LITHIUM ALLOYS
   Faculty Investigator: John R. Scully
   Graduate Student: Stephen W. Smith; PhD Candidate
   Department: Materials Science
   NASA-LaRC Contact: W.B. Lisagor and D.L. Dicus (Metallic Materials)
   Start Date: April, 1991
   Anticipated Completion Date: To be determined
   Cosponsor: Virginia CIT
   Program #7

AEROSPACE MATERIALS SCIENCE

9. INVESTIGATION OF THE EFFECT OF THERMAL EXPOSURE ON THE
   MECHANICAL PROPERTIES OF TITANIUM/SiC COMPOSITES
   Faculty Investigator: F.E. Wawner
   Graduate Student: Douglas B. Gundel; PhD candidate
   UVa Department: Materials Science
   NASA-LaRC Contact: D.L. Dicus and W.B. Brewer (Metallic Materials)
   Start Date: April, 1991
   Anticipated Completion Date: To be determined
   Program #8

10. QUANTITATIVE CHARACTERIZATION OF THE SPATIAL DISTRIBUTION OF
    PARTICLES IN MATERIALS: APPLICATION TO MATERIALS PROCESSING
    Faculty Investigator: John A. Wert
    Graduate Student: Joseph Parse; PhD candidate
    UVa Department: Materials Science
    NASA-LaRC Contact: D.R. Tenney (Materials Division)
    Start Date: September, 1988
    Anticipated Completion Date: December, 1991
    Program #9
11. PROCESSING AND SPF PROPERTIES OF WELDALITE™ SHEET
   Faculty Investigator: John A. Wert
   Graduate Student: To be named
   UVa Department: Materials Science
   NASA-LaRC Contact: T. Bayles (Metallic Materials)
   Start Date: September, 1991
   Anticipated Completion Date: To be determined
   Program #9

12. THE DEVELOPMENT OF LOW ALLOY HIGH STRENGTH ALUMINUM ALLOYS
   Faculty Investigator: E.A. Starke, Jr.
   Research Associate: Dr. A.K. Mukhopadhyay
   Graduate Student: None
   UVa Department: Materials Science
   NASA-LaRC Contact: W.B. Lisagor (Metallic Materials)
   Start Date: June, 1991
   Completion Date: June, 1992
   Program # 10

MECHANICS OF MATERIALS FOR AEROSPACE STRUCTURES

13. INELASTIC RESPONSE OF METAL MATRIX COMPOSITES UNDER BIAXIAL LOADING
   Faculty Investigators: Carl T. Herakovich and Marek-Jerzy Pindera
   Research Associate: Dr. Farshad Mirzadeh
   Graduate Student: Mr. Clifford J. Lissenden, PhD Candidate
   UVa Department: Civil Engineering
   NASA-LaRC Contact: W.S. Johnson (Mechanics of Materials)
   Start Date: September, 1990
   Anticipated Completion Date: September, 1993
   Program # 11
14. SHELL STRUCTURES ANALYTICAL MODELING
   Faculty Investigators: W.D. Pilkey and J.K. Haviland
   Graduate Student: Karen McCarthy; MS candidate
   UVa Department: Mechanical and Aerospace Engineering
   NASA-LaRC Contact: Dr. M.J. Shuart (Aircraft Structures)
   Start Date: April, 1991
   Anticipated Completion Date: To be determined
   Program # 12

15. EXPERIMENTAL STUDY OF THE NONLINEAR VISCOPLASTIC RESPONSE
    OF HIGH TEMPERATURE STRUCTURES
    Faculty Investigator: Earl A. Thornton
    Graduate Student: Marshall F. Coyle
    UVa Department: Mechanical and Aerospace Engineering
    NASA-LaRC Contact: James H. Starnes, Jr. (Aircraft Structures)
    Start Date: January, 1990
    Anticipated Completion Date: To be determined
    Program # 13
RESEARCH PROGRESS AND PLANS  (January 1 to June 30 , 1991)

Research progress, recorded during the period from January 1, 1991 to June 30, 1991 was reported at the Second Annual NASA-UVa LA²ST Program Review Meeting, held on July 9th and 10th at the NASA-Langley Research Center. The agenda for this meeting was as follows:

Tuesday, July 9, 1991

1:00-1:15 pm LA²ST Program overview: D.L. Dicus and R.P. Gangloff


2:00-2:45  "Investigation of the Effect of Thermal Treatment on the Mechanical Properties of Ti-1100/SCS-6 Composites"; D.B. Gundel and F.E. Wawner; Department of Materials Science and Engineering.

2:45-3:00  Break

3:00-3:45  "Experimental Study of the Nonlinear Viscoplastic Response of High Temperature Structures"; M.F. Coyle and E.A. Thornton; Department of Mechanical and Aerospace Engineering.

3:45-4:10  "Concluding Results of Cryogenic Tankage Buckling Analysis and Benchmark Tests for Thermally Loaded Finite Elements"; C. Copper and W.D. Pilkey; Department of Mechanical and Aerospace Engineering.

4:10-4:20  Break


4:40-5:10  "Hydrogen Interactions in Aluminum-Lithium Alloys"; J.R. Scully and S. Smith; Department of Materials Science and Engineering.

6:00  Social/Dinner  (The exact time will be announced at the meeting)
Wednesday, July 10, 1991

8:30-9:00 am "The Effects of Zn Additions on the Microstructure and SCC Performance of Alloy 8090"; R.J. Kilmer and G.E. Stoner; Department Materials Science and Engineering.

9:00-9:20 "Mechanisms of Localized Corrosion in Alloys 2090, X2094 and X2095"; F.D. Wall, R.G. Buchheit and G.E. Stoner; Department of Materials Science and Engineering.

9:20-10:00 "Environmental Fatigue of Alloy 2090 Sheet and Plate"; D.C. Slavik and R.P. Gangloff; Department of Materials Science and Engineering.

10:00-10:15 Break

10:15-11:00 "A Geometrical Description of Microstructure: Applications to Aluminum PM Processing"; J.B. Parse and J.A. Wert; Department of Materials Science and Engineering.

11:00-11:50 "Effects of Temperature and Microstructure on the Fracture of Alloy 2090+In and Weldalite"; J.A. Wagner and C.L. Lach; Metallic Materials Branch and R.P. Gangloff; Department of Materials Science and Engineering.

11:50-1:00 pm Lunch

1:00-1:45 "Elevated Temperature Fracture of RS/PM Aluminum Alloy 8009"; W.C. Porr, Jr., Yang Leng and R.P. Gangloff; Department of Materials Science and Engineering.

1:45-2:10 "The Detection and Analysis of Electrochemical and Chemical Damage to a Bismaleimide/Graphite Fiber Composite Using Electrochemical Impedance Spectroscopy"; F.D. Wall, G.L. Cahen, Jr. and S.R. Taylor; Department of Materials Science and Engineering.

2:10-2:20 Break

2:20-3:00 Group discussion between UVa investigators and LaRC technical contacts on the health and direction of the grant.

3:00- Individual discussions between UVa investigators and LaRC technical contacts on the direction and finances for 1992 renewal elements, to be written in August of 1991.

The visual aids for each presentation are provided in this progress report.
Objective

The broad objective of this PhD research is to characterize and understand the environmental fatigue crack propagation behavior of advanced Al-Li-Cu based alloys and metal matrix composites. Aqueous NaCl and water vapor, which produce atomic hydrogen by reactions on clean crack surfaces, are emphasized. The effects of environment sensitive crack closure, stress ratio and precipitate microstructure are assessed. We seek mechanistic models of intrinsic crack tip damage processes to enable predictions of cracking behavior outside of the data, metallurgical improvements in material cracking resistance, and insight on hydrogen compatibility.
Environmental Fatigue of Alloy 2090 Sheet and Plate

Donald C. Slavik and Richard P. Gangloff
Department of Materials Science

Abstract

Aluminum-lithium alloy 2090 (Al-2.7%Cu-2.2%Li-0.12%Zr in wt.%) is commercially available as sheet and plate. Enhanced near threshold fatigue crack growth rates have been found in reportedly recrystallized 2090 sheet compared to unrecrystallized 2090 plate in moist air. This effect was attributed to high levels of roughness induced crack closure in the 2090 plate due to textured grains and shearable δ' precipitates producing tortuous {111} slip band cracking in moist air. A low level of crack closure was found in the recrystallized sheet with a flat fracture path. These results were not compared based on $\Delta K_{th}$ for $R=0.75$ ($R = P_{min}/P_{max}$), presumably due to the difficulties inherent in defining a single crack opening load from the load-displacement data for high $R$. The exact nature of the texture of grains, recrystallization, and possible texture in the sheet after recrystallization were not quantified. The analysis was not extended to determine the environmental influence which can be important in dramatically changing the cracking mechanisms.

This examination compares intrinsic fatigue crack growth rates and crack path tortuosity for 2090 sheet and plate in the unrecrystallized condition. A constant $K_{max}$ of 17.0 MPa-m$^{1/2}$ was employed in the experiments to circumvent problems associated with defining crack closure levels. The role of grain size, texture, environment, and $\Delta K$ were explored. Experiments were performed in better than 6.5x10$^{-6}$ pascal dynamic vacuum, in moist air, and in an aqueous 1 wt% NaCl solution at a fixed potential. For vintage III sheet, 1 wt% NaCl was found to be mildly more aggressive than moist air, and $\Delta K_{th}$ increased by a factor of 3X in a vacuum over moist air. Crack growth rates were enhanced in moist air by 10X over vacuum in the near threshold regime, with the difference decreasing to 2X for the higher $\Delta K$ ranges examined. Vintage III sheet and vintage III plate were found to have similar intrinsic fatigue crack growth rates in moist air with the plate exhibiting a more tortuous crack path with regions of slip band cracking and possible cleavage cracking. Vintage III plate was compared to an early vintage 2090 plate and a factor of 1.5 reduction in $\Delta K_{th}$ was observed for vintage III plate which is not understood.

Future work will center on clearly characterizing the texture through the material thickness, identifying the exact nature of the grain and subgrain structure, and further monitoring the cracking mode. Proportions of slip band cracking, cleavage cracking, and intersubgranular cracking will be indentified for each environment in sheet and plate specimens. Critical experiments that evaluate the exact nature of damaging cracking modes will be identified for 2090 or model alloy systems.
Environmental Fatigue of Alloy 2090
Sheet and Plate

Donald C. Slavik and Richard P. Gangloff
University of Virginia

Supported by NASA Langley Research Center
D. L. Dicus, Project Monitor
Alloy 2090 Background

Applications
- Low density for aerospace applications
- Available as plate and sheet

Characteristics
- Al-2.7\%Cu-2.2\%Li-0.25\%Mg-0.12\%Zr (in wt. \%)
- Strengthening from $\Theta$ (CuAl$_2$), $T_1$ (Al$_2$CuLi), and $\delta'$ (Al$_3$Li)

- Heavily textured during processing
- Shearable coherent $\delta'$ can give Persistent Slip Bands (PSB)
- Texture and PSB can lead to tortuous crack profiles
Fatigue Crack Characteristics of 2090

- Cracks along \{111\} PSB
  - Multiple \{111\} facets result in a tortuous crack path

- Transgranular crystallographic cracks along \{100\} or \{110\} planes

- Intersubgranular cracks along subgrain boundaries

- Intergranular cracks along grain boundaries

Proportion of cracking characteristics set by conditions
  - \(\Delta K\)
  - Environment
  - Microstructure
Initial Questions

- What determines the amount of fatigue crack path tortuosity?
  - $\Delta K$
  - Environmental influence (Piascik)
  - Processing dependence
  - Texture
  - "Grain size"
  - R-ratio

- How are the intrinsic fatigue crack growth rates influenced by the microstructure, environment, and texture?
  - Crack growth rates
  - Shape of $da/dN-\Delta K$ Curve
2090 Sheet and Plate
(After Venkateswara Rao, Bucci, Jata, & Ritchie)

![Fatigue crack growth rate graph](image-url)
2090 Slip Band Cracking in 13mm Plate

T-Plane

(After Yoder, Pao, Imam, & Cooley)
Procedure

- Characterize 2090 sheet and plate microstructures optically
  - Vintage III 3.2mm Sheet (Age 18 hours at 155 C)
  - Vintage III 19.0mm Plate (Age 18 hours at 163 C)
  - 38.1 mm Plate (Age 4 hours at 190 C - Piascik)

- Determine the texture
  - Texture and texture gradients

- Measure intrinsic fatigue crack growth rates
  - Vacuum
  - Air
  - Aqueous 1.0 wt.% NaCl

- Examine fracture surfaces
Sheet Mid-Plane Texture

Recrystallized Textures
Cube \{100\}<001> 1.6X
Goss \{110\}<001> -.3X

Rolling Textures
Brass \{110\}<112> 30.8X
S \{123\}<634> 16.9X
Cu \{112\}<111> 6.3X

\{ \} = Rolling Plane = SN
< > = Rolling Direction = RD

\{111\} Diffraction Conditions
RD
Alloy 2090
Vintage III Sheet
$K_{\text{max}} = 17 \ \text{MPa-m}^{1/2}$

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

$\begin{array}{c}
10^{-8} \\
10^{-7} \\
10^{-6} \\
10^{-5} \\
10^{-4} \\
10^{-3}
\end{array}$

$\begin{array}{c}
10^{-8} \\
10^{-7} \\
10^{-6} \\
10^{-5} \\
10^{-4} \\
10^{-3}
\end{array}$

$\begin{array}{c}
1 \ \text{wt}\% \ NaCl, -0.84V \ \text{SCE} \\
\text{Moist Air} \\
\text{Vacuum}
\end{array}$

Stress Intensity Range (MPa-m$^{1/2}$)

37
Alloy 2090
1 Wt% NaCl, −.84V SCE
$K_{\text{max}} = 17 \text{ MPa} - \text{m}^{1/2}$
Alloy 2090
Inert Environments
$K_{\text{max}} = 17 \text{ MPa-m}^{1/2}$

![Graph showing da/dN vs. Stress Intensity Range (MPa-m^{1/2})](image)

- △ Vintage III Sheet
- * Plate (T/3)
Alloy 2090
Moist Air
$K_{\text{max}} = 17 \text{ MPa-m}^{1/2}$

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

Stress Intensity Range (MPa-m$^{1/2}$)

- Vintage III Plate
- Plate (T/3)

- Vintage III Sheet
2090 Vintage III Sheet in Air

Crack Direction

0.5 mm

2090 Vintage III Plate (T/2) in Air

0.5 mm
Conclusions

- A reliable experimental setup has been used to measure intrinsic fatigue crack growth rates in sheet specimens.
  - Vacuum
  - Moist air
  - Aqueous 1.0 wt% NaCl

- For 2090 sheet, $\Delta K_{th}$ was a factor of 3 higher in a vacuum as compared to moist air.

- For 2090 sheet, aqueous 1 wt. % NaCl was only mildly more damaging as compared to moist air.
2090 Intrinsic Crack Growth Rate Conclusions

- Vintage III plate versus vintage III sheet
  - Moist air equivalence
  - Some SBC with crack features scaling to the microstructure

- Plate versus vintage III sheet
  - In vacuum higher $\Delta K_{th}$ for vintage III sheet
  - In moist air higher $\Delta K_{th}$ for plate
  - Aqueous 1.0 wt% NaCl similar

- Plate versus vintage III plate
  - Significant difference in moist air
  - 190 C age not the cause
Future Questions

- What dramatically influences intrinsic fatigue crack growth rates in inert and aggressive environments?
  - R-ratio
  - Sheet versus plate texture and/or microstructure
  - Effect of ageing condition

- What factors determine the crack path?
  - Environment
  - \( \Delta K \), R-ratio
  - Microstructure (precipitates, grain size, grain orientation)
  - Texture

- What is the microscopic damage process for a given crack path?

- What is the basis and approach toward predicting \( \text{da/dN} \) versus \( \Delta K \)?
Program 2  Elevated Temperature Fracture of an Advanced Rapidly Solidified 
Powder Metallurgy Aluminum Alloy

William C. Porr, Jr. and Richard P. Gangloff

Objective
The goal of this PhD research is to characterize the fracture behavior of advanced 
powder metallurgy Al-Fe-V-Si alloy 8009 (previously called FVS0812) under monotonic 
loading, as a function of temperature. Particular attention is focused on contributions to the 
fracture mechanism from the novel fine grained dispersoid strengthened microstructure, 
dissolved solute from rapid solidification, and the moist air environment.

