MATERIALS DIVISION RESEARCH AND TECHNOLOGY
ACCOMPLISHMENTS FOR FY 87
AND PLANS FOR FY 88

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MATERIALS DIVISION
RESEARCH AND TECHNOLOGY ACCOMPLISHMENTS FOR FY 87
AND PLANS FOR FY 88

SUMMARY

The research program of the Materials Division is presented as FY 87 accomplishments and FY 88 plans. The accomplishments for each Branch are highlighted and plans are outlined. Publications of the Division are included by Branch. This material will be useful in program coordination with other government organizations, universities, and industry in areas of mutual interest.

ORGANIZATION

The Langley Research Center is organized by directorates as shown in figure 1. Each directorate is organized into divisions and offices. The Materials Division of the Structures Directorate consists of four branches as shown in figure 2. This figure also shows the technical areas addressed by each Branch. The Division consists of 69 NASA civil servants and 7 members of the Army Aerostructures Directorate, USAARTA, Army Aviation Systems Command, located at the Langley Research Center. In addition, about 38 non-personal support contractors work at the Center to add major support to the in-house research program.

FUNCTIONAL STATEMENT

The Materials Division initiates, organizes, and conducts experimental and analytical research on structural materials and their application to aircraft and spacecraft structural systems (figure 3). More specifically the Division:
- Conducts fundamental and applied research studies to develop novel polymeric, metallic, and ceramic materials for advanced structural applications.
- Establishes materials processing and fabrication technology for structural components.
- Demonstrates the application and benefits of advanced materials to specific flight vehicle structures.
- Defines, evaluates, and conducts research on thermal protection materials requirements for high-speed aircraft and space transportation systems.
- Studies the fatigue and fracture behavior of materials to establish practical methods for insuring the structural integrity of aircraft and space structures.
- Characterizes the behavior of structural materials in extreme service environments using test facilities and laboratories for simulation of the flight environment.
- Originates and develops requirements for new facilities and research techniques.
- Operates the fatigue and fracture, structural materials, polymer, metallurgical, and environmental effects laboratories.
The long range research thrusts of the Materials Division are shown in figure 4.

**FACILITIES**

The Materials Division has four major facilities to support its research program.

The Structures and Materials Laboratory houses various environmental effects labs and the metallurgical and metals processing labs. In the environmental effects labs, research is conducted to characterize and enhance the performance of structural materials operating in extreme service environments. Test techniques, instrumentation, and measurement techniques are developed to simulate environmental conditions required to evaluate high-temperature structural materials. The interaction of the space environment on properties of advanced composites, polymer films, and coatings for space systems is studied. Radiation and monoatomic oxygen damage in polymeric materials is studied and chemical formulations for enhanced long-term durability in space are identified.

Fundamental and applied research on advanced metallic and metal-matrix materials is conducted in the metallurgical and metals processing labs. Innovative processing methods for new alloy synthesis and development and fabrication of metallic structural components for future aircraft and space vehicles, including high-temperature applications, are explored. Metallic components are analyzed and tested to demonstrate improvements in advanced metallic alloys and their fabrication processes.

The Fatigue and Fracture Laboratory is used to conduct research on the structural integrity of metals and composites for aircraft structures. Tests are conducted to measure the effect of loads on materials under simulated flight conditions. Materials and methods of strength and life prediction for airframes are assessed to develop ways to improve the structural reliability of aircraft.

Fundamental and applied research using advanced polymer synthesis, composites and adhesives processing science, and advanced characterization methodology to develop improved materials concepts for efficient aerospace structures are conducted in the Composites Processing Laboratory. Novel polymeric materials are synthesized for applications such as matrices for fiber-reinforced composites, adhesives for bonding lightweight composite and metal structures, and high-performance films for spacecraft. Innovative processing methods for fabricating composite components for aircraft and spacecraft structures are developed.

Radiation testing of spacecraft materials is conducted in the Space Environmental Effects Laboratory. Spacecraft materials tested include polymeric and metal matrix composites, polymeric films, thermal control coatings, adhesives, solar cells, and laser mirrors.
In addition, the Materials Division is currently constructing a Carbon-Carbon Research Laboratory that is expected to be completed in July 1988. The Materials Division has expanded its research capability in carbon-carbon materials and this lab will house the processing equipment needed for fabricating carbon-carbon materials and for applying oxidation-protective coatings.

**FY 87 ACCOMPLISHMENTS**

**Polymeric Materials Branch**

The Polymeric Materials Branch (figure 5) conducts fundamental and applied research studies combining the disciplines of advanced polymer synthesis, composites and adhesives processing science, and advanced characterization methodology to develop improved materials concepts for efficient aerospace structures. These research and development activities are aimed at achieving maximum structural exploitation of advanced composites and adhesives through development of balanced mechanical/physical properties with good processability. The five year plan for this research is shown in figure 6.

The FY 87 accomplishments of the Polymeric Materials Branch are listed below and are highlighted in figures 7 through 11.

**High Performance Polymer Concepts**
- Polymer Technology Transfer
- Preparation of Amide-Imide Polymers

**Composite Matrices**
- Crystallization in Thermoplastic Resins - LARC-TPI

**Composite Processing and Adhesive Bonding**
- LARC-TPI Composites Via New Slurry Process
- The Effect of Diamic Acid Additives on the Processability of Polyimides

**Fatigue and Fracture Branch**

The Fatigue and Fracture Branch (figure 12) performs research on the integrity of materials for load-bearing structures of metals and composites. This research includes fatigue, fracture mechanics, and structural reliability. Equations and analytical methods are formulated to predict fatigue life and residual strength of damaged and undamaged materials. Design, construction, operation, and inspection methods applied to airframes are assessed to develop ways to improve the overall structural reliability of aircraft and spacecraft. The five year plan of the Branch is shown in figure 13.
The FY 87 accomplishments of the Fatigue and Fracture Branch are listed below and are highlighted in figures 14 through 25.

**Metals and Metal Matrix Composites**
- Fatigue Life of Material With a Machining Scratch
- Three-Dimensional Analysis of Fatigue Crack Closure
- Fiber-Matrix Separation in Silicon Carbide/Titanium Matrix Composites

**Composites**
- Delamination Fatigue Behavior of Composite Materials
- Matrix Yielding at a Delamination Front
- Interlaminar Shear Fatigue Thresholds For Composite Materials

**Computational Methodology**
- Calculation of Strain-Energy Release Rate Distribution Using Plate Analysis
- Boundary Force Method For Analyzing Cracked Laminates
- Strain Energy Release Rates for Edge-Delaminated Composite Laminates
- Fiber-Resin Micromechanics Analysis of Delamination
- Finite-Element Analysis of End Notched Flexure (ENF) Specimen
- Finite-Element-Alternating Method for Crack Analyses

**Applied Materials Branch**

The Applied Materials Branch (figure 26) conducts research to characterize and enhance the performance of structural materials operating in extreme service environments. The Branch identifies mechanisms of environmental degradation and failure in structural materials, provides quantitative understanding of degradation mechanisms and evolves models to predict the rate or extent of degradation for various advanced structural materials. Theoretical and experimental studies which relate to the environmental performance of high-temperature materials for thermal protection systems and hot structures of advanced space transportation systems and hypersonic vehicles are conducted. The interaction of the space environment on properties of advanced composites, polymer films, and coatings of interest for space systems is studied. The five year research plan for the Branch is shown in figure 27.

The FY 87 accomplishments of the Applied Materials Branch are listed below and are highlighted in figures 28 through 34.

**Space Materials**
- Development of Protective Coatings for Composite Tubes
- A Comparison of the Effects of Simulated Low-Earth and Geosynchronous Orbit Exposure on Composite Materials
- Thermally Induced Twist in Composite Tubes
Composite Materials for Rotorcraft and Aircraft Structures
- Thermally Stable Graphite-Reinforced Aluminum Alloys
- A Method of Predicting the Energy-Absorption Capability of Composite Subfloor Beams
- Using Fundamental Spectroscopic Data to Explain Changes in Applied Properties of Irradiated Polymers
- Innovative Fabrication of Composite Structures

Metallic Materials Branch

The Metallic Materials Branch (figure 35) conducts fundamental and applied research studies on advanced metallic and metal-matrix materials. The Branch performs research on advanced high-strength structural alloys and composites to achieve improved mechanical properties through understanding and control of microstructural features. A basic understanding of joining and forming processes for fabricating structural components from advanced metallic materials is developed and innovative processing methods for new alloy synthesis and development and fabrication of metallic structural components for future aircraft and space vehicles is explored. The five year research plan for the Branch is shown in figure 36.

The FY 87 accomplishments for the Metallic Materials Branch are listed below and are highlighted in figures 37 through 42.

Advanced Light Alloy and MMC Development
- B₄C Particulates Show Promise to Improve Properties in Aluminum Matrix Composites
- Material Property Verification of LaRC Processed PM Aluminum Alloys
- Improved Aging Characteristics by Minor Alloying Additions in Al-Li Alloys

Innovative Metals Processing
- Alleviation of Cavitation in Superplastically Formed 7475 Aluminum Alloy Using Post-Forming Pressure

High Temperature Thin Gage Metals and MMC for Airframes
- Emittance/Catalysis Coatings Improve Performance of Titanium-Aluminides
- Liquid Interface Diffusion Bonding of Titanium Aluminides Shows Promise for Elevated Temperature Applications to 1700°F
PUBLICATIONS AND PRESENTATIONS

The FY 87 accomplishments of the Materials Division are highlighted by a number of publications and presentations. These are listed by organization and are identified by the categories of formal NASA reports, quick-release technical memorandums, contractor reports, journal articles and other publications, meeting presentations, technical talks, tech briefs, and patents.

DIVISION OFFICE

Contractor Reports


Journal Articles and Other Publications


Meeting Presentations


Technical Talks


Tech Briefs


POLYMERIC MATERIALS BRANCH

Quick-Release Technical Memorandums


Contractor Reports


Journal Articles and Other Publications


**Meeting Presentations**


Technical Talks


64. Hergenrother, P. M.: Other High Performance Heterocyclic Polymers. Presented at the American Chemical Society Division of Polymer Chemistry Interdisciplinary Symposium on Recent Advances in Polyimides and Other High Performance Polymers, July 13-16, 1987, Reno, Nevada.


Tech Briefs


98. Hergenrother, P. M. (LaRC); and Harris, F. W.; and Beltz, M. W. (University of Akron): A New, Readily Processable Polyimide. NASA Tech Brief LAR-13675.


Patents


FATIGUE AND FRACTURE BRANCH

Quick-Release Technical Memorandums


**Contractor Reports**


Journal Articles and Other Publications


Meeting Presentations


Technical Talks


156. Poe, C. C., Jr.: Fracture of Boron/Aluminum Laminates With Various Proportions of $0^\circ$ and $45^\circ$ Plies. Presented at the 1986 ASME Winter Annual Meeting, December 7-12, 1986, Anaheim, California.


Tech Briefs


APPLIED MATERIALS BRANCH

Formal Reports


Quick-Release Technical Memorandums


Contractor Reports


Journal Articles and Other Publications


**Meeting Presentations**


Diego, California. Proceedings pending.