Program 2  Elevated Temperature Crack Growth in Advanced Aluminum Alloys
Yang Leng and Richard P. Gangloff

Objective
The objective of this study is to characterize time-dependent crack growth in advanced 
aluminum alloys at elevated temperatures with the fracture mechanics approach, and to 
examine cracking mechanisms with a metallurgical approach. Specific tasks in the past 
twelve months were:

to obtain sustained load crack growth experimental information from a refined testing 
system;

to correlate crack growth kinetics with the J-integral and time dependent C(t); and

to investigate the intermediate temperature embrittlement of 8009 alloy in order to 
understand crack growth mechanisms.
Dispersion strengthened, powder metallurgy aluminum alloys produced by rapid solidification and mechanical alloying techniques are candidate materials for next generation aerospace structures at intermediate elevated temperatures (150 - 225 °C). One of the most promising alloys identified from this class is the rapidly solidified, powder processed, Al-8.5Fe-1.3V-1.7Si (wt.%) alloy 8009 produced by Allied-Signal, Inc. Preliminary study of AA 8009 indicated excellent elevated temperature tensile strength retention due to thermal stability of the ultrafine grain structure and the high volume fraction Al$_{12}$(Fe,V)$_3$Si silicide. In the initial stages of this study, it was shown, however, that damage tolerance for 8009 decreases with increasing temperature and decreased loading rate. Similar degradation has been observed in other alloys of this class. The cause of this degradation is uncertain, however several mechanisms have been proposed including: dynamic strain aging due to excess solid solution iron, extrinsic delamination toughening, environmental embrittlement due to water vapor or oxygen reactions with aluminum, embrittlement by retained hydrogen from processing, or temperature enhanced localized deformation unique to ultrafine grain size alloys with a high volume fraction of strengthening dispersoids. The current objective of this study is to determine the mechanism(s) by which the damage tolerance of AA 8009 decreases with increasing temperature and decreasing loading rate.

Recent work has focused on confirming the intrinsic nature of the elevated temperature damage tolerance degradation of 8009 and on determining the role of the moist air environment and retained hydrogen from processing in this unusual fracture behavior. Monotonic increasing load plane strain fracture toughness, $K_{IC}$, was determined for cross-rolled 8009 plate as a function of temperature. A large decrease in fracture toughness was observed with increasing test temperature independent of specimen orientation ($K_{IC}$: 30 MPa/m at 25 °C, 10 MPa/m at 175 °C). This decrease occurred despite the absence of prior particle boundary delamination in specimens at both temperatures like that seen in the LT orientation of the extruded material at 25 °C. This confirms that the toughness decrease with temperature previously observed in extruded AA 8009 was intrinsic to the material and decreased extrinsic delamination toughening had, at most, a minor effect on $K_{IC}$.

Fracture mechanics characterizations of monotonic increasing and sustained load damage tolerance were conducted at 175 °C in air and high vacuum (< 30 μPa), for material as-processed and after an extended vacuum heat treatment. K-$\Delta$a R-curves were determined for extruded and plate 8009. Results indicated no effect of environment or the 75 hour 330 °C vacuum heat treatment on the fracture behavior of the 8009 in either plate or extruded form. The critical stress intensity for fracture initiation, $K_{IC}$, was approximately 16 MPa/m for all environment and heat treatment conditions in the extruded material at 175 °C ($K_{IC} = 10$ MPa/m for plate), reduced from a $K_{IC}$ of 30 MPa/m at 25 °C. Likewise the slopes of the R-curves, an indication of material resistance to stable crack growth, are...
similar for all conditions at 175°C and are much reduced from values at 25°C. Additionally, $K - da/dt$ sustained load crack growth results indicated subcritical crack growth at 175°C in vacuum, similar to results in air. Determination of the hydrogen content of the 8009; as received, after elevated temperature testing, and after vacuum heat treatment; indicated that hydrogen contents were similar in all cases, and hydrogen was not mobile at temperatures as high as 330°C in this material. These results imply that the degradation of damage tolerance of AA 8009 with increasing temperature is not due to embrittlement from a moist air environment or retained hydrogen from processing.

Stereo pair, matching fracture surface SEM fractography was performed on failed specimens and indicated a locally plastic fracture mode for experiments at 25 and 175°C, in air and vacuum. The nature of the fracture mode varied with temperature and environment, however. At 25°C, the fracture surface contained a dual distribution of dimples, .5 to 2 μm and 4 to 8 μm in diameter. At 175°C, fracture surfaces of specimens tested in air and vacuum had a uniform distribution of shallow dimples 2 to 4 μm in diameter. Specimens tested in vacuum also had a large number of submicron particles apparent on the fracture surface. The explanation for these differences is unknown at this time.

Future work will focus on interpretation of the elevated temperature fracture behavior of AA 8009 through microscopy of failed fracture mechanics specimens and sectioned notched bars from interrupted tensile experiments. With an understanding of the evolution of fracture in 8009, mechanisms will be proposed to account for the effect of temperature and loading rate in degrading the damage tolerance of this material.
ELEVATED TEMPERATURE FRACTURE OF RS/PM ALUMINUM ALLOY 8009

William C. Porr, Jr.
Yang Leng
Richard P. Gangloff

Funded by NASA Langley Research Center
C. E. Harris, Project Monitor
HIGH TEMPERATURE PM ALUMINUM ALLOYS

- Structural Applications - HSCT

Mach 2.0: 100°C Long Term
Temperature Requirement
-- 135°C Peak

Mach 2.4: 175°C Long Term
-- 200°C Peak
TEM micrograph of FVS0812 Al alloy
PROCEDURES

- Measured Parameters

LEFM: \( K - \Delta a \) ........... \( P, a \)
\( K - da/dt \) ...... \( P, a, t \)

EPFM: \( J - \Delta a \) ........... \( P, a, \delta \)
\( J - da/dt \) ...... \( P, a, \delta, t \)

- a from DC potential drop technique
CONTROLLED TEMPERATURE CHAMBER
-196°C TO 425°C

INSTRON 1362 SERVO-ELECTRIC TEST SYSTEM

10,000X AMPLIFIER

LVDT CONDITIONER

INTERFACE

LOAD

DC POWER SUPPLY

COMPUTER
Fracture Toughness, $K_{IC}$ (MPa·m$^{1/2}$)

Temperature (°C)
Fracture Toughness, $K_{IC}$ (MPa-m$^{1/2}$)

Tearing Modulus, $T$

Actuator Displacement Rate (mm/s)

AA 8009
LT Orientation
175°C
"CREEP BRITTLE"---Crack growth is faster than the crack tip creep zone expansion rate

- Stress Intensity Factor
- J-integral

"Creep Ductile"---Process zone or ligament deformation occur at substantial rate compared to $da/dt$

- $C^*(t)$ Path independent energy rate integral and amplitude of HRR strain rate field for large scale primary and steady state creep
- $C(t)$ Amplitude of HRR strain rate field for small scale transient to extensive creep regimes
- $C_t$ Energy dissipation rate integral relating creep zone expansion rate for small scale transient to extensive creep regimes
Al-Fe-V-Si (8009)

\( \frac{da}{dt} \) (mm/hour)

\( K \) (MPa√m)
POSSIBLE CAUSES

Delamination Toughening
-- Extrinsic

Creep
-- Novel Mechanism Due To Ultra Fine Microstructure

Environment
-- Oxidation
-- Hydrogen Embrittlement

Dynamic Strain Aging
-- Greater than equilibrium substitutional solute concentration
EXTRUSION DIRECTION 200µm

TRANSVERSE 200µm

Plane Strain

Through Thickness Constraint

Plane Stress

\[ \varepsilon_f \]
Low magnification SEM photographs of FVS0812 fracture surfaces for different test temperatures.
Low magnification SEM photographs of AA 8009 plate fracture surfaces tested in air
AA 8009
Extrusion
LT Orientation

Stress Intensity, $K$ (MPa-m$^{1/2}$)

25°C

175°C

Crack Growth (mm)
\[
\frac{da}{dN} (\text{mm/cycle})
\]

\[
\Delta K = 3.3 \text{ MPa-m}^{1/2}
\]

Air, 22°C
Air, 175°C
Vacuum, 175°C
HYDROGEN CONTENT

- Hydrogen content of AA 8009 determined by vacuum fusion technique:

  - As received: $4.3 \pm 0.3$ ppm

  - Vacuum heat treated at $330^\circ C$
    and fractured in vacuum at
    $175^\circ C$: $4.4 \pm 0.2$ ppm

  - As received and fractured in air
    at $175^\circ C$: $4.3 \pm 0.1$ ppm

  - Allied-Signal (D. Raybould): $\approx 3$ ppm
FRACTOGRAPHY

- Precision matching of fracture surfaces, coupled with stereo pairs fractography, indicated a locally plastic fracture mode at 25 and 175°C, in air and vacuum.

- 25°C: dual distribution of dimples — 0.5 to 2 μm and 4 to 8 μm

- 175°C: uniform distribution of shallow dimples — 2 to 4 μm

- Oxides critical?  ■ Boundary cracking?
High magnification SEM photographs of matching fracture surface features. Specimens tested in air at 175° C.
SEM micrographs of AA 8009 plate with oxide distribution and accompanying fracture surface (Fractured in Air, 175°C)
DYNAMIC STRAIN AGING

- None of the proposed mechanisms are supported by microscopic observations

- Localized solid solution strengthening resulting in localized deformation

  -- could explain similar behavior in MA Al-Ti
TEMPERATURE ENHANCED DEFORMATION

- Temperature enhanced dislocation flow without accompanying grain boundary sliding results in intergranular cavity formation.

- No microscopic evidence (yet) of intergranular cavity formation
CONCLUSIONS: PHENOMENOLOGY

- Elevated temperature damage tolerance degradation appears generic to the advanced PM aluminum alloys.

- AA 8009 exhibits intrinsic ductility and toughness decreases with increasing temperature.

- AA 8009 exhibits decreased toughness with decreasing loading rate at 175°C.
AA 8009 is susceptible to elevated temperature, time dependent crack growth at K levels less than $K_{IC}$ in both air and vacuum environments. Threshold K for crack growth is equal to slow loading $K_{IC}$.

AA 8009 is creep brittle; crack growth rates can be correlated to K, and more properly, J.

For a given applied J (K), time dependent crack growth rate is high at 175°C and decreases with increasing temperature to 316°C for 8009.
CONCLUSIONS: MECHANISM

- The unusual intrinsic mechanical behavior of AA 8009 is not the result of moist air environmental embrittlement.

- The hydrogen content of AA 8009 does not change with long term, elevated temperature moist laboratory air exposure and retained hydrogen is not mobile at temperatures as high as 330°C.
Fractography indicated a ductile mode of fracture at 25 and 175°C, in air and vacuum. Differences in feature sizes with temperature, but not environment, were apparent. Oxides from processing may be critical in the fracture evolution.

Degradation of the damage tolerance of AA 8009 at elevated temperatures is most likely due to dynamic strain aging or a unique temperature dependent deformation mechanism.
FUTURE WORK

- Continue microscopy of failed fracture mechanics specimens and sectioned notched bars from interrupted tensile experiments.

- Develop defendable mechanism for elevated temperature damage tolerance degradation of AA 8009.

- Complete PhD. Dissertation by July 1, 1992.
Program 3  Temperature Effects on the Deformation and Fracture of Al-Li-Cu-In Alloys

John A. Wagner and R.P. Gangloff

Objective

The objective of this PhD research is to characterize and optimize the crack initiation and growth fracture resistance of Al-Cu-Li and Al-Cu-Li-In alloys for cryogenic tank applications. The program aims to understand microscopic fracture mechanisms, as influenced by ambient to cryogenic temperature, stress state and microstructure.
Fracture of 2090 and 2090+In Alloys at Cryogenic Temperatures

John A. Wagner and R.P. Gangloff

The objective of this program is to understand, characterize and optimize the crack initiation and growth fracture resistance of Al-Cu-Li and Al-Cu-Li-In alloys for cryogenic tank applications. Presently, this work is concentrating on determining the effects of stress state and temperature on the fracture toughness and fracture mechanisms of commercially available Vintage 3H-2090-T81 and experimental 2090+In-T6. Precracked J-integral specimens of both alloys have been tested at ambient and cryogenic temperatures in the plane stress and plane strain conditions. Considering ambient temperature, results showed that 2090-T81 exhibited the highest toughness in both plane strain and plane stress conditions. For the plane strain condition, reasonable crack initiation and growth toughnesses of 2090-T81 are associated with a significant amount of delamination and transgranular fracture. Plane stress toughnesses were higher and fracture was characterized by shear cracking with minimal delaminations. In comparison, the fracture behavior of 2090+In-T6 is significantly degraded by subgrain boundary precipitation. Toughness is low and characterized by intersubgranular fracture with no delamination in the plane stress or plane strain conditions. Intersubgranular cracking is a low energy event which presumably occurs prior to the onset of slip band cracking. Copious grain boundary precipitation is atypical of commercially available 2090. At cryogenic temperatures, both alloys exhibit increased yield strength, toughness and amount of delamination and shear cracking. The change in fracture mode of 2090+In-T6 from intersubgranular cracking at ambient temperature to a combination of intersubgranular cracking, shear cracking and delamination at cryogenic temperature is the subject of further investigation.
FRACTURE OF AI-Li ALLOYS 2090 AND 2090+In AT CRYOGENIC TEMPERATURES

John A. Wagner

LA²ST Program Review
NASA Langley Research Center

July 9-10, 1991
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 intragranular features & grain boundary structure in governing the occurrence of various fracture mechanisms in Al-Li-Cu-X alloys at ambient and cryogenic temperatures.
FRACTURE OF 2090 AND 2090+In ALLOYS AT CRYOGENIC TEMPERATURES

Outline

- Summary of initial experimentation (sheet)
- Progress on plate material testing
- Future Direction
INDIUM ADDITIONS TO 2090-BASED ALLOYS

Sheet Product Form

Observations

• Increased in $\sigma_{ys} + \sigma_{ult}$ observed for 30 lb laboratory permanent mold casting attributed to increase number density of $T_1$

• For 350 lb DC castings indium additions increased $\sigma_{ult}$ but had no effect on $\sigma_{ys}$ regardless of product form

• Fracture of 2090+In alloy primarily characterized by intersubgranular failure

• TMT used to render 2090+In superplastic complicated interpretation of results
OBJECTIVES OF EXPERIMENTAL TESTING OF PLATE MATERIAL

- Determine the effect of key variables on J (Δa) behavior, fracture path and fracture mode of 2090 and 2090+In alloys with respect to
  - Temperature
  - Constraint
  - In addition
  - Material vendor
  - Orientation
MICROSTRUCTURES OF PLATE ALLOYS

R2090 + In-T6

A2090-T81

100 \mu m

S

T

T

ORIGINAL PAGE IS OF POOR QUALITY
COMPARISON OF SUBSTRUCTURE PRECIPITATION OF A2090-T6 AND R2090-T6 AGED 20 HRS AT 160°C
COMPARISON OF LABORATORY HEAT TREATMENTS AND QUENCH RATES ON 2090+In

- **Reynolds Metals**
  - SHT = 555°C, 0.5 hrs
  - Age = 160°C, 72 hrs
  - Cold water quench

- **NASA LaRC**
  - SHT = 560°C, 0.5 hrs
  - Age = 160°C, 72 hrs
  - Ice brine quench
TENSILE PROPERTIES 2090 AND 2090+In ALLOYS

- Ultimate
- Yield
- Elongation

Strength, ksi

Elongation, %

R2090 + In-T6
A2090-T81
FRACTURE TOUGHNESS R-CURVE FOR 0.47" THICK SPECIMENS WITH SIDEGROOVES AT ROOM TEMPERATURE

\[ J \text{ (in.-lb/in.}^2) \]

\[ K_{lc}^{A2090} = 27 \text{ ksi} \sqrt{\text{in.}} \]

\[ K_{lc}^{R2090+In} = 21 \text{ ksi} \sqrt{\text{in.}} \]
FRACTURE MORPHOLOGY OF 0.47" THICK COMPACT TENSION SPECIMENS AT ROOM TEMPERATURE

A2090-T81

R2090 + In-T6
FRACTURE SURFACE MORPHOLOGY OF PRE-CRACK/FAST FRACTURE TRANSITION REGION

A2090-T81

R2090 + In-T6
FRACTURE TOUGHNESS R-CURVE FOR 0.06" THICK SPECIMENS AT ROOM TEMPERATURE

\[ K_{ic}^{A2090} = 31 \text{ ksi} \sqrt{\text{in.}} \]

\[ K_{ic}^{R2090+In} = 27 \text{ ksi} \sqrt{\text{in.}} \]
TESTING AT CRYOGENIC TEMPERATURES
TENSILE PROPERTIES 2090 AND 2090+In ALLOYS

* Extrapolated from sheet cryoproperties
LOAD VERSUS DISPLACEMENT CURVES FOR 0.473 IN. 2090-T81 SPECIMENS AT 25°C AND -185°C
FRACTURE SURFACE OF 2090-T81 AT ROOM AND CRYOGENIC TEMPERATURES

B = 0.47 in. with sidegrooves

T = 25°C

T = -185°C
LOAD VERSUS DISPLACEMENT CURVES FOR 0.063 IN. 2090-T81 SPECIMENS AT 25°C AND -185°C

T = 25°C

T = -185°C

Load, lb

Displacement, in.