Technical Talks


Tech Briefs


Patents


Metallic Materials Branch

Formal Reports


Contractor Reports


Journal Articles and Other Publications


Meeting Presentations


Technical Talks


Tech Briefs


Patents


FY 88 PLANS

Polymeric Materials Branch

Major research thrusts for FY 88 in the Polymeric Materials Branch are in the areas of resin-matrix composite studies and high performance polymers. Plans for this research are outlined in figure 43. Plans include the synthesis and characterization of neat resins and their composites and the study of prepregging and processing of new matrices into composite materials. Also, the preparation of a series of new semi-crystalline imide copolymers having properties equivalent to or superior to the best available polyimide is planned. This research will be conducted under the following two RTR’s:

RTR 505-63-01-01 Resin-Matrix Composite Studies

Objective:
Develop and evaluate new damage tolerant, durable high performance composites for advanced structural applications.

Approach:
Synthesize, characterize and evaluate new/improved moderate and high temperature thermoplastics, semi-crystalline polymers and hybrid thermoplastic/thermosets and their carbon fiber composites. Study prepregging and processing of new matrices into composite materials, including development of new processes and processing equipment. Conduct initial evaluation of new composites, including thermal, thermomechanical, and fracture toughness studies. Support micromechanical analysis program by providing appropriate composites and characterization studies.

Milestones:
- Complete synthesis and characterization of thermally stable core shell rubbers and determine their efficacy as toughening agents for high temperature polymers - March 1988.
- Prepare large quantity (up to 20 lbs.) of the most promising experimental

- Determine thermal, thermomechanical and fracture toughness properties of the most promising experimental advanced composite matrix material - July 1988.

- Complete Phase II modification of Murdock prepregging machine; demonstrate machine capability with research thermoplastic - August 1988.


**RTR 506-43-11-01 High Performance Polymers**

**Objective:**
Develop high performance polymers as adhesives, composite matrices, and films for use in high temperature environments.

**Approach:**
Synthesize new polymers with an attractive combination of properties and evaluate as high temperature adhesives, composite matrices and films.

**Milestones:**
- Evaluate composite properties of a more processable polyimide sulfone containing a low molecular weight non-reactive imide - January 1988.

- Synthesize new polyimides from isomeric oxydiphthalic dianhydrides - February 1988.

- Improve the processability of a semi-crystalline polyimide (LARC-CPI) and evaluate as an adhesive and composite matrix - April 1988.

- Obtain composite properties on blends of acetylene imidothioethers and acetylene terminated polysulfones - May 1988.

- Optimize molecular weight and processing characteristics of LARC-TPI for sandwich structure - June 1988.


- Complete crystallographic study on crystalline polyimides - August 1988.

Fatigue and Fracture Branch

Composites research in the Fatigue and Fracture Branch for FY 88 will focus on micromechanics and multidirectional materials. The emphasis in metals research will be on advanced Al alloys and metal-matrix composites. Plans for this research are outlined in figure 44. The research will be carried out under the following three RTR's:

**RTR 505-63-01-05 Fatigue and Fracture - Composites and Metals**

Objective:
Develop micromechanical models to predict composite delamination toughness for guidance in the development of second-generation composites. Develop analysis methods to predict cyclic load endurance of aerostructural materials.

Approach:
Develop testing methods and elasto-plastic 3-D analyses to identify determination failure modes in matrix materials. For metallic structures similar analyses are developed to describe growth for small cracks.

Milestones:
- Fully integrate the 2-D Boundary Force Method for computing stress intensity factors into the fracture control analysis computer program (FLAGRO) - June 1988.
- Complete mode I cyclic delamination tests with PEEK laminate - July 1988.
- Complete AGARD small-cracks supplemental program on steel, Al-Li, and Ti alloys - September 1988.

**RTR 506-43-11-04 Damage Tolerance of Composites**

Objective:
To develop unified rational methodologies for the analysis and prevention of cracking damage in composites.

Approach:
Development of data and new analytical models based on fracture mechanics will make both tension and compression failures more tractable in composites.
Milestones:
- Complete combined analytical and experimental study of instability delamination - March 1988.

**RTR 506-43-71-03  Fatigue and Fracture - High Temperature Materials**

Objective: Develop analytical models to predict fatigue and fracture of high-temperature aerostructural materials.

Approach: Develop testing methods, elastoplastic micromechanics stress analyses, and failure criteria for high-temperature composites. Incorporate the failure criteria into stress analyses to develop fracture micromechanics models for fatigue and fracture.

Milestones:
- Order apparatus to upgrade test machines for high-temperature testing - November 1987.

**Applied Materials Branch**

Research emphasis in the Applied Materials Branch for FY 88 will be in the areas of carbon-carbon composites, low expansion resins for precision reflectors, and multidirectional, multilayered textile forms. Plans for this research are outlined in figure 45. This research will be carried out under the following five RTR's:

**RTR 505-63-01-06  Composites for Rotorcraft/Aircraft Structures**

Objective: Develop the technology for the application of advanced composite materials and innovative design concepts in rotorcraft and aircraft structures in order to improve performance, efficiency, damage tolerance, environmental durability, and energy absorption capability compared to metal structures.
Approach:
In-house, contractual, and grant studies will be conducted to develop innovative material forms, and processing science concepts for lightweight composite structure applications. Residual properties after environmental exposures and degradation mechanisms will be determined. Included will be studies on fabrication of near net-shape structural forms using automated textile processes such as weaving, braiding, knitting, and stitching. Composite structural elements fabricated by resin transfer molding and pultrusion processes will be evaluated. Improvements in through-the-thickness properties and damage tolerance will be studied.

Milestones:
- Complete specimen fabrication for investigation of through-the-thickness stitching and resin transfer molding - December 1987.
- Demonstrate the capability of in-process ultrasonics to measure voids in composites fabricated in autoclave and pultruder - March 1988.
- Award 4 year task assignment contract to fabricate and inspect composite materials for in-house evaluation - March 1988.
- Complete 1 year of flight service of DC-10 graphite/epoxy vertical stabilizer - March 1988.
- Complete 15 years of flight service of B-737 graphite/epoxy spoilers - June 1988.
• Demonstrate the capability to weave composite preforms with multilayer bias plies - June 1988.

• Evaluate the effects of included angle on the energy absorption capability of composite tubes and beams - June 1988.

• Complete evaluation of cruciform in-plane shear test specimen - June 1988.

• Establish baseline property data for pultruded composites and refine the fabrication process - September 1988.

• Develop process model for resin infiltration of woven fabrics - September 1988.

• Demonstrate dielectric process monitor for optimization of resin infiltration into woven fabrics - September 1988.

RTR 506-43-21-04 Composite Materials for Spacecraft Applications

Objective:
Develop new composite materials and protective/thermal control coatings for enhanced environmental and thermal-mechanical durability in long-life space structures.

Approach:
Advanced polymeric-, metallic-, and ceramic-matrix, fiber-reinforced composites will be developed and evaluated for long-term use in spacecraft structures. Evaluation will include thermal cycling, UV and atomic oxygen to simulate the GEO environment. Advanced laser interferometry will be used to determine dimensional stability. Thin metallic and oxide protective coatings will be evaluated on flat and tubular surfaces. The optical, chemical, and mechanical property degradation will be characterized and analytically modeled. Shuttle experiments will be used to verify models and laboratory simulations.

Milestones:
• Develop aluminum foil anodizing process for producing tailorable optical coatings for composite tubes - March 1988.

• Determine effects of long-term thermal cycling on the adherence and optical properties of anodized aluminum coatings on graphite/epoxy composite tubes - June 1988.

• Determine threshold doses of electron radiation for adverse effects on thermoplastic polymer films - June 1988.
Objective:
Develop high-strength, minimum gauge, oxidation-protected carbon-carbon materials for hot structure and TPS applications in advanced space transportation vehicles and hypersonic aircraft.

Approach:
Advanced processing methods, alternate precursor materials, fiber surface modifications, and alternate reinforcement concepts will be developed to improve substrate mechanical properties. Matrix and fiber oxidation inhibitors, sealants, and advanced coatings will be developed to improve oxidation resistance. Environmental testing will be performed in simulated mission dynamic environments and in multiparameter (temperature, pressure, load) facilities.

Milestones:
- Key processing parameters for phenolic-based carbon-carbon composites defined for simplified processing and improved interlaminar strengths - September 1988.

RTR 585-02-21-01 Advanced Materials for Precision Segmented Reflectors

Objective:
Develop advanced composite materials and coatings that are durable and have stable thermal and mechanical properties in the space service environment of precision segmented reflector spacecraft.

Approach:
New, novel low expansion polymer resins will be developed and used to fabricate composites. Advanced, highly stable graphite/glass laminates using low temperature fabrication methods will be developed. Material constitutive equations and analytical models will be developed to correlate/predict environmental effects on thermal and mechanical properties of the advanced composites. These models will aid in directing the materials development activities. The surface distortion of composite laminates/panels will be measured and modeled.

Milestones:

**RTR 763-01-41-17 Coatings for Carbon-Carbon Composites**

**Objective:**
Develop oxidatively protected carbon-carbon material concepts to meet specific airframe and control surface requirements in support of Aero-space Plane.

**Approach:**
Evaluate in simulated NASP environments various promising oxidation-protection systems which were developed for propulsion applications. Build on these results, tailoring a new oxidation-protection system (in-depth oxidation protection, sealants, coatings) to meet specific NASP mission requirements.

**Milestones:**
- Preliminary evaluation tests completed on samples of oxidatively protected c-c in multiparameter environmental simulator - March 1988.
- Initiate dynamic environment (arc jet) testing of candidate test materials - June 1988.

**Metallic Materials Branch**

Research in the Metallic Materials Branch for FY 88 will focus on advanced aluminum alloy technology for cryogenic tank applications, innovative metals processing and joining, and high temperature titanium aluminides and advanced metal-matrix composites for hypersonic vehicles. Plans for this research are outlined in figure 46. This research will be carried out under the following five RTR's:

**RTR 505-63-01-02 Advanced Structural Metallics for Service to 1000°F**

**Objective:**
Develop a fundamental understanding of the metallurgical structure/mechanical property interactions resulting from powder processing, consolidation, and subsequent thermomechanical processing of intermediate and high temperature aluminum alloys prepared by advanced ingot and powder metallurgy techniques. Demonstrate the property and durability advantages of advanced aluminum alloys for aerospace structures. Develop advanced inorganic composite materials for aerospace structural applications.
Approach:
Prepare new aluminum alloy compositions of laboratory quantities by advanced I/M and P/M techniques. Develop and evaluate promising composite materials systems with light alloy metallic matrices and correlate microstructural/mechanical property relationships. Identify metallurgical characteristics controlling specific properties through laboratory analysis and development of optimized processing techniques to obtain tailored properties.

Milestones:
- Complete metallurgical characterization and initial consolidation of Al-Mn-Ca-Si alloy for high temperature applications - October 1987.
- Produce PM aluminum alloy powders for high temperature applications using the LaRC gas atomization facility - April 1988.
- Develop processing to produce sheet material from aluminum alloy powder with Mn, Ca, and Si additions for high temperature applications - June 1988.
- Optimize chemistry and processing for enhanced superplasticity in advanced aluminum alloys - September 1988.

RTR 505-63-01-03 Innovative Metals Processing for Airframe Structures

Objective:
Develop improved aluminum alloys and innovative processing methods for fabricating lightweight aerospace vehicle structures. Develop advanced processing techniques for lightweight Al-Li and high temperature aluminum alloys and evacuated titanium honeycomb-core sandwich concepts.

Approach:
Combined in-house and contractual studies to define the potential of advanced aluminum alloys for airframe structural applications. Modify the composition of Al-Li alloys for enhanced superplasticity, weldability and post fabrication heat treatment. Explore the use of high temperature aluminum alloys and develop improved brazing and joining processes for fabricating evacuated titanium
honeycomb-core sandwich structure. Characterize material properties and design, fabricate and test structural elements.

Milestones:

- Explore the use of high temperature aluminum alloys and joining technologies for airframe and tank applications - July 1988.
- Screen joining processes for fabricating coupon size evacuated titanium honeycomb-core sandwich concepts - September 1988.
- Determine the SPF characteristics of Al-Cu-Li-Zr-In alloy for aerospace structural applications - September 1988.

RTR 506-43-71-01 Metallics for High Temperature Airframe Structures

Objective:
Develop new high temperature metallics, processing and joining techniques, and coatings for environmental protection for use at temperatures from 500°F to 2000°F including in-situ and continuously reinforced advanced metal matrix composites and light alloy intermetallics.

Approach:
Combined in-house and contract research studies to develop and characterize advanced metallic systems produced by deposition techniques, rapid solidification rate technology and conventional high temperature processing. Establish suitable joining processes for very thin gage, lightly loaded structure. Demonstrate technology readiness through design, fabrication, testing, and evaluation of structural sub-components.

Milestones:

- Complete preliminary evaluation of the potential for designing nonequilibrium phases to improve high temperature properties and stability of RSR
Objective:
Develop advanced joining processes for fabricating Ti$_x$Al metal-matrix composite, RSR titanium honeycomb-core sandwich structure and develop an analytical model for predicting composite properties.

Approach:
Conduct in-house studies using available titanium based ingot metallurgy (IM) model materials to develop joining processes suitable for fabricating Ti$_x$Al composite sandwich structure. Screen candidate processes including brazing, liquid interface diffusion bonding, and diffusion bonding based on both metallurgical studies and mechanical property tests. Evaluate alternate LID material compositions to improve elevated temperature properties of IM Ti$_3$Al-Ti$_3$Al joints. Develop an analytical model for predicting fatigue behavior and verify experimentally. Fabricate, test and evaluate small sandwich specimens and structural sub-elements using Ti$_3$Al composites as they become available.

Milestones:
- Determine the elevated temperature properties of LID bonded IM Ti$_3$Al-Ti$_3$Al coupon joints - October 1987.
- Determine the RT face-face tension properties of LID bonded IM Ti$_3$Al face sheet titanium honeycomb-core sandwich specimens - December 1987.
- Develop LID compositions for improved elevated properties of LID bonded joints - June 1988.
- Demonstrate the use of melt overflow process for casting RSR titanium foil - August 1988.
- Develop and evaluate an analytical model for fatigue of MMC - September 1988.
Objective:
Develop specific, high temperature metal matrix composites and associated fabrication technology for aero-space plane applications.

Approach:
Establish surface treatments and/or coating systems for selected ceramic and organic fibers for optimum fiber/matrix stability. Fabricate and test minimum gage composite panels to establish performance limits. Develop techniques for structural component fabrication. Define scale-up requirements for large panel manufacture.

Milestones:
- Unidirectional composites fabricated and evaluated - December 1987.

Concluding Remarks
This document presents the FY 87 accomplishments, presentations and publications and the FY 88 research plans of the Materials Division.
LANGLEY RESEARCH CENTER

Director
R. Petersen

Deputy
P. Holloway

Associate
S. Pauls

Chief Scientist
R. Barnwell
- Research quality/content
- University programs

Systems Engineering and Operations
R. Swain
- Engineering support
- Fabrication
- Facility operations

Management Operations
J. Stokes
- Administrative support

Electronics
W. Mace
- Instrumentation support
- Computer/simulator support
- Project management

Aeronautics
R. Harris
- Aerodynamics
- Propulsion integration
- Operating problems

Space
R. Nunamaker
- Aerothermodynamics
- Energetics
- Atmospheric Sciences
- System studies
- Shuttle and Space Station support

Structures
C. Blankenship
- Structures & materials
- Aerelasticity
- Acoustics

Flight Systems
J. Creedon
- Information systems
- Guidance & control
- Flight management

Electronics
W. Mace
- Space electronics
- Computer science

Figure 1.
MATERIALS DIVISION

LONG RANGE THRUSTS - AERONAUTICS

Lead Role

- Metallic materials for aircraft structures
- Carbon-carbon composites for hypersonic vehicles (Proposed)

Support Role

- Composite materials for primary aircraft structures
- Lightweight, hot structures for high-speed aircraft

Figure 4(a).
MATERIALS DIVISION

LONG RANGE THRUSTS - SPACE

Lead Role

- Materials durability in the space environment

Support Role

- Aerothermal structures and materials technology for Advanced Launch Systems
- Structures, materials, and dynamics technology for Space Station

Figure 4(b).
Figure 5.
POLYMERIC MATERIALS BRANCH
FIVE YEAR PLAN

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<th>MAJOR THRUST</th>
<th>FY87</th>
<th>FY88</th>
<th>FY89</th>
<th>FY90</th>
<th>FY91</th>
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<td>High performance polymer concepts</td>
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<td>Advanced processing and bonding technology for high performance materials</td>
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<td></td>
<td>Adhesives development and characterization</td>
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Figure 6.
POLYMER TECHNOLOGY TRANSFER

Terry L. St. Clair
Polymeric Materials Branch
Ext. 3041 October 1986
RTOP 506-43-11

Research Objective: To assure the widest use and greatest benefit from NASA-developed polymer transfer.

Approach: Over the past decade much novel polymer technology has been developed at LaRC in the high temperature/advanced resin area in support of U.S. aerospace activities. Developments by U.S. industry are limited in these areas because immediate applications or an established market are lacking. Patents are obtained by NASA on new polymer systems. These patents can then be licensed to companies interested in commercializing the materials. A special effort is being made to keep U.S. industry aware of recent NASA developments in advanced polymers.

Accomplishments: After an induction period as research capabilities were being formed, the polymer technology that has been developed in the Materials Division is beginning to be exploited in the industrial sector. The chart details some technology transfers that have occurred over the past few years. Several of these developments such as the LARC-160 and LARC-TPI are commercially available today. In other cases, such as the polyimidesulfone and the polyphenylquinoxaline, developmental quantities are being made. The other technologies such as the polyimide laminating and metal ions in polyimides are also beginning to come to the market in prototype forms. These will develop with time.

Significance: Langley-developed polymer technology is being put into commercial practice by industry. Because most of these materials are covered by NASA patents, industry is willing to invest the resources required to bring them to commercial production because their position can be protected through licensing agreements. Polymers that were originally developed with particular aerospace applications in mind are finding broader applications because of their commercial availability.

Future Plans: Continued research activities will be carried out on advanced polymer systems and invention disclosures will be filed. Close interactions with industry will continue in order to assure the widest possible use of NASA's technology.

Figure 7(a).
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<th>SYSTEM</th>
<th>DEVELOPED</th>
<th>PATENTED</th>
<th>LICENSEE</th>
<th>CONSIDERATION TO NASA</th>
</tr>
</thead>
<tbody>
<tr>
<td>LARC-160</td>
<td>Mid 1970's</td>
<td>1979 &amp; 1980</td>
<td>U.S. Polymeric, Fiberite, Hexcel, American Cyanamid, King Mar</td>
<td>None</td>
</tr>
<tr>
<td>LARC-TPI</td>
<td>Mid to late 1970's</td>
<td>1977 &amp; 1978</td>
<td>Mitsui Toatsu, Rogers, High Tech Services</td>
<td>$8000 * + percentage</td>
</tr>
<tr>
<td>Polyimide-Sulfone</td>
<td>Early 1980's</td>
<td>1983 &amp; 1984</td>
<td>M&amp;T Chemical, Celanese</td>
<td>$8000 * + percentage</td>
</tr>
<tr>
<td>Polyimide Laminating Technology</td>
<td>Late 1970's</td>
<td>1985</td>
<td>Rogers (Exclusive)</td>
<td>$5000 ** + percentage</td>
</tr>
<tr>
<td>Polyimide/Palladium</td>
<td>Early 1980's</td>
<td>1981</td>
<td>Rogers (Exclusive)</td>
<td>$5000 ** + percentage</td>
</tr>
<tr>
<td>Polyimide/Aluminum Ions</td>
<td>Early 1980's</td>
<td>1981</td>
<td>Mitsui Toatsu, High Tech Services</td>
<td>$2000 ** + percentage</td>
</tr>
<tr>
<td>Elastomer Toughened Polyimides</td>
<td>Late 1970's</td>
<td>1983 &amp; 1985</td>
<td>High Tech Services</td>
<td>$4000 *** + percentage</td>
</tr>
<tr>
<td>Polyphenyl-Quinoxalines</td>
<td>1960's</td>
<td>None</td>
<td>Hunt Chemical</td>
<td>None</td>
</tr>
</tbody>
</table>

* Four NASA Patents  
** One NASA Patent  
*** Two NASA Patents

Figure 7(b).
PREPARATION OF AMIDE-IMIDE POLYMERS

James F. Dezern
Polymeric Materials Branch
Ext. 3041 December 1986
RTOP 506-43-11

Research Objective: To prepare and characterize thermally-stable, tough polyimides incorporating amide linkage for potential advanced applications as coatings, adhesives and composite matrix resins.

Approach: Prepare a series of novel amide-imide polymers and evaluate physical and mechanical properties.

Accomplishments: Four polyimides were prepared from novel amide diamines. The structure, shown in the accompanying figure, differs from that of LARC-TPI by the incorporation of a more flexible amide linkage. These polyamide-imides exhibited high inherent viscosities and glass transition temperatures. They were made into tough, flexible films which showed good thermal stability and good resistance to organic solvents. Mechanical properties of the films (see figure) were better than those of LARC-TPI, especially the 4,4'-isomer. Its exceptionally high modulus (1068 ksi) value makes this material extremely attractive for composite applications. Films of the 4,4'-isomer also exhibited tough behavior during impact evaluation.

Significance: These polyamide-imides may be useful as high-temperature films and coatings and the 4,4'-isomer system should exhibit some highly sought-after properties as a matrix resin in graphite-reinforced structure.