Displacement, in.
LOAD VERSUS DISPLACEMENT CURVES FOR 0.473 IN. 2090+In-T6 SPECIMENS AT 25°C AND -185°C

T = 25°C

T = -185°C
FRACTURE SURFACE OF 2090+In-T6 AT ROOM AND CRYOGENIC TEMPERATURES

$B = 0.47$ in. with sidegrooves

$T = 25^\circ C$

$T = -185^\circ C$
FRACTURE SURFACE OF 2090+In-T6 AT -185°C
FRACTURE SURFACE MORPHOLOGY OF 2090+In-T6 AT -185°C
LOAD VERSUS DISPLACEMENT CURVES FOR 0.063 IN. 2090+In-T6 SPECIMENS AT 25°C AND -185°C

T = 25°C

T = -185°C
Fracture of 2090 and 2090+In Alloys at Cryogenic Temperatures

SUMMARY

- In additions to 2090-based alloys increase $T_1$ number density, increase $\sigma_{ult}$, and no change in $\sigma_{ys}$

- Fracture in Alcoa 2090-T81 is primarily characterized by delamination and transgranular shear at both 25°C and -185°C. Increase in toughness at -185°C associated with an increased level of delamination

- At 25°C the toughness of 2090+In-T6 is low, due to subgrain boundary precipitates, and characterized by intersubgranular fracture

- At -185°C the toughness of 2090+In-T6 is higher and there is a change in fracture mode from solely intersubgranular to intersubgranular with delaminations and transgranular shear
Fracture of 2090 and 2090+In Alloys at Cryogenic Temperatures

ISSUES

• Identify subgrain boundary particles and associated PFZ in 2090+In-T6
  Determine if In additions promote subgrain boundary precipitation

• Attempt to increase the toughness of 2090+In-T6 using modified aging treatments

• Determine the effect of stress state for the delamination/transgranular shear and intersubgranular fracture modes in 2090+In-T6 and 2090-T81

• Examine the mechanisms to account for the observed change in fracture mode of 2090+In-T6 at -185°C
Objective

The objective of this research is to characterize the uncertain effect of temperature on the deformation and fracture behavior of Weldalite™ 049 from cryogenic to elevated temperatures. We will measure fracture resistance and emphasize the determination of fracture mechanisms, including slip plane cracking, high angle boundary delamination, subgrain boundary cracking, and microvoid coalescence. Microstructure will be controlled to produce either predominantly $T_1$ or $T_1 + \delta^*$ (after Blankenship and Starke) and to examine the effect of dislocation-precipitate interaction on fracture toughness.
EFFECTS OF TEMPERATURE AND MICROSTRUCTURE ON THE FRACTURE OF WELDALITE™ 049

C. L. Lach

LA²ST Program Review

NASA Langley Research Center

July 9-10, 1991
OBJECTIVE

- To characterize the effect of precipitate slip interactions and temperature on the deformation and fracture behavior of Weldalite™ 049 from cryogenic to elevated temperatures.
APPRAOCH

- Limited fracture toughness data available as a function of temperature

- Test hypothesis of particle dislocation interactions involved with slip localization (Blankenship)

- Investigate the effect of temperature on slip localization and delamination (Wagner, Porr, Leng)
<table>
<thead>
<tr>
<th>Element</th>
<th>X2095 Alloy Registration for Weldalite™ 049</th>
<th>(\text{Weldalite}^\text{TM} 049)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>0.12 max</td>
<td>—</td>
</tr>
<tr>
<td>Fe</td>
<td>0.15 max</td>
<td>0.08</td>
</tr>
<tr>
<td>Cu</td>
<td>3.9-4.6</td>
<td>4.64</td>
</tr>
<tr>
<td>Mn</td>
<td>0.10 max</td>
<td>—</td>
</tr>
<tr>
<td>Mg</td>
<td>0.25-0.6</td>
<td>0.37</td>
</tr>
<tr>
<td>Zn</td>
<td>0.25 max</td>
<td>0.17</td>
</tr>
<tr>
<td>Ag</td>
<td>0.25-0.6</td>
<td>0.35</td>
</tr>
<tr>
<td>Li</td>
<td>1.0-1.6</td>
<td>1.53</td>
</tr>
<tr>
<td>Zr</td>
<td>0.04-0.18</td>
<td>0.17</td>
</tr>
<tr>
<td>Ti</td>
<td>0.10 max</td>
<td>—</td>
</tr>
<tr>
<td>Al</td>
<td>Bal</td>
<td>Bal</td>
</tr>
</tbody>
</table>
(111) POLE FIGURE FOR WELDALITE™ 049

File: SY0:Z04840.PFG
Sample: WELDALITE 049-T3
T/2 (LACH)
2-MAY-91 07:24:46
H= 1  K= 1  L= 1

Plot Levels:

- 2.6% 0.75
- 4.4% 1.05
- 7.0% 1.25
- 10.5% 1.50
- 14.0% 2.00
AS RECEIVED WELDALITE™ 049 (1.6% Li)
AS RECEIVED WELDALITE™ 049 (1.6% Li)
SUBGRAN STRUCTURE OF WELDALEITE™ 049 (1.6% Li)

ORIGINAL PAGE IS
OF POOR QUALITY
AGED AT 145°C FOR 72 HOURS

Precipitates

δ' (Al₃Li)
θ' (Al₂Cu)
T₁ (Al₂CuLi)
<table>
<thead>
<tr>
<th>Precipitates</th>
<th>$T_1$ (Al$_2$CuLi)</th>
<th>$S'$ (Al$_2$CuMg)</th>
<th>$\theta'$ (Al$_2$Cu)</th>
</tr>
</thead>
</table>

-T8 TEMPER

165°C for 24 hours
<table>
<thead>
<tr>
<th>Temper</th>
<th>145°C 24 hours</th>
<th>145°C 68 hours</th>
<th>165°C 24 hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield strength, ksi</td>
<td>83</td>
<td>91</td>
<td>89</td>
</tr>
<tr>
<td>Ultimate strength, ksi</td>
<td>88</td>
<td>91</td>
<td>92</td>
</tr>
<tr>
<td>Percent elongation</td>
<td>10.0</td>
<td>7.1</td>
<td>6.4</td>
</tr>
<tr>
<td></td>
<td>9.0</td>
<td>8.4</td>
<td>7.6</td>
</tr>
</tbody>
</table>

MECHANICAL PROPERTIES

Room Temperature
TEST PROCEDURE TO DETERMINE $J_{IC}$ AND $K_{IC}$

Fracture surface

$\Delta \alpha_{Ave}$

Precrack

Load, $P$

Partial unload about 10%

Load line displacement

$J = (2\sigma_{flow})(\Delta \alpha)$

Blunting line

$J_{IC}$

$K_{IC}$
# TEST MATRIX

<table>
<thead>
<tr>
<th>Weldalite™ 049</th>
<th>Temperature, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>-190</td>
</tr>
<tr>
<td>Non shearable</td>
<td></td>
</tr>
<tr>
<td>$T_1$</td>
<td></td>
</tr>
<tr>
<td>$165^\circ C/24$ hrs</td>
<td></td>
</tr>
<tr>
<td>$R_B$ 92</td>
<td></td>
</tr>
<tr>
<td>$T_1$ ($s', \theta'$)</td>
<td>TR</td>
</tr>
<tr>
<td>$P_E$</td>
<td></td>
</tr>
<tr>
<td>$R_J$</td>
<td></td>
</tr>
<tr>
<td>$P_{E_E}$</td>
<td></td>
</tr>
<tr>
<td>$J_{A_R}$</td>
<td></td>
</tr>
<tr>
<td>$T_{R (T)}$</td>
<td></td>
</tr>
<tr>
<td>$P_{E_E (P_E)}$</td>
<td></td>
</tr>
<tr>
<td>$J_{A_R (J_{A_R})}$</td>
<td></td>
</tr>
<tr>
<td>Shearable</td>
<td></td>
</tr>
<tr>
<td>$145^\circ C/24$ hrs</td>
<td></td>
</tr>
<tr>
<td>$R_B$ 90</td>
<td></td>
</tr>
<tr>
<td>$\delta'$, GPZ/$\theta'$, $T_1$</td>
<td>TR</td>
</tr>
<tr>
<td>$P_E$</td>
<td></td>
</tr>
<tr>
<td>$J_{A}$</td>
<td></td>
</tr>
<tr>
<td>$T_{R (T)}$</td>
<td></td>
</tr>
<tr>
<td>$P_E (P_E)$</td>
<td></td>
</tr>
<tr>
<td>$J_{A (J_{A})}$</td>
<td></td>
</tr>
</tbody>
</table>

$T = \text{Tensile}$

$P_{E} = \text{plane strain}$

$J_{A} = \text{Plane stress}$

$R = \text{Replication}$

( ) = Tested at UVA
SUMMARY

- 0.5" Weldalite™049 T-3 plate obtained

- Material characterization and heat treatments selected (Blankenship)

- NASA and UVA compact tension specimens machined and heat treated

- Fracture testing equipment developed and on-line (Wagner, Porr)

- Ready to begin experiments
QUESTIONS

- Does precipitate-dislocation interaction affect crack initiation and growth toughnesses for Weldalite™049?

- What is the effect of temperature?

Fracture mechanics data Microscopic behavior
Objectives

The objective of this research is to characterize the localized corrosion and stress corrosion crack initiation behavior of Al-Li-Cu alloy 2090 in aqueous environments, and to gain an understanding of the role of local corrosion and occluded cell environments in the mechanisms of pitting and stress corrosion crack initiation and early-stage propagation.
Mechanisms of Localized Corrosion in Alloys 2090 and X2095

F. D. Wall
G. E. Stoner

Department of Materials Science and Engineering

This report includes summary information on electrochemical aspects of stress corrosion cracking in alloy 2090 and an introduction to the work to be initiated on the new X2095 (Weldalite) alloy system.

Stress corrosion cracking (SCC) was studied in both S-T and L-T orientations in alloy 2090. A constant load TTF test was performed in several environments with a potentiostatically applied potential. In the same environments the electrochemical behavior of phases found along subgrain boundaries was assessed. It was found that rapid failure due to SCC occurred when the following criteria was met:

\[ E_{BR, T1} < E_{applied} < E_{BR, matrix phase} \]

Although the L-T orientation is usually considered more resistant to SCC, failures in this orientation occurred when the stated criteria was met. This may be due to the relatively isotropic geometry of the subgrains which measure approximately 15-25 \( \mu \text{m} \) in diameter.

Initial studies of alloy X2095 will include electrochemical characterization of three compositional variations each at three tempers. The role of \( \Gamma \) dissolution in SCC behavior will be addressed using techniques similar to those used in the research of 2090 described above. SCC susceptibility will also be studied using alternate immersion facilities at Reynolds Metals Corporation. Pitting will be investigated in terms of stability, role of precipitate phases and constituent particles, and as initiation sites for SCC.

In all research endeavors attempts will be made to link electrochemistry to microstructure. Previous work on 2090 will provide a convenient basis for comparison since both alloys contain \( \Gamma \) precipitates but with different distributions. In 2090 \( \Gamma \) forms preferentially on subgrain boundaries whereas in X2095 the microstructure appears to be more homogeneous with finer \( \Gamma \) particles. Another point for comparison is the strengthening phase found in 2090 but absent in X2095.

\[ T \] (sub \( \Gamma \))

Delta prime
Mechanisms of Localized Corrosion in Alloys 2090 and X2095

F. D. Wall
G. E. Stoner

Department of Materials Science and Engineering
University of Virginia
Charlottesville, Virginia 22901

NASA - LaRC Contact: D. L. Dicus

Co-sponsor: Reynolds Metals Corporation
Technical Contact: Alex Cho
OUTLINE OF PRESENTATION

I. Completed work on alloy 2090
   A. Summery of results by R.G. Buchheit
   B. New data and results

II. Proposed project on X2095 alloys
   A. Materials
   B. Initial objectives
The Role of Anodic Dissolution in the SCC of Alloy 2090

Graduate research assistant: Rudy Buchheit

Undergraduate assistant: Doug Wall
PREVIOUS WORK

(presented earlier by R. G. Buchheit)

(1) Study the electrochemical behavior of phases present along the subgrain boundaries.

(2) Evaluate SCC susceptibility in S-T direction as a function of applied potential.