Future Plans: Evaluation of the adhesive properties of these polyamide-imides is planned for the future. The ability to spin fiber from these polymers will also be evaluated. Quantitative measurements will be made on polymer moldings to determine toughness. A scale-up of the resin will be made and composites fabricated and evaluated.

Figure 8(a).
<table>
<thead>
<tr>
<th>Polyimide</th>
<th>Inherent Viscosity</th>
<th>Tg(C)</th>
<th>Tensile Str. (Ksi)</th>
<th>Yield Str. (Ksi)</th>
<th>Tensile Modulus (Ksi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3,3&quot; - DABA/BTDA</td>
<td>0.93</td>
<td>283</td>
<td>14.1</td>
<td>8.8</td>
<td>476</td>
</tr>
<tr>
<td>4,4&quot; - DABA/BTDA</td>
<td>1.26</td>
<td>-</td>
<td>26.1</td>
<td>14.3</td>
<td>1068</td>
</tr>
<tr>
<td>3,4&quot; - DABA/BTDA</td>
<td>0.82</td>
<td>310</td>
<td>18.4</td>
<td>9.5</td>
<td>555</td>
</tr>
<tr>
<td>4,3&quot; - DABA/BTDA</td>
<td>0.95</td>
<td>313</td>
<td>17.1</td>
<td>9.0</td>
<td>501</td>
</tr>
<tr>
<td>LaRC-TPI</td>
<td>0.91</td>
<td>236</td>
<td>16.5</td>
<td>11.1</td>
<td>464</td>
</tr>
</tbody>
</table>

Figure 8(b).
Research Objective: To develop an understanding of crystallinity in thermoplastic polymers and learn how to exploit it in polyimides such as LARC-TPI. High levels of crystallinity generally tend to improve processing characteristics. Semi-crystalline polymers, also, have higher moduli and are more solvent resistant than their amorphous counterparts.

Approach: Samples of LARC-TPI, a semi-crystalline thermoplastic resin, were synthesized by different procedures, and, in one case, subjected to an annealing treatment. They were then analyzed using wide angle x-ray scattering (WAXS) techniques to provide a better understanding of the crystalline forms of this material.

Accomplishments: Comparative x-ray diffractograms of LARC-TPI powder are shown on the accompanying figure (the intensity of the y-axes for each curve has been shifted for super position). This complex imide structure, which had previously been thought to exist only in the amorphous form (bottom curve), is shown to be capable of crystallizing in at least three different forms. The diffractogram of the commercial powder shows one crystalline form. The material in the upper curve was produced by a different synthetic route developed at LaRC. This sample exhibits a higher degree and slightly different type of crystallinity than the commercial sample. Either of these crystalline materials may be annealed at 301°C, slightly above their melting point, to produce the third crystalline form shown in the figure. This is the thermodynamically stable form of the polymer. Further heating to 350°C destroys all crystallinity and affords an amorphous polymer.

Significance: The data on the semi-crystalline commercial LARC-TPI has been correlated with rheological data on melts of this same crystalline form to help in the understanding of how crystalline melts affect viscosity/processability of polymers. To date this novel crystallinity in LARC-TPI has allowed us to prepare well consolidated graphite-reinforced composites. X-ray diffractograms have proven invaluable in helping to define the level of crystallinity in the LARC-TPI composites and neat resin moldings.

Future Plans: Rheological data will be generated on the new crystalline form of LARC-TPI that was prepared at LaRC and will be compared to existing data on the commercial form. Composites prepared from the LaRC form will be compared with existing data on composites from the commercial polymer.
COMAPRED DIFFRACTOGRAMS FOR DIFFERENT CRYSTAL FORMS OF LARC-TP1

Figure 9(b).
LARC-TPI COMPOSITES VIA NEW SLURRY PROCESS

Norman J. Johnston, Robert M. Baucom and Terry L. St. Clair
Polymeric Materials Branch
Ext. 3044 February 1987
RTOP 505-63-01

Research Objective: To improve the flow properties of thermoplastic polyimides in order to use them as matrices for high performance composites.

Approach: Standard high molecular weight polyimides have melt viscosities so high they are difficult to process. By adding to these materials a low molecular weight semicrystalline polyimide powder that has a very low melt viscosity, a polymer blend is obtained whose melt flow properties are greatly enhanced.

 Accomplishments: Insoluble LARC-TPI semicrystalline polyimide powder was added in varying percentages to LARC-TPI polyamide-acid diglyme solution, creating a stable slurry used to prepreg onto carbon fiber. The insoluble 2-4 micron particles nicely penetrated the 12,000 filament tow and were evenly distributed on the fiber, the polyamide-acid solution acting as a binder (see SEM).

Melt flow of the polymer blend varied with the percent powder as the chart shows and flow enhancement up to five times that of the virgin polyimide without powder was observed. Standard thermoplastic molding procedures (650°F/300 psi/1 hr) were employed to fabricate void-free well-consolidated composites whose short beam shear and flexure strengths at room temperature were extremely high and were 66 and 100 percent higher, respectively, than those of composites made without the semicrystalline powder. Flexure strengths at 350°F were also improved 100 percent.

Significance: LARC-TPI and polyimidesulfone (PISO2) compositions doped with semicrystalline polyimide powders are excellent candidate matrices for high performance composites applications such as ATF, high speed commercial transports, and rocket components.

Future Plans: Blends of other polyimide compositions such as Langley's PISO2 with LARC-TPI and other semicrystalline powders are being studied. Engineering properties of LARC-TPI and PISO2 graphite composites and neat resin moldings are being obtained. Thermoforming and filament winding studies are underway.

Figure 10(a).
LARC-TPI COMPOSITES VIA NEW SLURRY PROCESS

CHEMISTRY

Polyamideacid solution

\[
\begin{align*}
\text{Z} &= \text{H}_2\text{SO}_2 \\
\text{Semicrystalline polyimide solid}
\end{align*}
\]

PREPREG SEM

ENHANCED MELT FLOW

LARC-TPI COMPOSITE MECHANICAL PROPERTIES

Short beam shear strength, psi

<table>
<thead>
<tr>
<th>% LARC-TPI Semicrystalline powder</th>
<th>0</th>
<th>33</th>
<th>50</th>
<th>67</th>
</tr>
</thead>
<tbody>
<tr>
<td>RT</td>
<td>9</td>
<td>11</td>
<td>15</td>
<td>199</td>
</tr>
<tr>
<td>350°F</td>
<td>11</td>
<td>9</td>
<td>15</td>
<td>199</td>
</tr>
</tbody>
</table>

Figure 10(b).

Consolidated void-free laminates

Flexure strength, psi

Melt flow

<table>
<thead>
<tr>
<th>% LARC-TPI Semicrystalline powder</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
</tr>
<tr>
<td>33</td>
</tr>
<tr>
<td>50</td>
</tr>
<tr>
<td>67</td>
</tr>
</tbody>
</table>
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THE EFFECT OF DIAMIC ACID ADDITIVES ON THE PROCESSABILITY OF POLYIMIDES

Diane M. Stotley, Harold D. Burks and J. Richard Pratt
Polymeric Materials Branch
Ext. 3041 June 1987
RTOP 506-43-11

Research Objective: Conventional polyimides are tough, flexible, thermooxidatively stable and solvent resistant, but they are very difficult to process. LARC-TPI and 422 are polyimides developed at NASA-Langley that possess somewhat improved processability; however, further improvements are necessary to expand their use as high performance matrix resins. The current research was focused on improving the melt viscosity of polyimide resins by incorporation of diamic acid additives.

Approach: Compatible low molecular weight diamic acid additives were synthesized and added at varying concentrations to the polyamic acid resins of LARC-TPI and 422. The effect of incorporating these additives on the processability of LARC-TPI was determined by characterizing polymer films with thermal mechanical analysis, while the effect on 422 was determined by evaluating films and the melt flow properties of molding powders in a capillary rheometer.

Accomplishments: The apparent viscosity of the 422 control was lowered by an order of magnitude with the use of 2.5% of selected additives. This decrease in melt viscosity was accomplished with only a slight decrease in glass transition temperature. When composites were prepared from the TPI system containing a diamic acid additive the increased flow resulted in better consolidation which translated into improved room temperature and elevated temperature mechanical properties.

Significance: The incorporation of diamic acid additives into polyimides results in a significant decrease in melt flow properties extending their use as processable matrix resins.

Future Plans: This approach to improving resin processability is being evaluated and further developed by a variety of aerospace material suppliers and users.
APPARENT VISCOSITIES OF 422 COPOLYMIDE WITH ADDITIVES

- ▲ 422
- □ 422/An - BTDA-An
- ○ 422/An - PMDA-An
- ○ 422/PA - pPDA-PA

Apparent viscosity, Pa·sec

Strain rate, sec⁻¹

Figure 11(b).
COMPOSITE MECHANICAL PROPERTIES

Short beam shear strength, Ksi

Figure 11(c).
MECHANICS OF MATERIALS
- Inelastic constitutive behavior
- (Strength) Failure criteria
- Life prediction methodology

Stress analysis of cracks

Fatigue crack growth

Metallic Alloys

Figure 12.
<table>
<thead>
<tr>
<th>FY91</th>
<th>FY90</th>
<th>FY89</th>
<th>FY88</th>
<th>FY87</th>
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<tbody>
<tr>
<td><strong>EXPECTED RESULTS</strong></td>
<td>Strength and life prediction methodology</td>
<td>Fatigue and fracture technology</td>
<td>Guidelines for constituent property requirements</td>
<td>Efficient tools for computational fracture mechanics analyses</td>
</tr>
<tr>
<td><strong>MAJOR THRUST</strong></td>
<td>Metals and metal matrix composites</td>
<td>Composites</td>
<td>Computational methodology</td>
<td></td>
</tr>
<tr>
<td><strong>FIVE YEAR PLAN</strong></td>
<td>Short crack growth</td>
<td>3-D crack closure</td>
<td>Life prediction methods</td>
<td>Metal matrix failure mechanisms</td>
</tr>
</tbody>
</table>
FATIGUE LIFE OF MATERIAL WITH A MACHINING SCRATCH

R. A. Everett, Jr.
Fatigue and Fracture Branch
Ext. 2715 November 1986
RTOP 505-63-01

Research Objective: To determine the effects of a machining scratch on the fatigue life of a high strength steel typical of those used in dynamic parts of helicopters.

Approach: Constant amplitude fatigue tests were run on unnotched specimens with and without a 0.002 in. deep machining scratch that could pass quality control inspections. The data were compared to determine the effects of the machining scratch on the material endurance limit. Specimens with scratches that had been shot peened were also tested to see if the compressive residual stresses from the shot peening would provide any relief from the stress concentration caused by a scratch.

Accomplishment: The figure shows fatigue life in cycles for several alternating stress levels, $S_a$. The machining scratch caused about a 40% reduction in the material's endurance limit. The tests on the specimens that had been shot peened showed that the compressive residual stresses produced by the shot peening almost eliminated the effects of the stress concentration caused by the scratch.

Significance: These tests have shown that a scratch can significantly reduce the fatigue strength of a high strength steel. The test results have also shown that shot peening can negate the effects of small stress concentrations.

Future Plans: This work is generically related to the "small crack" effects in fracture mechanics. A program is currently being planned for a study of the "small crack" effect on high strength steels.

Figure 14(a).
STRESS VS. CYCLES TO FAILURE FOR 4340 STEEL

\[ F_{tu} = 210 \text{ Ks}\]

\[ R = -1 \]

Surface Finish (RMS)
- ○ 32
- △ 32 w/scratch
- ▲ 32 scratch & peened

Figure 14(b).
THREE-DIMENSIONAL ANALYSIS OF FATIGUE CRACK CLOSURE

Fatigue and Fracture Branch
Ext. 3192 March 1987
RTOP 505-63-01

Research Objective: To develop an analytical model to calculate plasticity-induced fatigue crack closure along the crack front in finite thickness bodies.

Approach: A three-dimensional, elastic-plastic, finite-element analysis code has been written to subject FEM models to cyclic loads and to simulate fatigue crack growth by extending the crack by one element at the maximum applied stress ($S_{\text{max}}$) in each cycle. As the model is loaded/unloaded, the code computes the crack-surface contact stresses that are caused by residual plastic deformations left behind the advancing crack. The progression of crack opening/closing along the crack front is determined by tracking the loads at which the contact stresses became zero/nonzero.

Accomplishment: A model of a finite-thickness (B) plate with a through-thickness crack of initial length "Ct" was subjected to a constant-amplitude loading at a stress ratio ($S_{\text{min}}/S_{\text{max}}$) of 0.1. The figure shows the calculated progression of the opening/closing profiles on the crack surface during the loading portion of a cycle. The shaded region indicates areas where the crack is closed. At a stress of $0.1 \, S_{\text{max}}$, the crack is closed at the crack tip along the entire crack front. At $0.36 \, S_{\text{max}}$, the interior is fully open but the exterior layers are still closed. For stress levels greater than $0.56 \, S_{\text{max}}$, the entire crack was open. This sequence was reversed upon unloading.

Significance: Knowledge of the crack closure behavior along the entire crack front will significantly improve the understanding of crack growth processes in thick plates. Current experimental techniques for measuring crack closure provide a single "averaged" result for the entire crack front.

Future Plans: The analysis code will be used to perform a parametric study of loading and finite-thickness parameters that may affect crack closure behavior.

Figure 15(a).
3-D ANALYSIS OF FATIGUE CRACK CLOSURE BEHAVIOR

Figure 15(b).
Research Objective: To determine elastic properties and identify failure modes in titanium matrix composites.

Approach: The material system studied in this investigation consisted of a titanium (Ti 15-3-3-3) matrix reinforced by continuous silicon carbide (SCS6) fibers. Room temperature quasi-static tension tests were performed on matrix material, and on [0]8, [90]8, [0/90]2s, [0/±45/90]s, and [02/±45]s composite laminates in order to evaluate an existing elastic-plastic model which had successfully predicted the response of other metal matrix systems. Initial moduli and Poisson's ratios were in good agreement with those predicted by the model. However, the laminates containing off-axis plies exhibited non-linear mechanical response at stress levels which were significantly lower than the onset of yielding predicted by the model. The edge replication technique was used to document damage development in the laminates. In the edge replication technique, a permanent impression of the specimen edge is produced in a cellulose acetate film. Edge replicas were taken at various stages of the quasi-static load history of a specimen, and were subsequently examined via scanning electron microscopy.

Accomplishment: It was found that after sufficient load was applied to specimens containing off-axis plies, fibers began to separate from the matrix material. Upon unloading, the fiber and matrix were again in contact. The figure shows a representative stress-strain curve for a [0/90]2s laminate which was reloaded after fiber-matrix separation had occurred. The micrograph at the left shows the fiber and matrix in contact at low stress, while the micrograph at the right shows evidence of separation at high stress. More detailed evaluation revealed that the separations occur primarily in the reaction zone between the silicon in the fiber and the titanium in the matrix. The "knee" in the stress-strain curve is indicative of the loss of stiffness that occurs when fibers in off-axis plies are no longer in contact with the matrix, and therefore no longer carry load.

Significance: The results of this study indicate that the fiber-matrix interface in the silicon carbide/titanium system is quite weak. Improved bonding between fiber and matrix is needed in order to raise the operating stress level of this material to a desired level.

Future Plans: We are currently planning both fatigue and fracture tests on the mentioned laminates to further evaluate failure modes and processes.

Figure 16(a).
FIBER/MATRIX INTERFACE FAILURE

SCS₆/TI-15-3

$[0/90/0/90]_8$

UNLOADED

60 KSI

60

40

20

0

0.001

0.003

STRESS ksi

STRAIN

Onset of Interface Separation

Figure 16(b).
DElamination Fatigue Behavior of Composite Materials

T. Kevin O'Brien
Fatigue and Fracture Branch
Ext. 2093 January 1987
RTOP 506-43-11

Research Objective: To develop a methodology for quantifying the improvement in delamination fatigue life gained from a toughened matrix.

Approach: Graphite reinforced composites often delaminate under repeated loading as a result of interlaminar stresses. Recently, composite materials with toughened matrix resins have been developed to improve the inherent delamination resistance. Edge delamination fatigue tests were conducted on graphite reinforced composites with a variety of matrices ranging from very brittle to very tough. The number of cycles to delamination onset was recorded at several maximum cyclic strain levels. The maximum cyclic strain was substituted into a closed-form equation for the strain energy release rate G. The maximum cyclic G was then plotted versus the cycles to delamination onset. The cycles to delamination onset were presented as a function of G to obtain a generic representation of the material behavior that is independent of layup and ply thickness.

Accomplishment: The figure shows the number of constant-amplitude, tension load cycles required to form an edge delamination in the 0/90 interface of (35/-35/0/90)_s laminates as a function of the critical strain energy release rate, G^M_e. Data are shown for four graphite reinforced composites whose matrices have varying degrees of toughness. The matrix materials were (1) Narmco 5208, a 350°F brittle epoxy, (2) Hexcel H205, a 250°F epoxy, (3) Cyanamid HST7, a 350°F epoxy with a tough adhesive interleaf between each ply, and (4) ICI Polyetheretherketone (PEEK), a semicrystalline thermoplastic. As the figure indicates, there is a large difference in the static interlaminar fracture toughnesses of these materials shown on the ordinate at N=1, but the cyclic strain energy release rate endurance limits at 10^6 cycles did not differ greatly.

Significance: This study indicates that toughened resins produced only small improvements in delamination durability. Therefore, structural behavior that depends primarily on the static interlaminar toughness may be improved for a toughened matrix composite; however, structural performance in fatigue, such as delamination around an open hole, may not be significantly improved.

Future Plans: The influence of residual thermal stresses on the delamination fatigue of composites will be investigated.

Figure 17(a).
DELAMINATION FATIGUE BEHAVIOR
OF COMPOSITE MATERIALS

\[ G_c^m \text{ in-lbs/in}^2 \]

- AS4/PEEK
- AS6/HST7
- C6000/H205
- T300/5208

Cycles, N

Figure 17(b).
MATRIX YIELDING AT A DELAMINATION FRONT

John H. Crews, Jr., K. N. Shivakumar, and I. S. Raju
Fatigue and Fracture Branch
Ext. 3048 July 1987
RTOP 505-63-01

Research Objective: To analyze the influence of bond thickness on yielding at a delamination front.

Approach: A finite-element model was developed to represent a double cantilever beam (DCB) specimen that is widely used to measure delamination toughness. This DCB specimen consisted of two graphite/epoxy laminates bonded together by an epoxy adhesive layer of thickness $t$, as shown in the first figure. The finite-element model was loaded to simulate an insipient growth condition at the delamination front, and elastic stresses were calculated for the adhesive layer near the delamination. These stresses were then used with the von Mises yield condition to estimate the delamination-tip yield zone. This procedure was repeated for a range of adhesive layer thicknesses.

Accomplishment: The estimated yield zones are shown in the next figure. The smallest adhesive layer thickness ($t = 0.01$ mm) represents the co-cured case typical of composite fabrication. For this case, yielding extended throughout the adhesive layer thickness to produce a long yield zone ahead of the delamination tip. The next case ($t = 0.10$ mm) represents a typical adhesive bondline thickness and shows a larger yield zone extending through the adhesive thickness. Similarly, yielding also extended through the adhesive thickness for $t = 0.20$ mm. For even larger values of $t$, however, the yield zones were smaller. The yield zone size increased and then decreased for increasing values of $t$, as shown in the third figure.

Significance: Interlaminar fracture toughness is widely believed to be proportional to the size of the yield zone at the delamination front. The present results can be interpreted to support this belief because the trend for the yield-zone-area curve agrees qualitatively with the well-known observation that interlaminar toughness has a peak value when measured over a range of adhesive thickness. However, recent research in the Fatigue and Fracture Branch suggests that the plastic zone height rather than zone size should correlate with delamination toughness. The dashed curve for plastic zone height has an appropriate peak, which qualitatively supports the new interpretation of how adhesive layer thickness influences fracture toughness.

Future Plans: An elastoplastic numerical simulation of delamination growth will be developed to quantitatively correlate plastic zone height with energy dissipation (toughness) during delamination.

Figure 18(a).
DOUBLE CANTILEVER BEAM SPECIMEN

Figure 18(b).
YIELD ZONE AT DELAMINATION FRONT

(a) $t = 0.01$ mm
(b) $t = 0.10$ mm
(c) $t = 0.20$ mm
(d) $t = 0.36$ mm
(e) $t = 0.51$ mm
(f) $t = 0.66$ mm

Figure 18(c).
YIELD ZONE AREA AND HEIGHT

\[ \text{Area} \]

\[ \text{Height} \]

\[ P = 364 \text{ N/m} \]
\[ h = 1.65 \text{ mm} \]

Figure 18(d).
INTERLAMINAR SHEAR FATIGUE THRESHOLDS FOR COMPOSITE MATERIALS

T. Kevin O'Brien, G. B. Murri and S. A. Salpekak
Fatigue and Fracture Branch
Ext. 2093 September 1987
RTOP 505-63-01

Research Objective: To evaluate the interlaminar shear fatigue thresholds for delamination of several composite laminates using the end-notched flexure test.

Approach: The end-notched flexure (ENF) test was used to determine interlaminar shear for delamination for T300/BP907 graphite-epoxy, S2/SP250 glass-epoxy, and AS4/PEEK graphite-thermoplastic. The ENF test consists of a small, 24-ply, unidirectional specimen with an insert at the mid-plane at one end. The specimen is loaded in 3-point bending, with the loads applied across the width, by means of pins which are free to roll. Specimens were cyclically loaded at 5 Hz until the onset of stable delamination growth was detected. The fatigue behavior was quantified in terms of the maximum cyclic mode II strain energy release rate, G\text{\textit{m}ax}. Delamination-onset threshold values were defined as the G\text{\textit{m}ax} values below which no delamination occurred after one million load cycles. Static interlaminar-shear fracture toughness, G\text{\textit{m}c}, was also determined for each material.