(3) Correlate electrochemical parameters to SCC behavior.
EXPERIMENTAL

Environments:
- 0.6M NaCl
- 0.1M NaCl + 0.1M Na$_2$CrO$_4$
- 0.6M NaCl + 0.1M Li$_2$CO$_3$

Potentiodynamic polarizations:

<table>
<thead>
<tr>
<th>Phase of Interest</th>
<th>Model</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha$-Al</td>
<td>SHT 2090</td>
</tr>
<tr>
<td>Cu-depleted zone</td>
<td>1100 Aluminum</td>
</tr>
<tr>
<td>$T_1$</td>
<td>cast ingot</td>
</tr>
</tbody>
</table>

Time-to-failure testing:

- Loading axis in S-T direction
- Constant load at 62%YS
- Smooth bar tensile samples
- Potentiostatic anodic polarization
## RESULTS OF ELECTROCHEMICAL TESTING

### 0.6M NaCl

<table>
<thead>
<tr>
<th>Material</th>
<th>$E_{corr}(V_{SCE})$</th>
<th>$E_{br}(V_{SCE})$</th>
<th>$i_{pass}(\mu A/cm^2)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha$-Al</td>
<td>-0.729</td>
<td>-0.731</td>
<td>not measured</td>
</tr>
<tr>
<td>Cu-DZ</td>
<td>-0.840</td>
<td>-0.749</td>
<td>0.024</td>
</tr>
<tr>
<td>$T_1$</td>
<td>-1.096</td>
<td>-0.723</td>
<td>400</td>
</tr>
</tbody>
</table>

### 0.1M NaCl + 0.1M Na$_2$CrO$_4$

<table>
<thead>
<tr>
<th>Material</th>
<th>$E_{corr}(V_{SCE})$</th>
<th>$E_{br}(V_{SCE})$</th>
<th>$i_{pass}(\mu A/cm^2)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha$-Al</td>
<td>-0.751</td>
<td>+0.020</td>
<td>3.7</td>
</tr>
<tr>
<td>Cu-DZ</td>
<td>-0.920</td>
<td>+0.100</td>
<td>4.0</td>
</tr>
<tr>
<td>$T_1$</td>
<td>-1.195</td>
<td>-0.504</td>
<td>80</td>
</tr>
</tbody>
</table>

### 0.6M NaCl + 0.1M Li$_2$CO$_3$

<table>
<thead>
<tr>
<th>Material</th>
<th>$E_{corr}(V_{SCE})$</th>
<th>$E_{br}(V_{SCE})$</th>
<th>$i_{pass}(\mu A/cm^2)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha$-Al</td>
<td>-1.600</td>
<td>-0.500</td>
<td>2.0</td>
</tr>
<tr>
<td>Cu-DZ</td>
<td>-1.633</td>
<td>-0.540</td>
<td>0.44</td>
</tr>
<tr>
<td>$T_1$</td>
<td>-1.143</td>
<td>-0.721</td>
<td>250</td>
</tr>
</tbody>
</table>
Breakaway Potentials of Subgrain Boundary Phases

- ○ matrix
- □ Cu-depleted zone
- + T₁ precipitate phase

Potential (V SCE)

0.6M NaCl + 0.1M Na₂CrO₄ + 0.1M Li₂CO₃

0.1M NaCl

0.6M NaCl
Time-to-Failure Results for Samples in 0.6M NaCl

<table>
<thead>
<tr>
<th>Applied Potential (Vsce)</th>
<th>Time to Failure (Days)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-0.650</td>
<td>1 @ &gt; 5 *</td>
</tr>
<tr>
<td>-0.700</td>
<td>1 @ &gt; 5 *</td>
</tr>
<tr>
<td>-0.715</td>
<td>2 @ &gt; 45 +</td>
</tr>
<tr>
<td>-0.720 (E_{corr})</td>
<td>3 @ &gt; 45 +</td>
</tr>
<tr>
<td>-0.725</td>
<td>1 @ &gt; 5 *</td>
</tr>
<tr>
<td>-0.730</td>
<td>1 @ &gt; 5 *</td>
</tr>
<tr>
<td>-0.900</td>
<td>1 @ &gt; 5 *</td>
</tr>
<tr>
<td>-1.150</td>
<td>2 @ &gt; 45 +</td>
</tr>
</tbody>
</table>

1 @ > 5 : read one specimen did not fail after 5 days
* per ASTM G49
+ per static load technique
TTF Results for samples in 0.1M NaCl + 0.1M Na$_2$CrO$_4$

![Graph showing TTF results for 0.1M NaCl + 0.1M Na$_2$CrO$_4$](image)

TTF Results for samples in 0.6M NaCl + 0.1M Li$_2$CO$_3$

![Graph showing TTF results for 0.6M NaCl + 0.1M Li$_2$CO$_3$](image)
CONCLUSIONS

(1) In the S-T direction rapid failure due to SCC occurs if the following criteria is met:

\[ E_{BR,T1} < E_{\text{applied}} < E_{BR,\alpha-Al} \]

(2) In 0.6M NaCl the criteria is not met since \( E_{BR,T1} \approx E_{BR,\alpha-Al} \) and no rapid failures occur.

(3) In NaCl solution with either Li\(_2\)CO\(_3\) or Na\(_2\)CrO\(_4\) added there exists a potential window in which the criteria can be met and rapid failures occur.
Results for L-T samples in 0.1M NaCl + 0.1M Na₂CrO₄

![Graph showing time to failure versus applied potential.](Image)

- Time to failure (hours)
- Applied Potential (V_{soc})
- 2090 LT 14 hours at 160° C
  0.1 M Na₂CrO₄ + 0.1 M NaCl

(no failures)
CONCLUSIONS

(1) Rapid failure due to SCC in the L-T direction occurs under the same conditions as seen for the S-T orientation:

\[ E_{BR, T_1} < E_{applied} < E_{BR, \alpha-Al} \]

(2) Since the crack appears to follow subgrain boundaries, embrittlement in the L-T orientation may occur due to the equiaxed subgrains in the 2090 plate.

(3) In the environments investigated the dissolution of the T₁ phase appears to be the primary mechanism for SCC due to anodic dissolution.
Mechanisms of Localized Corrosion in Alloy X2095 and Compositional Variations

F.D. Wall
G. E. Stoner

Department of Materials Science and Engineering
University of Virginia
Charlottesville, Virginia 22901

Nasa - LaRC Contact : D. L. Dicus

Co-sponsor : Reynolds Metals Corporation
Technical Contact : Alex Cho
MATERIALS

X2095, 3 tempers

Compositional variations: RX820, RX821, 3 tempers each

<table>
<thead>
<tr>
<th>Element</th>
<th>Percentage</th>
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<tr>
<td>Al</td>
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<td>1.0-1.6</td>
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<td>Ag</td>
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<td>Si</td>
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<td>Fe</td>
<td>0.15 Max</td>
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<tr>
<td>Mn</td>
<td>0.10 Max</td>
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<tr>
<td>Zn</td>
<td>0.25 Max</td>
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<tr>
<td>Zr</td>
<td>0.04-0.18</td>
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<tr>
<td>Ti</td>
<td>0.10 Max</td>
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<td>Others, each</td>
<td>0.05 Max</td>
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<tr>
<td>Others, total</td>
<td>0.15 Max</td>
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MATERIALS (PROPERTIES)

For X2095 in T8 condition

<table>
<thead>
<tr>
<th>Direction</th>
<th>UTS (ksi)</th>
<th>TYS (ksi)</th>
<th>EL (%)</th>
</tr>
</thead>
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<tr>
<td>L</td>
<td>90.0</td>
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<tr>
<td>L-T</td>
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<td>82.3</td>
<td>9.6</td>
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<tr>
<td>45 deg.</td>
<td>80.6</td>
<td>73.6</td>
<td>14.3</td>
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<table>
<thead>
<tr>
<th>Direction</th>
<th>$K_{1c}(w=1&quot;)$</th>
<th>$K_{R}^{max}(w=6&quot;)$</th>
<th>$K_{C}(16&quot;$wide)</th>
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</thead>
<tbody>
<tr>
<td>L-T</td>
<td>28.4</td>
<td>62.0</td>
<td>69.7</td>
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<tr>
<td>T-L</td>
<td>24.3</td>
<td>56.0</td>
<td>43.4</td>
</tr>
</tbody>
</table>

No $\delta'$ (Al$_3$Li)

$T_1$ (Al$_2$CuLi) is primary strengthening phase, distribution is more homogeneous than in 2090

TEMPERS

(1) 290°F/20 hrs
(2) 290°F/30 hrs
(3) 290°F/20 hrs + ramp to 400°F at 50°F/hr + 400°F/5 min
PROPOSED WORK - ELECTROCHEMISTRY, PITTING

(1) Examine parameters: passive current density
    open circuit potential
    breakaway potential
    each as a function of composition and temper.

(2) Are there preferential sites for pit initiation?
    Inclusions?
    $T_1$ precipitates?

(3) Will pitting or intergranular corrosion be observed
    below the breakaway potential of the matrix phase due
    to $T_1$ dissolution?
    Will such pitting be arrested once exposed $T_1$ is
    depleted?
PROPOSED WORK - SCC BEHAVIOR

(1) Does anodic dissolution play a key role in crack growth?

(2) Will homogeneous Ti distribution result in better SCC resistance?

(3) Is there a Cu depleted zone and what contribution does it have to SCC?

(4) Will the absense of ε/ affect SCC behavior?

Alternate Immersion testing at Reynolds Corporation.

Constant load TTF testing w/applied potentials.

Comparison of behavior to alloy 2090.
Program 6  The Effect of Zinc Additions on the Environmental Stability of Alloy 8090 (Al-Li-Cu-Mg-Zr)

Raymond J. Kilmer and G.E. Stoner

Objectives

The objectives for this PhD research are to document and characterize the effects that Zn additions have on the microstructure of alloy 8090 under different aging conditions and to correlate SCC behavioral changes with changes in alloy composition and microstructure. As an extension of this goal, emphasis will be placed on optimizing SCC behavior and alloy density.
The Effects of Zn Additions on the Microstructure and SCC Performance of Alloy 8090

R.J. Kilmer
G.E. Stoner

Stress corrosion cracking (SCC) remains a problem in both Al-Li and conventional Al-heat treatable alloys. It has recently been found that relatively small additions (< ~1 wt-%) of Zn can dramatically improve the SCC performance of alloy 8090 (Al-Li-Cu-Mg-Zr). Constant load time to failure experiments using cylindrical tensile samples loaded between 30 and 85% of TYS indicate improvements of orders of magnitude over the baseline 8090 for the Zn-containing alloys under certain aging conditions. However, the toughnesses of the alloys were noticeably degraded due to the formation of second phase particles which primarily reside on grain and subgrain boundaries. EDS revealed that these intermetallic particles were Cu and Zn rich. The particles were present in the T3 condition and were not found to be the result of quench rate, though their size and distribution were.

At 5 hours at 160°C the alloys displayed the greatest susceptibility to SCC but by 20 hours at 160°C the alloys demonstrated markedly improved TTF lifetimes. Aging past this time did not provide separable TTF results however, the alloys toughnesses continued to worsen. Initial examination of the alloys microstructures at 5 and 20 hours indicated some changes most notably the S' and <£) distributions. It was further noticed that Zn additions appear to increase the number fraction of S' precipitating out from the alloys. A possible model by which this may occur will be explored.

Polarization experiments indicated a change in the trend of E(B) and passive current density at peak aging as compared to the baseline 8090. Initial pitting experiments indicated that the primary pitting mechanism in chloride environments is one occurring at constituent (Al-Fe-Cu) particles and that the Cu and Zn rich boundary precipitates posses a breakaway potential similar to that of the matrix acting neither anodic or cathodic in the first set of aerated 3.5 w/o NaCl experiments.

Future work will focus on identification of the second phase particles, evaluation of K_{Scc} and plateau da/dt via both DCB and slow strain rate techniques. A lower Zn content variant will be examined in the near future in the hopes of optimizing toughness, density and SCC performance.

K_{Scc}

K_{Scc}

K_{Scc}
The Effect of Zn Additions on the Microstructure and SCC Performance of Alloy 8090

R.J. Kilmer
G.E. Stoner

Center for Electrochemical Sciences and Engineering
Department of Materials Science
University of Virginia
Charlottesville, Virginia 22901

Sponsored by NASA, Langley Research Center, Hampton Virginia

NASA Contact: B.A. Lisagor

Co-sponsored by Alcoa Technical Center, Alcoa Center PA

Alcoa Contact: J.M. Newman
<table>
<thead>
<tr>
<th>Alloy Code</th>
<th>Li</th>
<th>Cu</th>
<th>Mg</th>
<th>Zn</th>
<th>Zr</th>
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<td>Alloy A</td>
<td>2.53</td>
<td>1.22</td>
<td>0.67</td>
<td>1.36</td>
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<td>Alloy B</td>
<td>2.47</td>
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<td>Alloy C</td>
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<td>1.23</td>
<td>0.49</td>
<td>1.00</td>
<td>0.12</td>
</tr>
<tr>
<td>Alloy D</td>
<td>2.55</td>
<td>1.16</td>
<td>0.69</td>
<td>0.02</td>
<td>0.12</td>
</tr>
</tbody>
</table>
Alloy C

S-T

10μm

ORIGINAL PAGE IS OF POOR QUALITY
Quench Sensitivity

Alloy C  SHT at 545 °C for 30 minutes
Aged 5 hours at 160 °C

Air quench after SHT

Cold water quench after SHT
Aging Time

Material Property

SCC Resistance

Strength

Toughness
<table>
<thead>
<tr>
<th>Alloy Code</th>
<th>Aging Condition</th>
<th>Li</th>
<th>Cu</th>
<th>Mg</th>
<th>Zn</th>
<th>$K_{IC}$ (S-T) (ksi·in$^{-1}$)</th>
<th>TYS (ksi)</th>
</tr>
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<tr>
<td>Alloy A</td>
<td>5 hr @ 160C</td>
<td>2.53</td>
<td>1.22</td>
<td>0.67</td>
<td>1.36</td>
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<td></td>
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<td>2.53</td>
<td>1.22</td>
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<td>0.99</td>
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<td>1.23</td>
<td>0.74</td>
<td>0.99</td>
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<td>56.4</td>
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<tr>
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<td>0.49</td>
<td>1.00</td>
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<td>0.49</td>
<td>1.00</td>
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<tr>
<td>Alloy D</td>
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<td>0.02</td>
<td>10.7</td>
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### 8090 and 8090 + Zn Variants

**Alternate Immersion Testing (ASTM G-49)**

30 day tests, 1/4" tensile samples

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<tr>
<th>Alloy Code</th>
<th>Aging at 160C (hrs)</th>
<th>20 ksi</th>
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<th>30 ksi</th>
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<th>40 ksi</th>
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<tr>
<td></td>
<td></td>
<td>F/N</td>
<td>Days</td>
<td>F/N</td>
<td>Days</td>
<td>F/N</td>
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#### Alloy A

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<td>------</td>
<td>N/A</td>
</tr>
<tr>
<td></td>
<td>40</td>
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<tr>
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<td>0/3</td>
<td>------</td>
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<tr>
<td></td>
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<td>0/3</td>
<td>------</td>
<td>N/A</td>
<td>0/3</td>
<td>------</td>
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<tr>
<td></td>
<td>100</td>
<td>0/3</td>
<td>------</td>
<td>N/A</td>
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#### Alloy B

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#### Alloy D

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</table>

* indicates sample fractured at threads
**Microstructure**

5 hr @ 160°C
- small or no δ' free zones
- no S' apparent
- boundary ppts
- solute distribution (?)

20 hr @ 160°C
- well developed δ'-FZ
- S' well developed
- boundary ppts
δ' distribution in alloy A after 20 hr at 160°C
Alloy A
20 hrs at 160C
Precipitate D.F.
Effects of alloying on the corrosion behavior of Aluminum

\[ \text{Potential (volts)} \]

\[ E_{br} \]

\[ i_{pass} \]

\[ \log i (\text{amps/cm}^2) \]
Alloy B - T3

3.5 w/o NaCl
Aerated

0 min

30 min

75 min

* Alloy was lightly etched in Keller's etchant
Future Work

* DCB Samples to evaluate $K_{1_{\text{SCC}}}$ and plateau da/dt
* Compare/Contrast with slow strain rate approach
  ** Environmental changes
* Scanning auger spectroscopy
* Identification of precipitates via CBED
  ** Casting
* Different 8090 + Zn variant(s) to attempt improving:
  ** toughness
  ** maintain improved SCC performance
** Correlation between microstructure and SCC behavior **
SCC Testing of 8090 + Zn Alloys

Approach

* Evaluate SCC performance of 8090 and 8090 + Zn alloys in S-T orientation

Method

* Using a constant displacement rate technique, determine, rank and quantify pertinent SCC characteristics of these alloys and correlate the results with associated microstructure. These techniques should allow for determination of $K_{th}$ and the plateau cracking velocities.

Experimental

* (See attached figures)

* Sample will be a cylindrical S-T specimen with an EDM induced chord across the edge of the sample followed by fatigue pre-cracking via stepped load shedding (decreasing K) so that final $K_{max} < K_{scc}$.

* The test will be run under free corrosion potential.

* Grips will be pin loaded to allow for rotation.

* Displacement rates will be varied.

* $da/dt$ will be continuously measured via a DC potential drop technique.

* $K_{th}$ vs. displacement rate will be plotted for air and 3.5 NaCl environment to aid in determining appropriate displacement rate.

* $K$ vs. $da/dt$ will be plotted for a number of microstructures (i.e vs. aging time and temp.). Plateau cracking velocities and threshold K's will be determined.

* $K_{th}$ and plateau cracking velocities can be compared/contrasted with DCB tests.
Figure 1. Schematic of load cell for constant displacement rate and constant load experiments
S' and T₁ Distribution in Alloys A-C after 100 hrs at 160°C

Alloy A

Alloy B

Alloy C
* Zn incorporated into delta prime increasing the interfacial mismatch

* area adjacent to delta prime enriched in vacancies, Cu and Mg, creating environment conducive to $S'$ nucleation

* heterogeneous nucleation site whose competitiveness increases as the degree of stretch decreases and the degree of solute supersaturation increases
Objective

The objective of this work is to develop a fundamental understanding of the effects of dissolved and trapped hydrogen on the mechanical properties of selected Al-Li-Cu-X alloys. We propose to: (a) distinguish hydrogen induced EAC from aqueous dissolution controlled processes, (b) correlate hydrogen induced EAC with mobile and trapped hydrogen concentrations and (c) identify significant trap sites and hydrides (if any) through the utilization of model alloys and phases.
Hydrogen Interactions in Aluminum Lithium Alloys

John R. Scully
and
Stephen W. Smith

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

This program seeks to develop a fundamental understanding of the effects of dissolved and trapped hydrogen on the mechanical properties of selected Al-Li-Cu-X alloys. We propose to (a) distinguish hydrogen induced EAC from aqueous dissolution controlled EAC, (b) correlate hydrogen induced EAC with mobile and trapped hydrogen concentrations, and (c) identify significant trap sites and hydride phases (if any) through utilization of model alloys and phases. A review of the literature indicates three experimental factors which have impeded progress in the area of hydrogen EAC for this class of alloys. These are: (i) inter-subgranular fracture in Al-Li alloys when tested in the S-T orientation in air or vacuum make it difficult to readily detect hydrogen induced fracture based on straight forward changes in fractography; (ii) the inherently low hydrogen diffusivity and solubility in Al alloys is further compounded by a native oxide which acts as a hydrogen permeation barrier; these factors complicate hydrogen detection and measurement; and (iii) hydrogen effects are masked by dissolution assisted processes when mechanical testing is performed in aqueous solutions. This program will attempt to circumvent these experimental barriers through the use of novel breaking load, hydrogen analysis, and metallurgical techniques. The intended approach and current program status is reviewed.
Hydrogen Interactions in Aluminum Lithium Alloys

John R. Scully
and
Stephen W. Smith

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

Sponsored by NASA-Langley Research Center. Pending support from the Alcoa Technical Center Foundation Program
Objective

We seek to develop a fundamental understanding of the effects of dissolved and trapped hydrogen on the mechanical properties of selected Al-Li-Cu-X alloys. We propose to (a) distinguish hydrogen induced EAC from aqueous dissolution controlled processes, (b) correlate hydrogen induced EAC with mobile and trapped hydrogen concentrations, (c) identify significant trap sites and hydrides (if any) through the utilization of model alloys and phases.
Experimental Problems associated with Investigation of Hydrogen Effects in Al-Li-Cu-X Alloys

1. **Problem:** Low fracture toughness is observed for aged Al-Li-Cu-X alloys stressed in the S-T orientation. Intergranular and inter-subgranular fracture occurs in air or in vacuum making it difficult to detect hydrogen assisted fracture on the basis of fractography. **Solution:** Test specimens in L-T orientation after appropriate aging to produce shear band cracking in air or vacuum.

2. **Problem:** Low hydrogen diffusivity and solubility in aluminum alloys is compounded by an oxide permeation barrier. These factors make hydrogen analysis difficult. **Solution:** Use thermal desorption spectroscopy and Pd coated samples.

3. **Problem:** Aqueous EAC response may be dominated by dissolution assisted processes even during cathodic charging. This tends to mask hydrogen effects. Fractographic features distorted by dissolution. **Solution:** Use modification of method originally described by Gruhl and co-workers or use Pd coated breaking load samples.
Approach

1. Age alloys to produce shear band fracture in air.

2. Perform modified breaking load and slow strain rate tests using pre-charged Al-Li-Cu-X alloys in L-T orientation. Use Pd coated samples with native oxide removed by sputter etching. Use alloy with a known hydrogen response (i.e. 7075-T6) as a control.

3. Analyzed fracture surfaces using "advanced" methods

4. Conduct hydrogen analysis on hydrogenated model alloys as well as Al-Li-Cu-X alloys specimens:
   a) Modified Devanathan-Stuchurski permeation method
   b) Thermal desorption spectroscopy
   c) hydride detection methods
   d) nuclear methods
Fabrication of Flat Tensile Bar in L-T Orientation for Breaking Load Studies
Constant Deflection Apparatus for use in Breaking Load studies
Advantages to Using Palladium Coatings

- Can remove \( \text{Al}_2\text{O}_3 \) layer, which impedes hydrogen diffusion.

- Surface of specimen will not be affected by cathodic charging.

- Can distinguish between aqueous and hydrogen effects.

ex.

```
\begin{center}
\begin{tikzpicture}
\draw[thick] (0,0) rectangle (4,2);\node at (2,1) {\text{hydrogen assisted cracking}};
\draw[thick] (0,0.5) rectangle (4,1.5);\node at (2,1) {\text{film rupture}};
\draw[thick] (0,0.5) rectangle (4,1.5);\node at (2,1) {\text{aqueous effect}};
\end{tikzpicture}
\end{center}
```
Etching of an Aluminum Alloy Substrate by dc Sputter Etching
Deposition of Palladium by dc Sputtering

Diagram showing the process of palladium deposition by dc sputtering, including the flow of ions (Ar⁺) and electrons (e⁻), and the various components such as the target, substrate, gas inlet, cooling, and vacuum line.
Tensile Specimen for Modified Breaking Load Studies

Notched Surface:

Uncoated.
Exposed to lab air and/or vacuum.

Back Surface:

Coated with Pd.
Cathodically charged.
## Test Matrix for Breaking Load Studies

<table>
<thead>
<tr>
<th>Material</th>
<th>Orientation</th>
<th>Treatment</th>
<th>Fractography at Applied Load</th>
</tr>
</thead>
<tbody>
<tr>
<td>7075 - T6</td>
<td>L - T</td>
<td>Glove Box / Lab Air</td>
<td>slip band  slip band  slip band</td>
</tr>
<tr>
<td>2090 -</td>
<td>L - T</td>
<td>Glove Box / Lab Air</td>
<td>slip band  slip band  slip band</td>
</tr>
<tr>
<td>8090 -</td>
<td>L - T</td>
<td>Glove Box / Lab Air</td>
<td>slip band  slip band  slip band</td>
</tr>
<tr>
<td>7075 - T6</td>
<td>L - T</td>
<td>Cathodic Charging</td>
<td>to be determined</td>
</tr>
<tr>
<td>2090 -</td>
<td>L - T</td>
<td>Cathodic Charging</td>
<td>to be determined</td>
</tr>
<tr>
<td>8090 -</td>
<td>L - T</td>
<td>Cathodic Charging</td>
<td>to be determined</td>
</tr>
</tbody>
</table>
## Hydrogen Analysis Methods Pertinent to Aluminum-Lithium Alloys

<table>
<thead>
<tr>
<th>Method</th>
<th>Advantage</th>
<th>Facility</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Devanathan</td>
<td>Inexpensive</td>
<td>U.Va-CESE</td>
<td>Favors alloys with permeability and mobile H</td>
</tr>
<tr>
<td>Stuchurksi</td>
<td>Significant</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Permeation</td>
<td>Experience</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nuclear Reaction</td>
<td>Absolute conc.</td>
<td>Sandia</td>
<td>Not readily avail. trapped+mobile D must use D$_2$O</td>
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<tr>
<td>$^3$He(d,p)$^4$He</td>
<td>depth profile</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>used for Al</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Thermal Desorption</td>
<td>relative conc.</td>
<td>U.Va-CESE</td>
<td>proven for Al-Li must use D$_2$O</td>
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<td>Spectroscopy</td>
<td>Spectroscopic</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>assess trap strength</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Neutron Act.</td>
<td>thick samples</td>
<td>U. Va.-Nuclear</td>
<td>quantitative qualitative</td>
</tr>
<tr>
<td>Neutron Rad.</td>
<td>&quot;</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Hydrogen Concentration Profile During Hydrogen Permeation Studies

Charging side:
hydrogen reduction

\[ C_H = f(n_c) \]

\[ H^+ + e^- \rightarrow H \]

Exit side:
hydrogen oxidation

\[ C_H \rightarrow 0 \]

\[ H \rightarrow H^+ + e^- \]
Schematic of Devanathan–Stachurski Permeation Cell
Schematic of Differential Permeation Cell

charging side

control cell

exit side

CE  RE  WE

potentiostat/galvanostat

WE2

WE1  RE  CE

bipotentiostat
Hydrogen Transport in Endothermic Hydrogen Absorbers (Fe) is a Strong Function of The Nature and Density of Traps

\[ K = \frac{N_1 k_r}{p_r} = \exp\left(-\frac{E_B}{RT}\right), \quad \text{k is the trap rate, p is the release rate} \]

\[ N_1 \text{ (lattice sites)} = 2.6 \times 10^{23} \text{ octahedral sites/cm}^3 \text{ for BCC iron} \]

\[ E_B > E_s \text{ Strong Trap (~29 KJ/mole)} \]
A Series of Permeation Rise and Decay Transients is Utilized to Separate Reversible from Irreversible Trapping

\[ \frac{I_{CH2}}{I_{CH1}} = 1 - \frac{2}{\sqrt{\pi t}} \cdot \exp \left( \frac{-1}{4\tau} \right) \]

\[ \frac{I-I_{1}}{I_{2}-I_{1}} = \frac{2}{\sqrt{\pi t}} \cdot \exp \left( \frac{-1}{4\tau} \right) \]

REARRANGING:

\[ \log \left( I_{CH1} - I \right) \sqrt{t} = \log \frac{2F \sqrt{D} (C_{1} - C_{2})}{\sqrt{\pi}} - L^2 \log e \times \frac{1}{4D} \cdot \frac{1}{t} \]

\[ \log \left( I_{1} - I \right) \sqrt{t} = \log \frac{2F \sqrt{D} (C_{2} - C_{1})}{\sqrt{\pi}} - L^2 \log e \times \frac{1}{4D} \cdot \frac{1}{t} \]

WHERE: \( \tau = D t / L^2 \)
Diffusion coefficient of Hydrogen in various Al alloys

Temp. (K)

$D$ (cm$^2$/sec)

$1/T \times 10^4$ (1/K)

- Al-1% Li
- Al-2% Li
- Al-3% Li
- 99.99% Al
- Al-2Li extrapolated to RT

Anyalebechi

Watson & Meshii
for 99.99% Al
Design of Thermal Desorption Spectroscopy system

Thermal Desorption Spectroscopy Offers Several Advantages

Constant Temperature

Temperature Ramp
Solubility of Hydrogen in Al alloys at 10,000 atm.

Hydrogen Solubility

mol H / cm metal

1/T x 10^4 K⁻¹

195
# Hydrogen Trapping

<table>
<thead>
<tr>
<th>Model Alloy</th>
<th>Exploitable Trap Site</th>
<th>Possible Trapping - Interaction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al - 3Li</td>
<td>$\delta, \delta', \text{Li}_{ss}$</td>
<td>$\delta'$-interphasal; $\delta, \delta'$-hydride; Li-solute</td>
</tr>
<tr>
<td>Al - 3Cu</td>
<td>$\theta^+, \theta^-, \theta, \text{Cu}_{ss}$</td>
<td>$\theta^+, \theta^-$-interphasal; $\theta$-void</td>
</tr>
<tr>
<td>Al - Li-Cu</td>
<td>$\delta', T_1, T_2$</td>
<td>$\delta', T_1, T_2$-hydride; $T_1$-interphasal; $T_2$-void</td>
</tr>
<tr>
<td>$T_1$</td>
<td>$T_1$</td>
<td>$T_1$-hydride</td>
</tr>
<tr>
<td>Al</td>
<td>g.b., vacancies, voids, microvoids</td>
<td>interfacial, point defects, voids</td>
</tr>
<tr>
<td>Al - Zr</td>
<td>$\beta, \beta'$</td>
<td>$\beta$-void; $\beta'$-interphasal; $\beta, \beta'$-hydrides</td>
</tr>
<tr>
<td>Al$_2$Zr ($\beta'$)</td>
<td>$\beta'$</td>
<td>$\beta'$-hydride</td>
</tr>
<tr>
<td>Al - Li - Zr</td>
<td>$\delta'$ coats $\beta'$</td>
<td>interphasal; $\delta', \beta'$-hydride</td>
</tr>
</tbody>
</table>
Program Status - June 1991

1. Hydrogen permeation cells assembled. Thin foils must be prepared.

2. Breaking load configuration built and tested. Specimen exposures to begin this summer.

3. Thermal desorption system in design stages. Equipment purchases to follow.

4. Hydrogen evolution reaction kinetics studies to be undertaken in July to ascertain hydrogen production capability of model alloys and phases.
Objective

The objective of this study is to investigate the effect of thermal exposure (isothermal and thermal cycling) on the longitudinal and transverse tensile properties of Ti 1100/SCS-6 composites. The property degradation will be correlated to microstructural changes in the matrix, fibers and interface.
The objective of the present study is to evaluate the influence of thermal exposure, both isothermal and cyclic, on the reaction kinetics, mechanical properties and fracture behavior of Ti-1100 alloy/SiC fiber composites.

During the last reporting period, it was determined that composites made with TiB₂ coated SiC fiber (Sigma) reacted at the same rate as SCS-6 fibers. As a result, the thinner surface coating on the Sigma fiber was completely consumed at shorter times than that on the SCS-6.

Thermal cycling experiments were conducted on longitudinal and transverse Ti-1100/SCS-6 composites over a temperature range of 150–800°C for 500 cycles. The thermal exposures were carried out in air and in argon. No appreciable tensile strength degradation was observed for samples cycled in argon, although a strength loss was noted for the samples cycled in air. Fracture surface characterization showed brittle matrix failure in regions near the surface and in regions where there was a path for oxygen ingress.
RESEARCH OBJECTIVE

The objective of this research is to investigate the influence of thermal exposure, both isothermal and cyclic, on the microstructure and mechanical properties of Ti–1100/SiC composites.
SCS-6 AND SIGMA IN Ti-1100
AS-FABRICATED

SCS-6

SIGMA
REACTION RATES OF SCS-6 AND SIGMA IN TI-1100 AT 800 AND 900°C

![Graph showing reaction rates of SCS-6 and Sigma in Ti-1100 at 800 and 900°C](image)

- **SCS-6 As Fab.**
- **Sigma As Fab.**
- **Sigma 900C**
- **SCS-6 900C**
- **Sigma 800C**
- **SCS-6 800C**

Reaction Zone Thickness, (µm)

(Exposure Time)^{1/2}, (hrs^{1/2})
SCS-6 AND SIGMA IN Ti-1100 REACTED AT 900°C FOR 50 HOURS

SCS-6

SIGMA
Ti-1100/SCS-6 composite

200 μm
RESIDUAL THERMAL STRESSES

ELASTIC THERMAL STRESS MODEL
Ti3Al + Nb/SCS-6

Matrix
Fiber

\[ \sigma_z, \sigma_\theta, \sigma_r \]

- Stress (MPa)
- Distance from fiber axis (microns)

Graph showing stress distribution with labels for hoop, axial, and radial stresses.
THERMAL CYCLING APPARATUS

- Chain Driven System
- Thermocouple Wire
- Reversible Motor
- Chain Driven System
- Sample Holder
- Fluidized Cooling Bed
- Resistance Furnace
- Control Panel
- Pressurized Air to Fluidized Bed

Graph:
- Temperature (°C)
- Time (min)
- Temperature increases sharply from 0 to 900°C in approximately 2 minutes, then decreases sharply to 0°C in the following minute.
THERMAL EXPOSURES

- Panels fabricated at NASA LaRC

- Tensile coupons cut prior to exposure:
  Longitudinal, Transverse, Matrix alone

- Thermal exposures:
  Cycled in air 150 to 700°C 500X
  Cycled in air 150 to 800°C 500X
  Cycled in argon 150 to 800°C 500X

- Samples tensile tested (2 per condition)
MATRIX TENSILE TESTS

<table>
<thead>
<tr>
<th></th>
<th>AS FAB</th>
<th>150-700°C 500X AIR</th>
<th>150-800°C 500X AIR</th>
<th>150-800°C 500X ARGON</th>
</tr>
</thead>
<tbody>
<tr>
<td>MODULUS (GPa)</td>
<td>112</td>
<td>107</td>
<td>103</td>
<td>113</td>
</tr>
<tr>
<td>UTS (MPa)</td>
<td>1023</td>
<td>1038</td>
<td>910</td>
<td>982</td>
</tr>
</tbody>
</table>
Ti-1100/SCS-6 INTERFACE

150-800C 500X Air

As-Fabricated

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ORIGINAL PAGE IS OF POOR QUALITY
TRANSVERSE SURFACE CRACK

Tested Longitudinal Sample
Cycled in Air 500X 150–800C
SURFACE CRACKING
(0.8% TENSILE STRAIN)

Composite
As-Fabricated

Composite
Air 150-700°C 500X

Composite
Air 150-800°C 500X

Matrix
Air 150-800°C 500X
FRACTURE SURFACES

Longitudinal Samples

As-Fabricated

Air 150-800°C 500X
FRACTURE SURFACES

Transverse Samples

As-Fabricated

Air 150–800°C 500X
SUMMARY

- After fabrication the Sigma fiber reacted at nearly the same rate as SCS-6

- Thermal cycling in argon had no significant effect on the composite tensile properties

- Thermal cycling in air up to 700°C shows little decrease in tensile strength, but cycling to 800°C had an appreciable effect

- Fracture surface characterization indicates that oxidation of the matrix leads to a localized brittle failure mode
FUTURE WORK

- Investigate matrix microstructural changes
- Compare thermal cycling to equivalent isothermal exposure
- Compare to composite of BETA 21S
- Cryogenic thermal cycling
Objective

The objective of the present investigation is development of a broadly-applicable method for the quantitative analysis and description of the spatial distribution of second phase particles. This method should characterize the spatial distribution and its inhomogeneities in a manner which is of use to the materials scientist. As the method is intended to be available to a wide range of researchers in materials science, it has been designed to operate on a desktop computer. A second objective is application of the method for characterizing second phase particle distributions in a materials processing problem. The problem we have selected in consultation with the NASA technical monitor is understanding the effect of consolidation processing parameters on oxide particle distributions in a PM aluminum alloy.
A GEOMETRICAL DESCRIPTION OF MICROSTRUCTURE
WITH APPLICATION TO Al PM-PROCESSING

Joseph B. Parse
Prof. J.A. Wert
(faculty advisor)

Funded by NASA-Langley
(Light Aerospace Alloys and Structures Technology Program)

Inhomogeneities in the spatial distribution of second phase particles in engineering materials are known to affect certain mechanical properties. Progress in this area has been hampered by the lack of a convenient method for quantitative description of the spatial distribution of the second phase. The objective of this effort is the development of a widely accessible technique for the quantitative characterization and description of the spatial distribution of second-phase particles.