Accomplishment: The results of the tests are shown in the figure. For all three materials, fatigue-induced delamination occurred at strain energy release rates significantly below the static fracture toughness values. Of the materials tested, the glass-epoxy material performed best in fatigue, but even for this material, the delamination threshold was only about 20 percent of the static fracture toughness. Residual static toughness tests on glass-epoxy specimens that had undergone one million cycles without delamination showed that the toughness had been significantly reduced by the cyclic loading.

Significance: These test results indicate that interlaminar-shear fatigue thresholds may be an important material property in the design of composite structures that will be subjected to load spectra containing many relatively low-level load cycles. Designs may be driven by the material fatigue performance rather than static toughness.

Future Plans: Because of the variability of properties of individual panels of PEEK, a detailed study will be conducted using a variety of tests, including static and cyclic ENF, to characterize the toughness of AS4/PEEK.

Figure 19(a).
DELAMINATION-ONSET FATIGUE BEHAVIOR
FOR INTERLAMINAR SHEAR

R=0.1  f=5 Hz

Figure 19(b).
CALCULATION OF STRAIN-ENERGY RELEASE RATE DISTRIBUTION USING PLATE ANALYSIS

John D. Whitcomb and K. N. Shivakumar
Fatigue and Fracture Branch
Ext. 3046 November 1986
RTOP 505-63-01

Research Objective: To develop a relatively inexpensive technique for calculating strain-energy release rate for use in predicting delamination in three-dimensional bodies.

Approach: Previously, the calculation of the distribution of strain-energy release rate for three-dimensional (3-D) bodies required 3-D solids analysis, which is very expensive. To obtain the strain-energy release rate more economically, a plate analysis technique was developed that is inherently much less expensive than 3-D solids analysis.

Accomplishment: A popular technique for calculating strain-energy release rate is the virtual crack closure technique. This technique relates strain-energy release rate to the energy required to close a crack over a very small distance. Traditional implementation of this technique requires parameters which are available from 3-D solids analysis but not from plate analysis. In the current effort, a virtual crack closure technique was developed which only includes terms available from plate analysis. As a check of the new method, analyses of a transversely-loaded, delaminated region were performed using plate analysis and 3-D analysis. The figure shows a sketch of the configuration and the distribution of strain-energy release rate obtained using the two analyses. The agreement is quite good.

Significance: It is economically impractical to use 3-D solids analysis for analyzing numerous configurations. In many cases, plate analysis offers a much less expensive alternative. Using the formula developed in this effort will permit relatively inexpensive calculation of strain-energy release rates for use in delamination prediction.

Future Plans: The possibility of estimating the mode I and mode II components of the total strain-energy release rate will be investigated.

Figure 20(a).
Distribution of Strain-Energy Release Rate

(For X = a/2)

3-D Solids Analysis

Plate Analysis

Strain-Energy Release Rate, J/m^2

Y/a

0 0.1 0.2 0.3 0.4 0.5

12 10 8 6 4 2

Figure 20(b)
BOUNDARY FORCE METHODS FOR ANALYZING CRACKED LAMINATES

P. W. Tan and C. A. Bigelow
Fatigue and Fracture Branch
Ext. 3047 January 1987
RTOP 505-63-01

Research Objective: To extend the Boundary Force Method (BFM), a form of an indirect boundary element method, to the analysis of orthotropic laminates.

Approach: To extend the BFM to composite materials, the orthotropic elasticity solution for a concentrated horizontal and vertical force applied at a point in a cracked infinite sheet was used as the fundamental solution. The necessary stress functions for this fundamental solution were formulated using the complex variable theory of elasticity. The simple configuration of a center-crack specimen subjected to uniaxial tension was used to evaluate the BFM for composite materials.

Accomplishment: The figure shows the normalized stress-intensity factors for a center-crack tension specimen with H/W = 3 for three graphite/epoxy laminates: [0/±45/90]s, [±45]2s, and [0]. The curves represent the current BFM results and the symbols represent results from a boundary integral solution by Snyder and Cruse. As shown in the figure, the BFM results agree well (within ±3 percent) with results from Snyder and Cruse. This close agreement validates the extension of the BFM to composite laminates.

Significance: The orthotropic BFM can now be applied to complex composite configurations for which no solutions exist. In the BFM, the fundamental solution models the crack faces, exactly satisfying the stress-free conditions there; thus, the numerical analysis does not have to model the crack faces. Only the boundaries of the region of interest are modeled, resulting in a significant savings in the number of degrees of freedom required for an accurate analysis and, consequently, in the time needed for modeling.

Future Plans: A concentrated moment will be used with the horizontal and vertical forces in the formulation of the fundamental solution to speed convergence. More complex configurations for which orthotropic solutions are currently not available will be analyzed using the BFM.


Figure 21(a).
BOUNDARY FORCE METHOD FOR ANALYZING CRACKED LAMINATES

![Graph showing the relationship between crack length and stress intensity factor](image)

\[ \frac{K_I}{S\sqrt{\pi a}} \]

\[ \text{versus} \quad 2a/W \]

**Figure 21(b).**
STRAIN ENERGY RELEASE RATES FOR EDGE-DELAMINATED COMPOSITE LAMINATES

I. S. Raju, J. H. Crews, Jr. and M. A. Aminpour
Fatigue and Fracture Branch
Ext. 3178 March 1987
RTOP 505-63-01

Research Objective: To evaluate the accuracy of finite-element models used to obtain mode-I, mode-II, mode-III and total strain energy release rates for edge-delaminated composite laminates.

Approach: Composite laminates have thin resin layers of thickness 0.0004 in. (about 1/20 of the ply thickness) between individual plies. Finite-element modeling of such a practical laminate requires considerably larger number of degrees of freedom than models in which the resin layers are neglected ("bare" interface models). Therefore, in this research the strain energy release rates obtained with both the resin layer models and the "bare" interface models are compared. Both the models used a quasi-three-dimensional finite-element analysis and a virtual crack closure technique to compute the strain energy release rates.

Accomplishment: The figure shows the mode-I, mode-II, mode-III and the total strain energy release rates for an edge-delaminated [0/±35/90]s laminate with a delamination at the -35/90 interface. The strain energy release rates are plotted against the size of the delamination tip elements (Δh). The abscissa is plotted on a log scale. The "bare" interface model (circular symbols) do not show any convergence for the individual modes as smaller delamination tip elements are used, while the total strain energy release rates do remain unchanged with mesh refinement. In contrast, the resin layer model (horizontal dashed lines) shows convergence for the individual as well as the total strain energy release rates with mesh refinement. The reason for the non-convergence of the "bare" interface model was explored and it was determined that the non-convergent behavior was inherent in the elasticity formulation of problems involving crack tips between dissimilar materials. Therefore, the non-convergence of the finite-element solution was to be expected. Although the "bare" interface model does not produce accurate results for small elements, the results for larger elements (Δh = .25 to .50) were within four percent of results from the resin model.

Significance: The current research explained the difference in convergence behavior of the "bare" and resin interface models. The research also showed that the "bare" interface models, which are easier to model and require significantly fewer degrees of freedom than resin layer models, can be useful if used judiciously.

Figure 22(a).
STRAIN ENERGY RELEASE RATES FOR EDGE DELAMINATIONS

Figure 22(b).
FIBER-RESIN MICROMECHANICS ANALYSIS OF DELAMINATION

John H. Crews, Jr., K. N. Shivakumar and I. S. Raju

Fatigue and Fracture Branch
Ext. 3048 May 1987
RTOP 505-63-01

Research Objective: To develop a micromechanics analysis of resin yielding near the delamination front in a double cantilever beam (DCB) specimen.

Approach: A local-global approach was used with a finite-element procedure to analyse a DCB specimen. First, a 3-D finite-element model was developed for an orthotropic, homogeneous DCB specimen and was used to calculate displacements near the delamination. The displacements were found to be uniform along most of the delamination front, varying only near the specimen edge. The displacements computed for the interior of the specimen were then imposed on a local fiber-resin model of a small 3-D region near the delamination. The DCB sketch in the figure shows this local 3-D region, which includes the four fibers closest to the delamination. For convenience, the fibers were assumed to be arranged in a square array; therefore, local symmetry existed about a plane through the center of the fibers. This local fiber-resin model had about 23,000 nodes and 69,000 degrees-of-freedom, and was analyzed using the LaRC CDC-205 (VPS-32) super computer.

Accomplishment: Elastic stresses were computed for a T300/5208 graphite epoxy DCB specimen, loaded up to the critical condition for delamination growth \( (G_{IC} = 85 \text{ J/m}^2) \). The local stresses corresponding to delamination growth were then used with the von Mises yield criterion to estimate the extent of resin yielding near the delamination front. The shaded regions in the figure indicate yielding in a section \( (x = 0 \text{ plane}) \) through the model and along its front face. Yielding developed in the resin-rich layer ahead of the delamination, as expected; however, it also developed in three regions between the fibers, because of stress concentrations in those regions. These results suggest that delamination is accompanied by energy-dissipating yielding within the laminate, even for this brittle T300/5208 material system.

Significance: The yielding in this fiber-resin model was compared to that for a crack in an all-resin specimen loaded to the same critical growth condition. The yield zone area for the fiber-resin case was larger than in the all-resin case. The fibers appear to promote yielding and therefore should cause the laminate fracture toughness to be greater than the all-resin case. This trend has been observed for low-toughness resins but has previously been attributed to “fiber-bridging” effects. This study has identified yielding between adjacent fibers as an energy-dissipation mechanism during delamination.

Future Plans: The present elastic micromechanics study will be extended to include elastoplastic analyses of tough-resin laminates.

Figure 23(a).
FIBER-RESIN MICROMECHANICS ANALYSIS OF DELAMINATION

T300/5208 Graphite/Epoxy

\[ V_f = 0.63 \]

\[ G_I = 85 \text{ J/m}^2 \]

Figure 23(b).
Research Objective: To evaluate the mode-II strain energy release rate, $G_{II}$, for the end notched flexure (ENF) specimen using beam theory and determine its validity by comparison with a finite-element analysis.

Approach: The ENF test is used to determine the mode-II critical strain energy release rate for delamination in composite materials, and is one of four test methods being considered as ASTM standard tests for measuring interlaminar fracture toughness. Therefore it is necessary to establish a theoretical, as well as experimental, basis for calculation of $G_{II}$ from this test. The ENF test is a three point bend test of a 24 ply unidirectional composite with a delamination insert at the mid-surface on one end. The crack length, $a$, and beam span, $l$, as well as material properties, may influence the ENF test. Therefore, theoretical estimation of $G_{II}$ for this test must be accurate for any material and $a/l$ ratio chosen. To demonstrate this accuracy, a finite element analysis was performed. Finite-element analysis solves the boundary problem accurately and yields $G_{II}$ and compliance values. Classical beam theory with shearing deformation gives the corresponding approximate values. The two methods were compared for the ENF test for a wide range of $a/l$ ratios and material properties.