The Dirichlet tessellation technique (a geometrical method for dividing an area containing an array of points into a set of polygons uniquely associated with the individual particles) has been selected as the basis of an analysis technique, which has been implemented on a PC. The analysis characterizes properties of individual particles; such as local particle density, near-neighbor distances, etc.; and describes inhomogeneities such as clustering in the particle distribution.

This analysis technique is being applied to the production of Al sheet by PM-processing methods; vacuum hot-pressing, forging and rolling. The effect of varying hot working parameters (forging reduction, deformation temperature and total reduction) on the spatial distribution of aluminum oxide particles in consolidated sheet is being investigated. Changes in distributions of properties such as through-thickness near-neighbor distance correlate with hot-working reduction.
A GEOMETRICAL DESCRIPTION OF MICROSTRUCTURE
WITH APPLICATION TO Al PM-PROCESSING

Joseph B. Parse
(Graduate Student)

Prof. J. A. Wert
(Faculty Advisor)

Funded by NASA Langley
Light Aerospace Alloy and Structures Technology Program

(7/10/91)
INTRODUCTION

- The spatial distribution of second-phase particles in materials is known or predicted to affect properties such as toughness, modulus, and strength.

- Progress in this area has been hindered by the lack of a convenient method for quantitative characterization of the spatial distribution of second-phase particles.

- The objective of this effort is the development of a widely accessible technique for the quantitative characterization of the spatial distribution of second-phase particles.

- The Dirichlet tessellation technique was selected as the basis for the analysis.
PREVIOUS WORK IN MATERIALS SCIENCE:

- Previous work in this area has focused on the relationship between microstructure and properties.

- Embury found that clusters of second-phase particles were preferred sites for damage initiation and accumulation.

- Liu and coworkers studied effects of clustering of SiC particles in 7XXX-series MMC's; fracture experiments revealed that clustered sites were preferred sites for damage initiation.
IMPLEMENTATION:

- Implement analysis on a PC
  - generate tessellations
  - generate distribution of properties (polygon areas, near-neighbor distances, etc.)
  - verify operation with computer-generated point arrays
Tessellations: Random

Polygons Area: Random

Max.-Nbr. Distance: Random

Clustered

Clustered

Clustered
EXPERIMENTAL PROGRAM:

INVESTIGATE RELATIONSHIP BETWEEN PROCESSING PARAMETERS AND SPATIAL DISTRIBUTION OF SECOND-PHASE PARTICLES

Material and process:
- use RS Al-Mn-Si PM-Process sheet as model material
- alloy system / process is under investigation by NASA - Langley
- typical processing sequence:
  i) gas atomize powder (≈27μm)
  ii) cold compaction (to ≈70% density)
  iii) vacuum hot press
  iv) forge and roll to ≈95% reduction
- distribution of oxide as affected by hot-working path (forging & rolling) is thought to affect mechanical properties such as toughness.
Experimental variables:

- Duplicate processing procedures used at NASA with small samples
  - powder produced at NASA-Langley
  - consolidation and hot working performed at UVA
- Vary hot working parameters subsequent to consolidation.
  - temperature
  - forging reduction
  - total reduction
- Analyze oxide particle distributions
- Select processing conditions for production of larger samples at NASA for mechanical property measurements
Experimental Conditions for Analysis
As Hot-Pressed

70% Forged

10 µm

70% Forged + Rolled to 95%
Tessellations: Forged 70%

Forged 70%; Rolled to 95%

Polygon Area: Forged 70%

Forged 70%; Rolled to 95%

Through-thickness near-nbr. Distance: Forged 70%

Forged 70%; Rolled to 95%
EXPERIMENTAL RESULTS

- Evolution of oxide distribution during hot-working

  -> gradual breakup and dispersion of oxide during deformation; prior particle boundaries still distinguishable at 70% forging reduction

  -> striking difference in oxide distribution between 70% forging reduction and subsequent rolling to 95% total reduction

  -> difficult to discern prior particle boundaries at 95% reduction

- Results of analysis

  -> distributions of polygon area show little change between 70% forging reduction and subsequent rolling to 95% reduction

  -> distributions of through-thickness nearest-neighbor distance show significant differences corresponding to reduction in thickness and elimination of prior particle boundaries
CONCLUSIONS AND RECOMMENDATIONS:

- Preliminary application of analysis to a model material has been made.

- Microstructural differences resulting from processing conditions can be quantitatively characterized by this technique.

- Future efforts will focus on analyzing the effects of hot-working on the spatial distribution of oxide particles for a wide range of hot-working procedures.
Objectives

The long-term objective of this investigation is aimed at attaining a complete understanding of the inelastic response of metal matrix composites subjected to arbitrary, biaxial load histories. The core of the research program is a series of biaxial tests conducted on different types of advanced metal matrix composite systems using the combined axial/torsional hydraulic load frame in the Composite Mechanics Laboratory at the University. These tests involve primarily tubular specimens and include tension, compression, torsion and combinations of the above load histories in order to critically assess the inelastic response of advanced metal matrix composites in a wide temperature range.
Inelastic Response of SCS-6/Ti-15-3 Metal Matrix Composite

C. J. Lissenden, C. T. Ilarakiovich & M-J Pindera

ABSTRACT

Theoretical predictions and experimental results have been obtained for inelastic response of unidirectional and angle-ply composite tubes subjected to axial and torsional loading. The composite material consists of silicon carbide fibers (40% fiber volume fraction) in a titanium alloy matrix. This particular material system is known to be susceptible to fiber/matrix interfacial damage. A method to distinguish between matrix yielding and fiber/matrix interfacial damage is suggested.

Biaxial tests have been conducted on the two different layup configurations using an MTS Axial/Torsional test frame with a PC based data acquisition system. To date two \( [0_4] \) tubes and one \( [\pm45]_t \) tube have been tested. The experimentally determined elastic moduli of the SiC/Ti system are compared with those predicted by a micromechanics model. The test results indicate that fiber/matrix interfacial damage occurs at relatively low load levels and is a local phenomenon.

The micromechanics model used is the method of cells originally proposed by Aboudi. This model has the capability to generate the effective response of metal matrix composites in the linear and inelastic range in the presence of imperfect bonding between the fiber and matrix. The model has also been used to determine initial yield surfaces for comparison with experimental results and as a guide in the selection of the biaxial loadings to be considered.

Finite element models using the ABAQUS finite element program have been employed to study end effects and fixture/specimen interactions. The finite element studies have shown large stress concentrations near the fixtures.

The results to date have shown good correlation between theory and experiment for response prior to the initiation of damage. The correlation is less satisfactory after damage occurs. Damage is evident in the tests from the stress-strain results and audible acoustic events. Damage has been observed to occur before the onset of matrix yielding.

Low axial strength obtained for the \( [0_4] \) tube has been attributed to the stress concentrations associated with the fixture.
Inelastic Response
of SCS-6/Ti-15-3
Metal Matrix Composite

by
C.J. Lissenden
C.T. Herakovich
M.J. Pindera

NASA Technical Monitor:
W.S. Johnson

July, 1991
NOMINAL TUBE GEOMETRY

\[ X = \text{S.G.} \]

<table>
<thead>
<tr>
<th>([0]_4)</th>
<th>([\pm 45]_s)</th>
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<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
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</thead>
<tbody>
<tr>
<td>(R_i(\text{in}))</td>
<td>0.718</td>
</tr>
<tr>
<td>(R_o(\text{in}))</td>
<td>0.750</td>
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<tr>
<td>(A(\text{sq in}))</td>
<td>0.148</td>
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<td>(J(\text{in}^4))</td>
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<tr>
<td>(v_1)</td>
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<td>(J(\text{in}^4))</td>
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<tr>
<td>(J(\text{in}^4))</td>
<td>0.4</td>
</tr>
</tbody>
</table>
SCS-6/Ti-15-3 Tube
Manufacturing Process

1. Lay fibers flat
2. Weave fibers and 2 mil molys
3. Wrap foil around mandrel
4. Wrap fiber cloth around foil
5. Continue laying up foil and fiber cloth
6. Enclose in steel tube
7. Hot press at 1800 °F and 15 ksi
8. Cool
9. Machine
10. Dip in liquid bath
SERIES 319
AXIAL TORSIONAL LOAD UNIT
# Predicted Elastic Properties

## Perfect Bond \((R_n = 0, R_t = 0)\)

<table>
<thead>
<tr>
<th></th>
<th>(E_{xx}) (msi)</th>
<th>(G_{xy}) (msi)</th>
<th>(G_{12}) (msi)</th>
<th>(v_{xy})</th>
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</thead>
<tbody>
<tr>
<td>([0]_4)</td>
<td>31.2</td>
<td>7.92</td>
<td>----</td>
<td>0.313</td>
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<tr>
<td>([+45]_s)</td>
<td>23.3</td>
<td>10.50</td>
<td>7.86</td>
<td>0.375</td>
</tr>
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</table>

## Total Debond \((R_n = 0.01, R_t = 0.1)\)

<table>
<thead>
<tr>
<th></th>
<th>(E_{xx}) (msi)</th>
<th>(G_{xy}) (msi)</th>
<th>(G_{12}) (msi)</th>
<th>(v_{xy})</th>
</tr>
</thead>
<tbody>
<tr>
<td>([0]_4)</td>
<td>31.2</td>
<td>1.79</td>
<td>----</td>
<td>0.360</td>
</tr>
<tr>
<td>([+45]_s)</td>
<td>6.0</td>
<td>8.1</td>
<td>1.8</td>
<td>0.70</td>
</tr>
</tbody>
</table>
SCS-6/Ti 15-3

$[0]_4 v_f=0.4$

- $R_n=0, R_t=0$
- $\Delta T=-1800^\circ F, R_n=0, R_t=0$
- $R_n=0.01, R_t=0.1$

\[ \tau_{xy}/Y \]
\[ \sigma_{xx}/Y \]
SCS-6/Ti 15-3

$[\pm 45^\circ]_S V_t=0.4$

- $R_n=0, R_t=0$
- $\Delta T=1800^\circ F, R_n=0, R_t=0$
- $R_n=0.01, R_t=0.1$

Graph showing stress distribution with axes labeled $\tau_{xy}/Y$ and $\sigma_{xx}/Y$.
SCS-6/Ti-15-3
as a
Three Phase Composite

- Theoretical Volume Fractions
  - SCS-6 39.4%
  - Ti-15-3 60.4%
  - Molybdenum 0.2%
SCS-6/Ti 15-3 Axial Stress/Strain
(±45), $v_f=0.4$
Specimen#5 Test#9

- □ Rosette#1 $E_m=15.4$msi
- □ Rosette#2 $E_m=16.2$msi
- ○ Rosette#3 $E_m=17.7$msi

![Graph showing the relationship between axial stress (ksi) and strain (%)](image)
SCS-6/Ti 15-3 Shear Stress/Strain
(±45)° v=0.4
Specimen#5 Test#9

- Rosette#1 G_{xy}=8.52 ksi
- Rosette#2 G_{xy}=9.29 ksi
- Rosette#3 G_{xy}=8.33 ksi

\begin{align*}
\tau_{xy} (\text{ksi}) & \, \text{vs} \, \gamma_{xy} (\%) \\
0.0 & \quad 0.00 E-01 \quad 0.0 \quad 0.010 \quad 0.020 \quad 0.030
\end{align*}
SCS-6/Ti 15-3 [+45] Specimen #5
Axial Modulus (Loading)

PERFECT BOND (P.B.) PREDICTION

TOTAL DEBOND (T.D.) PREDICTION

Test #
SCS-6/Ti 15-3 [-45], Specimen #5
Shear Modulus

Test #

Gxy (mksi)

Rosette 1
Rosette 2
Rosette 3
SCS-6/Ti 15-3 Axial Stress/Strain

\((0_\text{A}) \nu_1=0.4\)

Specimen#2 Test#11

- - Rosette#1 \(E_m=31.60\text{ksi}, \nu_2=0.265\)
- - Rosette#2 \(E_m=31.96\text{ksi}, \nu_2=0.300\)
- - Rosette#3 \(E_m=32.44\text{ksi}, \nu_2=0.309\)

\(\sigma_{XX} (\text{ksi})\)

\(\varepsilon_{XX} (%)\)
SCS-6/Ti 15-3 Shear Stress/Strain

$(0_4) V_f = 0.4$

Specimen#2 Test#11

- $\tau_{xy}$ vs $\gamma_{xy}$
- Rosette#1 $G_{xy} = 7.764$ msi
- Rosette#2 $G_{xy} = 7.575$ msi
- Rosette#3 $G_{xy} = 7.984$ msi

- $\tau_{xy}$ (ksi)
- $\gamma_{xy}$ (%)

UVA APPLIED MECHANICS

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SCS-6/Ti 15-3 [0]_4 Specimen #2
Axial Modulus (loading)

Test #

E11 (ms)

• Rosette 1
• Rosette 2
• Rosette 3
SCS-6/Ti 15-3 [0]_s Specimen #2
Shear Modulus

G_{12} (ksi)

Test #

P.B. PREDICTION

T.B. PREDICTION

Rosette 1
Rosette 2
Rosette 3
SCS -6/Ti 15-3 [0], Specimen #4
Axial Modulus (Loading)
SCS-6/Ti 15-3 [0], Specimen #4
Shear Modulus (Loading)

G12 (ksi)

Test #

P.B. PREDICTION

Rosette 1
Rosette 2
Rosette 3

T.D. PREDICTION

Tesl#
SCS-6/Ti 15-3 Axial Stress/Strain
(±45) s V_f=0.4
Specimen#5 Test#10

- □ - Rosette#1  F_w=15.4msi
- □ - Rosette#2  F_w=16.1msi
- □ - Rosette#3  F_w=17.5msi

\( \sigma_{XX} \) (ksi)

\( \epsilon_{XX} \) (%)
SCS-6/Ti 15-3 Axial Stress/Strain
(±45), V_f=0.4
Specimen#5 Test#10

- ▼ - Rosette#1 E_u=15.4 msi
- ▲ - Rosette#2 E_u=16.1 msi
- ▼ - Rosette#3 E_u=17.5 msi
SCS-6/Ti 15-3 Axial Stress/Strain
(±45), V_f=0.4
Specimen#5 Test#10

- $\Delta T=0^\circ F, R_n=0, R_s=0$
- Rosette#1 $R_n=15.4$msi
- $\Delta T=0^\circ F, R_n=0.01, R_s=0.1$

\[ \sigma_{XX} \text{ (ksi)} \]

\[ \epsilon_{XX} \text{ (%)} \]
SCS-6/Ti 15-3 Shear Stress/Strain
$(\pm 45)\_s, \nu_f=0.4$
Specimen#5 Test#10

Rosette#1
Rosette#5

$\tau_{xy}$ (ksi)

$\gamma_{xy}$ (%)
GAGE ORIENTATION

Fiber Angle

outer ply fiber direction

G3 G2 G1

JUMP IN MEASURED STRAIN

Data Range

Difference in Measured Strain (%)

1 2 3

G1 G2 G3

UVA APPLIED MECHANICS
SCS-6/Ti 15-3 Shear Stress/Strain

$[0]_4 V_f = 0.4$

Specimen#4 Test#18

- Rosette#1 $G_{xy} = 6.566$ msi
- Rosette#2 $G_{xy} = 7.526$ msi
- Rosette#3 $G_{xy} = 5.718$ msi
- Rosette#4 $G_{xy} = 7.998$ msi
- Rosette#5 $G_{xy} = 8.735$ msi
SCS-6/Ti 15-3

$[0^\circ]_4 V_f=0.4$

- $\Delta T=0^\circ F, R_{n}=0, R_{r}=0$
- $\Delta T=-1000^\circ F, R_{n}=0.5E-5/1E2$
- $\Delta T=-1000^\circ F, R_{n}=3E-5/1E2$
- Specimen#4, Test#17, Rosette#3
- Specimen#4, Test#18, Rosette#3

\[ \begin{array}{ccccccc} 
    \tau_{xy} \text{ (ksi)} & 70.0 & 60.0 & 50.0 & 40.0 & 30.0 & 20.0 & 10.0 & 0.0 \\
    \gamma_{xy} \text{ (%)} & 1.20 & 0.80 & 0.40 & 0.0 & & & & \\
\end{array} \]

UVA
APPLIED
MECHANICS

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Finite Element Model
of $[0]_4$ Tube

Tube Model-1483 nodes
434 quad. axisym. elements
Finite Element Model of [0]_4 Tube

Tube Model Partial Mesh Enlargement
Finite Element Model
of [0]_4 Tube
Displaced Geometry

Displacement due to Axial Loading
Finite Element Model

SCS-6/Ti 15-3

$[0^\circ]_4$ Normalized Stresses

- $\sigma_r/S_r$
- $\sigma_z/S_z$
- $\sigma_{0z}/S_r$
- $\sigma_{rz}/S_{rz}$

Normalized SiC/Ti Stresses at the top of the plug

$S_r = 50$ ksi;
$S_z = 230$ ksi;
$S_{rz} = 42$ ksi.

Normalized SiC/Ti Stresses at the top of the plug
Finite Element Model

SCS-6/Ti 15-3

\[ [0^\circ]_4 \text{ Normalized Stresses} \]

- \( \sigma_r / S_r \)
- \( \sigma_{rr} / S_r \)
- \( \sigma_{\theta\theta} / S_r \)
- \( \sigma_{rr} / S_\tau \)

Normalized SiC/Ti Stresses at the top of the fixture

\( S_r = 40 \text{ ksi} \)
\( S_{\theta} = 230 \text{ ksi} \)
\( S_{\tau\tau} = 42 \text{ ksi} \)
Future Work

- Continue testing tubes
  - 2–[0]_4 tubes
  - 6–[±45]_s tubes
- Enhancements to analytical model
Conclusions

- Good correlation between theory and experiment for elastic moduli of [0]_4 tube

- Poor correlation between theory and experiment for elastic moduli of [±45]_s tube

- Stress concentrations due to the fixture cause failure in the [0]_4 tube during tension tests

- No degradation of moduli with damage

- Damage is a local phenomenon

- Nonlinear response of [±45]_s tube occurred at a stress level of 25% that of predicted initial yielding
Objectives

The initial objective of this project was to investigate the use of superplastically formed corrugated hat-section stringers and frames in place of integrally machined stringers over separate frames for the tanks of large launch vehicles subjected to high buckling loads. The ALS has been used as an example.

The objective of the follow-on project is to study methods of designing shell structures subjected to severe combinations of structural loads and thermal gradients, with emphasis on novel combinations of structural arrangements and materials. Typical applications would be to fuselage sections of high speed civil transports and to cryogenic tanks on the National Aerospace Plane.
SECOND ANNUAL NASA-UVA LAST PROGRAM MEETING

CRYOGENIC TANK BUCKLING ANALYSIS, BENCHMARK TESTS FOR THERMALLY LOADED FINITE ELEMENTS, AND PROPOSED STUDY OF METHODOLOGIES FOR DESIGN OF SHELL STRUCTURES FOR THERMAL ENVIRONMENTS.

W. D. Pilkey, J. K. Haviland, C. Copper, K. McCarthy (UVA)
Dr. M. J. Shuart (NASA)

Department of Mechanical and Aerospace Engineering
School of Engineering and Applied Science
University of Virginia, Charlottesville, VA 22901.
9-10 July, 1991

Research Objectives
Initial: To investigate the use of superplastically formed corrugated hat-section stringers and frames in place of integrally machined stringers over separate frames for the tanks of large launch vehicle subject to high buckling loads. The ALS has been used as an example.

Revised: To study methods of designing shell structures subjected to severe combinations of structural loads and thermal gradients, with emphasis on novel combinations of structural arrangements and materials. Typical applications would be to fuselage sections of high speed civil transports and to cryogenic tanks on the National Aerospace Plane.

Progress on the buckling of tanks of large launch vehicles.
It had been shown previously that superplastically formed corrugated hat-section stringers could replace integrally machined stringers and equivalent beam properties were determined. Using these properties, both for stringers and for frames, the allowable compression loads on a 30 foot diameter tank have been determined and have been shown to be adequate. The proposed design would eliminate a costly machining process and would replace the deep frames which would otherwise have to be built into the tank structure.

Progress on benchmark test
Previous work in this area has been performed by MacNeil and Harder, but they did not include thermal loading or the effect of thermal loading on material properties. The MacNeil/Harder tests are being revised to include thermal loads and new tests are being derived. The "exact" solutions for comparison are from closed form solutions and highly accurate numerical solutions. Eventually comparison with experimental results is possible.

Progress on Design of shell structures subject to thermal loads
An evaluation of analytical methods has been started, in which benchmark tests for the SPAR elements used in the COMET program have been compared with exact solutions, and with elements for other finite element programs.

Proposed program
Our proposed program includes benchmark tests, literature search, selection of finite element programs, evaluation of design methods and of candidate designs for tanks and shell structures using linear theory. Configurations may include straight and tapered cylinders, various types of stiffeners and frames, double walled skins, composites, various combinations of insulation. Environments may include various pressure and temperature gradients. Certain models may be used to compare different finite elements or to search for optimum materials.
MACHINED-OUT I-BEAMS
SPF HAT STRINGER
DETAIL MODEL OF SPF HAT STRINGER
LOADED SPF HAT STRINGERS

COMPRESSION, $A_{\text{eff}} = 0.368 \text{ in}^2$

BENDING, $I_{\text{eff}} = 0.129 \text{ in}^4$

TORSION, $J_{\text{eff}} = 0.249 \text{ in}^4$
QUARTER SPF STRINGER/FRAME PANEL
UPPER QUARTER OF TANK
top ring

bottom 1/2 stringer

overall buckling mode

top ring

bottom 1/2 stringer

panel buckling mode
Comparison of Results of Analysis of 1/16 Tank Section, Panels, and Theory

Buckling Strength (lbs) (Millions)

Model type

1/16 tank section

20 X 30 mesh panel

4 X 6 mesh panel

Theoretical panel

- panels
- complete model
- no rotat. stiff.
- & no internal pres.
4 Characteristics

- Element Geometry
  Taper, Skew, Aspect Ratio, Warp

- Problem Geometry
  Single and Double Curvature,
  Slenderness Ratio

- Material Properties

- Loading and Constraints
  Deform Elements in all possible directions
Flat Disk Test
(Plane Stress or Plane Strain)

Inner Radius: 3
Outer Radius: 9
Thickness: 1

Temp(r) = -ln(r/3)/ln3 + 100
E = 11x10^6
v = .325
alpha = 14x10^-6

Boundary Conditions:
Free at inner and outer radius
Symmetry elsewhere
### Ansys Element Test Results

<table>
<thead>
<tr>
<th>Element</th>
<th>Percent Error</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td><strong>Plane Stress</strong></td>
</tr>
<tr>
<td></td>
<td>(.0012864)</td>
</tr>
<tr>
<td>Stif63</td>
<td>5.1</td>
</tr>
<tr>
<td>Stif93</td>
<td>0.02</td>
</tr>
<tr>
<td>Stif42</td>
<td>13.3</td>
</tr>
<tr>
<td>Stif82</td>
<td>7.8</td>
</tr>
<tr>
<td>Stif45</td>
<td></td>
</tr>
</tbody>
</table>

The percent error calculated by comparing the displacement at the inner radius of the disk.
Objectives

The basic objective of this research program is to investigate experimentally the viscoplastic response of thermal structures for high speed flight. An additional objective of the experimental program is to provide high quality data for validation of finite element analysis using unified viscoplastic constitutive models.
EXPERIMENTAL AND COMPUTATIONAL STUDIES OF THERMOVISCOPLASTIC PANELS

Earl A. Thornton
Marshall Coyle

Mechanical and Aerospace Engineering
EXPERIMENTAL AND COMPUTATIONAL STUDIES
OF THERMOVISCOPLASTIC PANELS

Earl A. Thornton, Professor
Marshall F. Coyle, Graduate Student

Department of Mechanical and Aerospace Engineering

Abstract

The presentation describes computational and experimental studies of the thermal-structural behavior of thin panels subjected to localized heating. Three research tasks are described: (1) development of a finite element thermoviscoelastic computational approach; (2) experimental determination of material parameters for Bodner-Partom constitutive models of panel materials; and (3) experimental study of "Heldenfels" panels subjected to intense local heating. Recent research progress in each task is reviewed. Development of a new experimental set-up for the panel tests is described in detail and preliminary test results are presented. Plans for future research are highlighted.
RESEARCH OBJECTIVES

- Investigate Thermoviscoplastic (TVP) response of thin panels subject to intense local heating.

- Evaluate finite element Thermal-Structural analyses with unified TVP constitutive models by comparison with experimental data.
THERMOVISCOPLASTIC RESEARCH PROGRAM

• FINITE ELEMENT TVP ANALYSIS
  J. D. KOLENSKI

• BODNER-PARTOM CONSTITUTIVE MODELS
  MARK A. ROWLEY

• THERMAL-STRUCTURAL TESTS OF PANELS
  MARSHALL F. COYLE
FINITE ELEMENT THERMOVISCOPLASTIC ANALYSIS
FINITE ELEMENT THERMOVISCOPLASTIC ANALYSIS

- ASSUMES QUASI-STATIC THERMAL STRESS BEHAVIOR
  - Neglects Thermal-Mechanical Coupling in Energy Equation
  - Neglects Inertia Forces in Equations of Motion

- ASSUMES PLANE STRESS

- USES BODNER-PARTOM CONSTITUTIVE MODEL

- IMPLEMENTS EQUATIONS IN RATE FORM AND USES TIME-MARCHING ALGORITHM
HASTELLOY-X PANEL

Finite Element Meshes:

Slowly Heated  Rapidly Heated

Nodes:  176 nodes  187 nodes
uniform  stretched

Elements:  150 quads  320 triangles
HASTELLOY-X PANEL TEMPERATURES

SLOWLY HEATED

RAPIDLY HEATED

![Graph showing temperature distribution for slowly heated and rapidly heated panels.](image-url)
YIELDED REGIONS

RAPIDLY HEATED

SLOWLY HEATED
RECTANGULAR PANEL INELASTIC STRESSES

Slowly Heated

Rapidly Heated

STRESS (ksi)

Y (inches)

0 1 2 3 4 5

0 10 20 30

90 min 60 min 35 min

180s 60s 10s 2s

STRESS (ksi)

Y (inches)

0 1 2 3 4 5

0 10 20 30

-30
RECTANGULAR PANEL INELASTIC STRESSES

Slowly Heated

Rapidly Heated

Stress (ksi)

Y (inches)

25

20

15

10

5

0

-5

90 min

60 min

35 min

2s

10s

60s

180s

Y (inches)
CONCLUSIONS FROM PLANE STRESS COMPUTATIONS

- TEMPERATURE RISE TIMES AND LEVELS SIGNIFICANT

- FOR RAPID TEMPERATURE RISES:
  - HIGHER YIELD STRESSES FROM STRAIN-RATE EFFECTS

- AT ELEVATED TEMPERATURES:
  - MATERIAL YIELD STRENGTH AND STIFFNESS DEGRADE RAPIDLY
  - PRONOUNCED PLASTIC DEFORMATION
EXTENSION OF COMPUTATIONS TO PLATE BENDING

- FINITE ELEMENT PLATE BENDING
  WITH VON KARMAN PLATE THEORY

- REPRESENT INITIAL PANEL DEFORMATIONS AND THERMAL BUCKLING

- BODNER-PARTOM CONSTITUTIVE MODEL

- QUASI-STATIC RESPONSE

- THERMAL AND MECHANICAL LOADS

- TVP RATE FORMULATION
FINITE ELEMENT FORMULATION

\[
[K_m + K_b + K_g(N)]\ddot{\delta} + [K_g(N)]\dot{\delta} = \{F_p\} + \{F_T\} + \{F_\sigma\}
\]

where:

\[ [K_m] = \text{Membrane Stiffness Matrix} \]

\[ [K_b] = \text{Bending Stiffness Matrix} \]

\[ [K_g(N)] = \text{Geometric Stiffness Matrix} \]

\[ N = \text{Membrane Forces} \]

\[ \{F_p\} = \text{Plastic Strain} \]

\[ \{F_T\} = \text{Temperature} \]

\[ \{F_\sigma\} = \text{Surface Tensions} \]
FINITE ELEMENT APPLICATIONS

VALIDATION STUDIES:

1. CLASSICAL ELASTIC PLATES (COMPLETED)
2. ELASTIC VON KARMAN PLATES
3. ELASTIC AND INELASTIC STUDIES (PLANNED):
   - LEVY AND CLOUGH FOR PRESSURE LOADS (CURRENT)
   - GOSSARD, ET AL. FOR HELDENFELS PLATE (PLANNED)
4. SLOWLY HEATED HASTELLOY PANELS
5. RAPIDLY HEATED HASTELLOY PANELS
6. ALUMINUM ALLOY PANELS
BODNER-PARTOM CONSTITUTIVE MODELS
APPROACH FOR CONSTITUTIVE MODEL DEVELOPMENT

1. Review Bodner-Partom Model

2. Study SwRI Experimental Procedure

3. Simulate Material Constant Determination Procedure for B1900+Hf

4. Begin Experimental Program for
   - Hastelloy-X
   - 8009 Aluminum Alloy

5. Validate Bodner-Partom Model
ESSENTIAL EQUATIONS FOR THE UNI-DIRECTIONAL BODNER-PARTOM CONSTITUTIVE MODEL

1. $$\dot{\varepsilon}_t = \dot{\varepsilon}_e + \dot{\varepsilon}_p$$

2. $$\dot{\varepsilon}_p = (2/\sqrt{3})D_0\{\sigma/|\sigma|\}\exp\{-0.5(Z/\sigma)^2\}$$