Accomplishment: A finite-element analysis of the ENF specimen was performed for various delamination lengths. A wide range of material properties, including glass/epoxy and graphite/PEEK, were analyzed. The virtual crack closure technique (VCCT) was used to calculate $G_{II}$ using local forces and displacements around the delamination tip. Alternatively, the compliance values from the analysis are used to calculate $G_{II}$ as a global change in energy with delamination length. The values of $G_{II}$ thus obtained on local and global basis agreed within one percent. The equation for compliance, which includes bending and shear deformation effects, is available in the literature. The derivative of this equation yields an expression for approximate calculation of $G_{II}^{SH}$. As shown in the figure, the corresponding $G_{II}^{SH}$ values were in close agreement with the finite-element solution; the differences were less than eight percent.

Significance: This study indicates that beam theory with shear deformation gives a fairly accurate value of $G_{II}$ in the ENF test, and establishes the validity of using the rate of change in compliance with crack length to measure $G_{II}$ in the ENF test.

Figure 24(a).
COMPARISON OF $G_{II}$ FROM FINITE ELEMENT ANALYSIS AND BEAM THEORY WITH SHEAR DEFORMATION

![Graph showing comparison of shear moduli $G_{SH_{II}}$ and $G_{FE_{II}}$ against $a/L$.]

Figure 24(b).
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FINITE-ELEMENT-ALTERNATING METHOD FOR CRACK ANALYSES

Fatigue and Fracture Branch
Ext. 3178 July 1987
RTOP 505-63-01

Research Objective: To study the performance of the finite-element-alternating method in the analysis of cracked solids by comparing it to the conventional finite-element method.

Approach: A three-dimensional finite-element-alternating method code was written for stress-intensity factor analyses of elliptical or part-elliptical cracks in solids. This code uses the solution of an embedded elliptical crack in an infinite solid subjected to arbitrary crack-face tractions. The method is based on Schwarz-Neumann alternating method. The finite-element method is used in conjunction with the continuum solution method to alternate between the two methods to satisfy the required boundary conditions.

Accomplishment: The first figure shows the stress-intensity factor distributions obtained by the finite-element-alternating and conventional methods for a quarter-elliptical corner crack subjected to remote tension. The quarter-elliptical crack is very oblong and shallow. The uncracked body was modeled with a simple rectangular mesh with about 3000 degrees of freedom. The stress-intensity results agreed well with the conventional finite-element method using about 6500 degrees of freedom. The computing time for the alternating method was about 450 seconds and for the conventional method was about 400 seconds on the VPS-32 computer. For the alternating method, most of the computing time was used in factorizing the global stiffness matrix. But after this is accomplished, little additional computing time is required to analyze other crack shapes and sizes.

Significance: The second figure shows the stress-intensity factors for three crack shapes and three crack sizes (9 crack configurations) obtained by this method. The computing time required for these cases is 630 seconds. The conventional finite-element method requires nine separate calculations and therefore requires $9 \times 400 = 3600$ seconds. Thus, the computing time with the alternating method was about one-sixth that for the conventional finite-element method. The alternating method is a very efficient tool for analyzing a range of crack configurations. Further, simple rectangular idealizations are much easier to model than the complex modeling needed with the conventional finite-element method.

Future Plans: The finite-element-alternating method will be applied to configurations for which very few stress-intensity solutions are available. Further, the performance of the method will be studied in mixed-mode loading situations.

Figure 25(a).
STRESS-INTENSITY FACTORS FOR A CORNER CRACK

\[ \frac{K}{S_t \sqrt{\pi a/G}} \]

Finite-element-alternating method

Finite-element method

\[ \frac{a}{c} = 0.2 \]
\[ \frac{a}{t} = 0.1 \]

CORNER CRACK IN A PLATE

Figure 25(b).
STRESS-INTENSITY FACTORS FOR VARIOUS CRACK SHAPES AND SIZES

\[ \frac{K}{S_t \sqrt{\pi a/\theta}} \]

Figure 25(c).
APPLIED MATERIALS
MATERIALS FOR SPACE STRUCTURES

DIMENSIONAL STABILITY

Laser Interferometry
Durable Materials
Coated Composite Tube
Radiation Effects

PROTECTIVE COATINGS

AIRCRAFT COMPOSITE MATERIALS

Material Forms and Processing
Material Testing and Analysis

CARBON-CARBON COMPOSITES

Oxidation Resistant Hot Aerostructure

Figure 26.
### APPLIED MATERIALS BRANCH
#### FIVE YEAR PLAN

<table>
<thead>
<tr>
<th>MAJOR THRUST</th>
<th>FY87</th>
<th>FY88</th>
<th>FY89</th>
<th>FY90</th>
<th>FY91</th>
<th>EXPECTED RESULTS</th>
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<td>New concepts for space-stable materials</td>
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<td>Life assessment of SDA C-C for NASP control surfaces</td>
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<td>Strong delamination resistant, lightweight C-C for aerostructure</td>
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<td>Extended-life C-C for TPS and hot structure</td>
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<td><strong>Composite materials for rotorcraft and aircraft structures</strong></td>
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<td>Efficient composite materials for structural applications</td>
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<td>Demonstrated tough, efficient, and cost effective materials</td>
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<td>Demonstrated long-term durability for tough composites</td>
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**Figure 27.**
DEVELOPMENT OF PROTECTIVE COATINGS FOR COMPOSITE TUBES

Louis A. Teichman
Applied Materials Branch
Ext. 3027  October 1986
RTOP 506-43-21

Research Objective: To develop protective coatings for graphite/epoxy tubes proposed for Space Station truss structure.

Approach: This research was performed by the Boeing Aerospace Corporation under NASA contract. Boeing/NASA defined the structural configuration of the composite tubes which maximizes stiffness and results in little microcracking. Coatings were developed which are suitable for use in the low Earth orbit (LEO) environment. Coated composite tubes were subjected to a simulated LEO environment and tested for degradation of mechanical and thermo-optical properties.

Accomplishment: Composite tubes constructed of a high-modulus fiber (Union Carbide P75S) and a 350°F-cure epoxy resin (Fiberite 934) and layed up with alternating shallow (20°) angles and 0° plies result in a stiff (35 Msi) structural element suitable to be used in a Space Station truss. When these tubes were wrapped with a chromic acid anodized aluminum foil or a sputter-coated (Al/SiO2) aluminum foil, they yielded a broad range of thermo-optical properties (solar absorptance, $\alpha_s = 0.20$ to 0.35; thermal emittance, $\varepsilon = 0.15$ to 0.25). The coated aluminum wrapping served to moderate thermal excursions in LEO without significant change in modulus of thermal expansion properties. The program also demonstrated that measurements of simple optical properties, from which $\alpha_s$ and $\varepsilon$ are derived, are similar for flat and curved surfaces and might therefore eliminate the need for extensive optical testing of tubes. Furthermore, when the coated tubes were subjected to such LEO parameters as thermal cycling, solar radiation, atomic oxygen, and vacuum for periods equivalent to several months in space, they suffered no microcracks or serious loss of structural or thermo-optical properties.

Significance: The project demonstrated that high quality composite tubes can be readily fabricated from graphite/epoxy prepreg adhesively wrapped with a coated aluminum foil. These tubes showed promise of meeting the structural requirements for a Space Station truss structure with regard to stiffness, crack resistance, and durability in the expected space environment.

Future Plans: In the coming year, coated composite tubes will be subjected to 10,000 thermal cycles between +150°F and -150°F. In addition, tubes will be fabricated in lengths up to 10 ft with a straightness of ±15 mil. Also, coated composite tubes will be subjected to other LEO parameters for greater periods of time.

Figure 28(a).
LONGITUDINAL ELASTIC MODULUS AS A FUNCTION OF PLY ORIENTATION

Analysis of P75S/934 flat composite plate

\( (0_2, \pm \theta, 0_2)^s \)

Elastic modulus, Msi

Ply orientation, \( \theta \), deg

No foils

2 mil Al foil one side

2 mil Al foil both sides

Figure 28(b).
LONGITUdINAL COEFFICIENT OF THERMAL EXPANSION AS A FUNCTION OF PLY ORIENTATION

Analysis of P75S/934 flat composite plate

(0₂, ±θ, 0₂)s

- 2 mil Al foil both sides
- 2 mil Al foil one side
- No foils

Coefficient of thermal expansion, μ in/in/F

Ply orientation, θ, deg

Figure 28(c).
REFLECTANCE OF CHROMIC ACID ANODIZED ALUMINUM SPECIMENS

Reflectance, %

Wavelength, nanometers

Figure 28(d).
A COMPARISON OF THE EFFECTS OF SIMULATED LOW-EARTH AND GEOSYNCHRONOUS ORBIT EXPOSURE ON COMPOSITE MATERIALS

Joan G. Funk
Applied Materials Branch
Ext. 4582 June 1987
RTOP 506-43-21

Research Objective: To evaluate the effects of simulated space environmental exposure on several polymer-matrix composites for spacecraft applications.

Approach: Characterize the materials' response to 500 thermal cycles simulating low-earth orbit (LEO) and to $10^{10}$ rads exposure to 1 MeV electrons followed by 500 thermal cycles simulating geosynchronous orbit (GEO). Two different upper temperature limits, 65°C and 93°C, were used during thermal cycling to insure that the glass transition temperature ($T_g$) of the material was not exceeded. The lower thermal cycling temperature was -150°C. The amount of microdamage in each composite system was measured by the microcrack density as determined by X-ray photography.

Accomplishment: The figure shows the X-ray photographs for T300/934. The baseline material, which had not undergone either thermal cycling or radiation exposure, shows no evidence of microcracking. The X-ray photograph of the material exposed to simulated LEO showed microdamage in the form of microcracks in the 0° and 90° directions. The T300/934 material exposed to simulated GEO had an even higher microcrack density with microcracks in all ply directions.

The table summarizes the observed microdamage for all six of the commercially available aerospace material systems studied and gives a brief description of the materials. For the baseline materials, only the C6000/P1700 system exhibited microcracks in the as-fabricated condition. The simulated LEO environment produced major microdamage in all the composites except the T300/BP907 system and the AS4/PEEK system. The GEO simulated environment produced microcracking in all systems. Overall, the microcrack density for all materials was greater than that found following the LEO simulation showing that radiation affects the matrix in all of the composites and thus the thermal cycling response. The results show that the composite system with the least sensitivity to microdamage during LEO simulated exposure was the most damaged by simulated GEO environment. Thus, composite materials suitable for LEO spacecraft applications may not have the required durability for long-term GEO missions.

Significance: This study evaluated the space durability of a variety of composites with matrices ranging from a brittle epoxy to a tough semicrystalline thermoplastic.

Future Plans: The composite materials which showed the best performance in this study will be evaluated to determine which properties lead to their improved space durability.