3. $$Z = Z^I + Z^D$$

4. $$\dot{Z}^I = m_1(Z_1-Z^I)\dot{W}_p - A_1Z_1\{Z^I/Z_1\}r_1$$

5. $$\dot{Z}^D = m_2(Z_3-Z^D)\dot{W}_p - A_2Z_1\{Z^D/Z_1\}r_2$$

6. $$\dot{W}_p = \sigma(\dot{\varepsilon}_p)$$
### MATERIAL CONSTANTS IN THE
**BODNER-PARTOM CONSTITUTIVE MODEL**

<table>
<thead>
<tr>
<th>TEMP. INDEPENDENT</th>
<th>TEMP. DEPENDENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>( D_0 ) : Limiting shear strain rate ([\text{sec}^{-1}])</td>
<td>( Z_0 ) : Initial value of isotropic hardening variable ([\text{psi}])</td>
</tr>
<tr>
<td>( Z_1 ) : Limiting (maximum) value of ( Z^I ) ([\text{psi}])</td>
<td>( Z_2 ) : Fully recovered (minimum) value of ( Z^I ) ([\text{psi}])</td>
</tr>
<tr>
<td>( Z_3 ) : Limiting (maximum) value of ( Z^D ) ([\text{psi}])</td>
<td>( n ) : Kinetic parameter</td>
</tr>
<tr>
<td>( m_1 ) : Hardening rate coeff. of ( Z^I ) ([\text{psi}^{-1}])</td>
<td>( A_1 ) : Recovery coeff. for ( Z^I ) ([\text{psi}])</td>
</tr>
<tr>
<td>( m_2 ) : Hardening rate coeff. of ( Z^I ) ([\text{psi}^{-1}])</td>
<td>( A_2 ) : Recovery coeff. for ( Z^D ) ([\text{psi}])</td>
</tr>
<tr>
<td></td>
<td>( r_1 ) : Recovery exponent for ( Z^I )</td>
</tr>
<tr>
<td></td>
<td>( r_2 ) : Recovery exponent for ( Z^D )</td>
</tr>
</tbody>
</table>
PROCEDURE FOR OBTAINING
BODNER-PARTOM CONSTANTS (SwRI)

1. Conduct a series of Multi-Strain-Rate Uniaxial Tensile Tests

2. Obtain a Polynomial that approximates $\sigma$ vs. $\varepsilon_p$

3. Using the polynomial data, generate a plot of $\gamma$ vs. $\sigma$

   where $\gamma = (1/\sigma) (d\sigma/d\varepsilon_p)$

4. Obtain $m_1$ and $m_2$ from the slopes of $\gamma$ vs. $\sigma$

5. Set $D_0$ (usually taken to be $1 \times 10^4$ sec$^{-1}$)

6. Obtain $n$ from saturation stress ($\sigma_s$) vs. strain rate

7. Calculate sum of $Z_1$ and $Z_3$ from $\sigma_s$, $n$, and $\varepsilon_p$

8. Obtain $Z_0$ from 0.2% offset yield stress; Set $Z_2 = Z_0$

9. Calculate $Z_1$ from $\sigma_{\text{yield}}$ and $\sigma_s$; Obtain $Z_3$

10. Calculate $A_1 = A_2$ and $r_1 = r_2$ from slow rate ($\varepsilon_2$) tensile data
SIMULATED TENSION TEST WITH STRAIN RATE JUMPS

\[
\begin{align*}
\dot{\varepsilon}_1 &= 1E-04 \quad \text{1/sec} \\
\dot{\varepsilon}_2 &= 1E-06 \quad \text{1/sec} \\
\dot{\varepsilon}_3 &= 1E-05 \quad \text{1/sec} \\
\dot{\varepsilon}_4 &= 1E-04 \quad \text{1/sec} \\
\dot{\varepsilon}_5 &= 1E-03 \quad \text{1/sec}
\end{align*}
\]
THERMAL-STRUCTURAL TESTS OF PANELS
HELDENFELS PROBLEM

Coolant

Plastic Tube

Test Panel

Coolant

\( \dot{q} \)

a = 15 in

b = 10 in

Tentlike Temperature Distribution
EXPERIMENTAL PROGRESS

PHASE 1 - INSTRUMENTATION OF HASTELLOYS PANELS

- Measured Hastelloy-X Panels' Initial Deformations
- Installed PC Based Data Acquisition System
- Installed Strain Gages and Thermocouples
- Installed LVDTs To Measure Out of Plane Displacement

PHASE 2 - DESIGN AND FABRICATE TEST FIXTURE

- Incorporated Line Heater
- Installed and Tested Coolant Tubes and Chill Water System
- Provided Four Point Supports For Test Panel
- Provided Mounts for LVDTs

PHASE 3 - BEGAN TESTING HASTELLOYS-X PANELS

- Using Line Heater
- Evaluated Temperature Distribution
- Evaluated LVDTs Data

Color Slides Will Show The Experiments.
HASTELLOY-X PANEL

MEASURED INITIAL DISPLACEMENTS
TEST FIXTURE FOR THERMAL-STRUCTURAL TESTS OF PANELS
INITIAL TEST RESULTS

FOR SLOWLY HEATED HASTELLOY-X PANEL

- TEMPERATURE DISTRIBUTIONS
- TEMPERATURE HISTORIES
- DISPLACEMENT HISTORIES
EXPERIMENTAL TEMPERATURES FOR TEST PANEL

TEMPERATURE PROFILE ALONG Y AXIS
EXPERIMENTAL TEMPERATURES FOR TEST PANEL

TEMPERATURE PROFILE ALONG Y AXIS (WITHOUT LVDT)
EXPERIMENTAL TEMPERATURES FOR TEST PANEL

TEMPERATURES TAKEN ALONG X AXIS

TIME = 30 min
TIME = 90 min
TIME = 15 min
TIME = 0 min
EXPERIMENTAL TEMPERATURES FOR TEST PANEL

TEMPERATURES TAKEN NEAR THE COOLED EDGE \((Y = -4.3)\)
EXPERIMENTAL TEMPERATURES FOR TEST PANEL

TEMPERATURES TAKEN ALONG Y AXIS VARYING WITH TIME

![Graph showing temperature variations along the Y axis over time.](image-url)
EXPERIMENTAL TEMPERATURES FOR TEST PANEL

TEMPERATURES TAKEN AT X = ±7.5, Y = 0

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EXPERIMENTAL TEMPERATURES FOR TEST PANEL

THROUGH THE THICKNESS TEMPERATURE VARIATION (X = 0, Y = 1)
EXPERIMENTAL TEMPERATURES FOR TEST PANEL

THROUGH THE THICKNESS TEMPERATURE VARIATION (X = 0, Y = -1)
EXPERIMENTAL DISPLACEMENTS FOR TEST PANEL

DISPLACEMENT OF THE PANEL ALONG X AXIS

[Diagram showing displacement over time for different X values]
HEAT LAMP INCIDENT FLUX VARIATIONS

● PRELIMINARY TESTS INDICATE SIGNIFICANT FLUX VARIATION IN X-Y FOCAL PLANE

● SUMI (REF. 6) FOUND MAJOR VARIATION OF FLUX WITH DISTANCE Z FROM LAMP

● DEPENDENCE OF FLUX ON Z COULD COUPLE THERMAL AND STRUCTURAL RESPONSE

● AUTOMATED "X-Y-Z" FLUX MEASUREMENT FIXTURE UNDER DEVELOPMENT
FUTURE RESEARCH PLANS

- LAMP CHARACTERIZATION TESTS
- ACQUIRE STRAIN GAGE DATA ACQUISITION SYSTEM
- CONTINUED TESTS OF HASTELLOY-X PANELS
- TEST PANEL WITH STRAIN GAGES
- BEGIN CORRELATION WITH ANALYSIS
CONCLUDING REMARKS

Recent progress of a research program focused on understanding the thermoviscoplastic behavior of structural panels is described. The program has three tasks: (1) finite element simulations of nonlinear material and geometric behavior, (2) experimental determination of parameters for the Bodner-Partom constitutive models of panel materials, and (3) thermal-structural tests of panels subjected to localized heating.

Plane stress finite element computations are providing insight into panel behavior under different experimental conditions and have shown the importance of thermal loading rates. Finite element analysis of nonlinear panel bending is under development. This capability will permit the simulation of the panel tests and direct correlation of predicted displacements and strains with measured values.

A research task focused on the experimental determination of the constitutive model parameters was recently initiated. This task will provide data for the panel materials for the range of temperatures and strain rates to be used in the thermal-structural test program. Initial tests will be conducted for the available Hastelloy X material; later tests will characterize the 8009 aluminum alloy as material becomes available.

Thermal-structural testing has progressed with the design and fabrication of a panel test fixture. The fixture supports the quartz lamp line heater, the coolant system and panel insulation. It also provides point supports for a panel and supports for LVDTs to measure panel displacements. Preliminary tests have measured Hastelloy-X panel temperature and displacement histories. Unexpected variations of panel temperatures appear to be related to nonuniform incident panel heat fluxes. An experimental program to investigate lamp heat flux variations was recently initiated.

Future plans include continued development of each of the research tasks. Within the next year correlations of simulated thermoviscoplastic panel behavior with experimental data will be initiated.
REFERENCES


APPENDIX I: GRANT PUBLICATIONS (January 1 to June 30, 1991)


APPENDIX III: ABSTRACTS OF GRANT PUBLICATIONS
ELEVATED TEMPERATURE CRACKING OF RSP ALUMINUM ALLOY 8009: 
THE ENVIRONMENTAL INFLUENCE

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Introduction

Dispersion strengthened, ultrafine grain size aluminum alloys, produced by rapid solidification or mechanical alloying and powder processing, are candidate materials for next generation aerospace structures, particularly for applications at temperatures between 150 and 350 °C. The planar flow casting, thermomechanical processing, elevated temperature strength and microstructural stability of a particular material, alloy 8009, are well established (1).

Alloy 8009, like others of this class, is prone to "intermediate temperature embrittlement", specifically tensile ductility and fracture toughness reductions (2-5), subcritical "creep" crack growth (6), and time dependent enhanced fatigue crack propagation rates (7,8) at temperatures between 150 and 225 °C. Speculative mechanisms for this behavior have been proposed, including: (a) dynamic strain aging due to nonequilibrium solute such as iron (2-4,9), (b) reduced extrinsic delamination toughening associated with prior ribbon boundaries (2,10), (c) environmental hydrogen or oxygen embrittlement due to water vapor or O\textsubscript{2} reactions with aluminum (2,7,11), (d) embrittlement by dissolved hydrogen from decomposition of hydrated oxides entrained on ribbon particle surfaces (12), and localized deformation unique to submicron grain size alloys with a high volume fraction of fine dispersoids (2).

The objective of this study is to define the role of the moist air environment in degrading the elevated temperature fatigue and fracture resistance of alloy 8009. Fracture mechanics characterizations of initiation (K\textsubscript{IC}) and propagation (K-\Delta a R-curve) fracture toughnesses, sustained load crack growth rates (da/dt-K) and fatigue crack propagation kinetics (da/dN-\Delta K) were conducted at 175 °C in moist air and high vacuum, and with both as-processed and vacuum heat treated specimens. The results show that each measure of intermediate temperature cracking is equivalent for loading in moist air and vacuum, and is unaffected by vacuum heat treatment prior to cracking.

Experimental Procedure

Ribbon cast, comminuted and compacted alloy 8009 (Al-8.5Fe-1.3V-1.7Si by wt%) was provided by Allied-Signal Inc. in the forms of 10 mm thick extrusion and 6.4 mm thick cross-rolled plate. The rapidly solidified powder contained 25 volume percent of Al\textsubscript{14}(Fe,V)\textsubscript{3}Si dispersoids, sized less than 100 nm in diameter, and an ultrafine grain size of less than 500 nm (1,2). Extruded material contained oxide decorated prior particle boundaries (1000 x 200 x 20 μm) that were largely randomized by cross rolling.
VISCOPLASTIC RESPONSE OF STRUCTURES FOR INTENSE LOCAL HEATING

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Abstract

A thermoviscoplastic finite element method employing the Bodner-Partom constitutive model is used to investigate the response of simplified thermal-structural models to intense local heating. With rapid rises of temperature, the nickel alloy structures display initially higher yield stresses due to strain rate effects. As temperatures approach elevated values, yield stress and stiffness degrade rapidly and pronounced plastic deformation occurs.

Introduction

As hypersonic vehicles accelerate at high speeds in the atmosphere, shocks sweep across the vehicle interacting with local shocks and boundary layers. These interactions expose structural surfaces to severe local pressures and heat fluxes. One example is leading edges of integrated engine structures which experience intense, highly localized aerothermal loads (Fig. 1). Reference 1 studies issues relevant to the thermal-structural response of hydrogen cooled, super thermal-conducting leading edges subject to intense aerodynamic heating. A thermostructural analysis with experimental verification of a cowl lip design demonstrated that inelastic effects occur and may be significant.

Until recent years the study of structural response at elevated temperatures due to dynamic loads was not possible because of an inability to model inelastic material behavior. Over the last twenty years, unified viscoplastic constitutive models have evolved to meet this need. These constitutive models provide a means for representing a material's response from the elastic through the plastic range including strain-rate dependent plastic flow, creep and stress relaxation. Rate-dependent plasticity effects are known to be important at elevated temperatures. The first multidimensional formulations of viscoplastic constitutive relations was due to Bodner and Partom. Since then, a number of constitutive models have been developed; many of these theories, including the model of Bodner and Partom, are summarized in review articles that appear in reference 3.

Unified constitutive models implemented in finite element programs provide an important simulation capability. Finite element analysis with unified constitutive models have been under development for about 15 years. Reference 4 describes these efforts and presents a thermoviscoplastic finite element computational method for hypersonic structures. Applications of the approach to convectively cooled hypersonic structures illustrate the effectiveness of the approach and provide insight into the transient viscoplastic behavior at elevated temperatures. A recent paper5 uses the computational method presented in reference 4 to perform quasi-static finite element thermoviscoplastic analysis of aerospace structures including a convectively-cooled engine cowl leading edge subjected to aerodynamic shock interference heating. A study6 of dynamic effects in thermoviscoplastic vibrations shows that inelastic strains introduces significant damping.

There is a need for further computational thermoviscoplastic studies to: (1) understand the phenomena that occur in the viscoplastic response of structures for intense local heating, (2) investigate the finite element modeling techniques required to represent thermoviscoplastic behavior, (3) assist in planning thermal-structural experiments to validate computational methods, and (4) aid in understanding the high-temperature behavior of difficult design problems such as leading edges of hypersonic vehicles.

The purpose of this paper is to describe recent studies that address the first three needs. The paper begins with a description of quasi-static thermoviscoplastic structural analysis. The initial value viscoplasticity problem statement is presented, the Bodner-Partom constitutive model is described, the finite element formulation is outlined, and the viscoplastic solution method is detailed. Computational studies of thermoviscoplastic behavior of two thermal structural applications are then presented. An appendix presents thermal and structural data for materials used in the numerical computations.

Thermo-Viscoplastic Structural Analysis

The behavior of a thermo-viscoplastic structure subjected to intense heating is analyzed assuming that: (1) thermomechanical coupling in the conservation of energy equation can be neglected, (2) the structural response is quasi-static, and
THE EFFECT OF TRACE ADDITIONS OF Zn ON THE PRECIPITATION BEHAVIOR OF ALLOY 8090 DURING ARTIFICIAL AGING

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Abstract

The effect(s) of trace additions of Zn to the artificial aging behavior of alloy 8090 (Al-Li-Cu-Mg-Zr) was investigated in the approximate composition range 0 - 1 wt-% Zn. Trace Zn additions were found to delay aging and under equivalent aging treatments (100 hrs at 160C) the alloy without Zn and the 1.07 wt-% Zn alloy developed $\delta'$-free zones along subgrain boundaries, while the alloys of 0.21 and 0.58 wt-% Zn did not. DSC analysis indicated that Zn was being incorporated into the $\delta'$, shifting its exotherm to higher temperatures, while having little if any effect on its associated endotherm making it unlikely that it is an artifact of a solvus shift. In the 8090 + 1.07 wt-% Zn alloy, coarse precipitates were found to reside on subgrain boundaries and EDS indicated that they were rich in Cu and Zn. It was also noted that in the Zn containing 8090 variants, the $S'$ precipitates were more coarse in size than the baseline 8090.


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