Figure 29(a).
Figure 20(b): X-ray photographs of T300/934

Baseline
500 Thermal cycles
Irradiated (10^{10} rads)
and
500 thermal cycles

1 cm
**SUMMARY OF MICRODAMAGE IN COMPOSITE MATERIALS**

**MICROCRACK DENSITY, CRACKS/CM**

<table>
<thead>
<tr>
<th>MATRIX DESCRIPTION</th>
<th>BASELINE</th>
<th>THERMAL CYCLED</th>
<th>IRRADIATED AND THERMAL CYCLED</th>
</tr>
</thead>
<tbody>
<tr>
<td>T300/934 177°C CURE, MY720 BASED EPOXY</td>
<td>0</td>
<td>7</td>
<td>17</td>
</tr>
<tr>
<td>T300/BP907 177°C CURE, SINGLE PHASE TOUGHENED EPOXY</td>
<td>0</td>
<td>0</td>
<td>&gt;50</td>
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<tr>
<td>T300/CE339 121°C CURE, 2-PHASE ELASTOMER TOUGHENED EPOXY</td>
<td>0</td>
<td>8</td>
<td>25</td>
</tr>
<tr>
<td>C6000/P1700 POLYSULFONE, AMORPHOUS THERMOPLASTIC</td>
<td>5</td>
<td>21</td>
<td>24</td>
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<tr>
<td>AS4/PPS POLYPHENYLENE Sulfide SEMICRYSTALLINE THERMOPLASTIC</td>
<td>0</td>
<td>19</td>
<td>22</td>
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<tr>
<td>AS4/PEEK POLYETHERETHER KETONE, SEMICRYSTALLINE THERMOPLASTIC</td>
<td>0</td>
<td>1</td>
<td>5</td>
</tr>
</tbody>
</table>

1 Irradiated to 10^10 Rads at 5 x 10^7 Rads/hr followed by 500 thermal cycles.
2 Cracking and delamination extensive.

Figure 29(c).
THERMALLY INDUCED TWIST IN COMPOSITE TUBES

Stephen S. Tompkins and Carl Q. Rousseau
Applied Materials Branch
Ext. 4558 August 1987
RTOP 481-33-13

Research Objective: To measure and model the dilatation and stress state of tubular structural elements subjected to thermal cycles typical of Earth orbiting spacecraft.

Approach: Measure the thermal expansion and distortion of high modulus graphite fiber reinforced epoxy composite tubes subjected to thermal cycles typical of the Space Station. Develop analytical models of the thermal expansion, distortion, and stress state using elasticity theory with temperature-dependent material properties. Compare the analytical and experimental results and modify the analysis as required.

Accomplishment: Thermal distortions of tubes made of different composite materials, P75S/ERLX1962A, T300/ERLX1962A and AS4/976, were measured during thermal cycling between -200°F and 200°F. Tubes of each material with symmetric-balanced, symmetric and asymmetric wall construction, about 70 mils thick and 2 inches in diameter were studied. All of the tubes exhibited measurable twist due to temperature change. The twisting is due to the difference in the radial positions of off-axis layers which resulted in a moment about the tube axis. Thermal shear strain typical of the tubes with asymmetric wall construction is shown in the first figure. The experimental results are in good agreement with a generalized plane strain elasticity analysis, with temperature dependent material properties, developed for the tubes.

All of the tubes exhibited thermal twist, as shown in the chart. Both the symmetric and the asymmetric tubes had about the same amount of twist. Even the tube with the symmetric-balanced wall twisted, although the magnitude of twist was very small compared to that of the other tube configurations.

Significance: Twisting of tube elements can have serious consequences if the tube is part of a high-precision structure. In long unbalanced tubes, when the temperature change is large, the rotation of one end relative to the other can be significant. Over a long service life, such as 30 years, thermal fatigue could be detrimental to the structure. Also, this thermal twisting could introduce torsional loads into a structure that was not designed for torsional loads. Symmetric balanced fiber layups will avoid such consequences.

Future Plans: Future research will investigate the stresses induced in the end fittings and joints by thermal twisting of the tubular element.
EFFECT OF COMPOSITE TUBE WALL CONFIGURATION ON THERMALLY
INDUCED TWIST

Thermal Shear Strain in P75/ERLX1962A
[-102/012] 2" Dia Composite Tube.

Shear Strain, \( \mu \text{rad} \)

Temperature, K

Figure 30(b).
<table>
<thead>
<tr>
<th>TWIST IN 23' TUBE</th>
<th>ΔT=-600°F</th>
<th>0.006 deg.</th>
<th>12.3 deg.</th>
<th>11.2 deg.</th>
</tr>
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<tbody>
<tr>
<td>ΔT=-500°F</td>
<td>0.005 deg.</td>
<td>10.2 deg.</td>
<td>9.3 deg.</td>
<td></td>
</tr>
</tbody>
</table>

**TUBE WALL LAYUP**

- Symmetric balanced
- Symmetric unbalanced
- Asymmetric unbalanced

**TUBE WALL LAYUP**

- [10/05/-102/05/10]τ
- [10/012/10]τ
- [102/012]τ
THERMALLY STABLE GRAPHITE-REINFORCED ALUMINUM ALLOYS

Stephen S. Tompkins and Gregory A. Dries
Applied Materials Branch
Ext. 4558 December 1986
RTOP 506-43-21

Research Objective: To develop continuous-fiber-reinforced aluminum-matrix composites that are thermally and mechanically stable over long times for dimensionally critical spacecraft.

Approach: Define high strength aluminum alloys that can be used as the matrix for P100/Al composites. Determine the thermal expansion behavior before and after long-time thermal cycling between -250°F and 250°F. Measure thermal expansion behavior using a laser interferometric dilatometer.

Accomplishments: Large residual strain and thermal strain hysteresis is typical of the thermal expansion behavior of P100/6061 composite shown in the first figure. This undesirable response has been significantly reduced, but not eliminated, by heat treatment. Heat treatment was not successful in increasing the yield strength of the alloy to the level needed to eliminate hysteresis. Significant loss of important alloy elements during fabrication was a key reason for lower than expected strengthening as seen in the second figure. Several high strength Al alloys were therefore investigated.

Combinations of commercial high strength aluminum alloy matrices and post-fabrication processes developed at Langley have resulted in metal-matrix composites that do not exhibit residual thermal strain or strain hysteresis during thermal cycling. The third figure shows the thermal expansion of composites composed of P100 graphite-reinforced 2024 and 201 Al alloy after 1000 thermal cycles between -250°F and 250°F. After post-fabrication processing, as well as after 1000 thermal cycles, neither composite exhibited residual strain or hysteresis.

Significance: A metal-matrix composite made with P100 and a high strength Al alloy such as 2024 or 201 provides a dimensionally stable Gr/Al composite for spacecraft that does not have the stability problems associated with moisture dryout, electron radiation, and atomic oxygen which are contributors to dimensional instability in polymer-matrix composites.

Future Plans: Future research will investigate thermal expansion behavior of Gr/Al angle-plied laminates using the high strength alloys. Analytical models of the thermal expansion of this class of material will be developed and verified.

Figure 31(a).
THERMAL EXPANSION BEHAVIOR OF P100 Gr REINFORCED HIGH STRENGTH ALUMINUM COMPOSITES

T6 Conditioned with Stress Relief at -268°F

P100 Gr/201 Al  P100 Gr/2024 Al

Figure 31(b).
A METHOD OF PREDICTING THE ENERGY-ABSORPTION CAPABILITY OF COMPOSITE SUBFLOOR BEAMS

Gary L. Farley  
Applied Materials Branch  
Ext. 2850  
February 1987  
RTOP 505-63-01

**Research Objective:** To develop a simple and accurate method of predicting the energy-absorption capability of composite subfloor beam structure.

**Approach:** Develop a basic understanding of the crushing characteristics of composite materials. Determine the effects material and component geometry variables have on the energy-absorption capability of composite materials. Compare the crushing response of circular and square cross section tubes with structural elements.

**Accomplishment:** The subfloor beam structure of a helicopter must be designed to carry non-crash loads as well as to crush in a progressive manner to dissipate energy in a crash. Different types of stiffened and sine-wave beam concepts have been evaluated as shown in the first figure. Through a comprehensive study of the crushing characteristics of composite material, energy absorbing subfloor beam concepts have been developed that are superior energy absorbers to comparable metallic beams. However, the current method of designing energy absorbing composite beams is based upon a limited beam test data base because no means of predicting the energy-absorption capability exists.

LaRC research results showed that the crushing modes of structural composite beams were similar to those of circular and square cross section tubes of similar composite material and fiber-reinforcement architecture. A hypothesis was formulated for predicting the energy-absorption capability of structural elements. The hypothesis is as follows: the crash energy-absorption capability of a structural element is the sum of the weighted average of the energy-absorption capability of its characteristic elements. Therefore, if the energy-absorption capabilities are known for composite tubes of different geometries, such as in the second figure, then the energy-absorption capability of a structural element can be predicted. Using this technique an energy-absorption capability of composite sine-wave and stiffened beams was predicted. Excellent agreement between prediction and experiment was obtained, as shown in the third figure.

**Significance:** The designer using the previously described prediction method can conduct design studies in a manner consistent with conventional structural design practice. This will reduce the cost of designing energy absorbing subfloor structure and will result in more efficient structure.

**Future Plans:** Future research will focus on the development of analytical procedures to predict the energy-absorption capability of composite tube specimens.

**Figure 32(a).**
ENERGY ABSORBING BEAM CONCEPTS

Sine-wave beam (tangent-half circle)

Circular tube stiffened beam

Rectangular tube stiffened beam

Figure 32(b).
ENERGY ABSORPTION CAPABILITY
OF SQUARE KELVAR/EPoxy TUBES

Figure 32(c).

- Internal tube width
  - □ 1.27 cm
  - △ 2.54 cm
  - ◇ 3.81 cm
  - ▲ 7.62 cm

- Specific sustained crushing stress, Nm/g

- Denotes range of data

- Symbols represent average of three tests

- Tube width/wall thickness ratio, w/t
COMPARISON OF COMPOSITE ENERGY ABSORBING BEAMS PREDICTION WITH EXPERIMENT

Figure 32(d).
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USING FUNDAMENTAL SPECTROSCOPIC DATA TO EXPLAIN CHANGES IN APPLIED PROPERTIES OF IRRADIATED POLYMERS

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Applied Materials Branch
Ext. 3892           April 1987
RTOP 506-43-21

Research Objective: To obtain a fundamental understanding of the changes caused by ionizing radiation on mechanical and electrical properties of advanced polymeric materials.

Approach: Polymer films are exposed to electron radiation for total doses up to the equivalent of 30 years exposure in geosynchronous orbit. The mechanical and electrical properties of irradiated specimens are compared to those of nonirradiated specimens to determine radiation durabilities. Spectroscopic analyses using electron paramagnetic resonance, infrared, and dielectric methods are used to determine changes in molecular structure caused by the irradiation. These combined analyses can explain which changes in the molecular structure caused the changes in the functional properties.

Accomplishment: Three generic polymeric systems have been studied: the polyetherimide Ultem, the polyimide Kapton, and the polyethylene terephthalate Mylar. Aromatic-structure-containing polymers such as these have been previously thought to be inherently radiation durable, but recent studies have shown that the durability depends on additional aspects of their molecular structures. For example, Ultem and Kapton contain the same aromatic structural components, but the effects of electron radiation on their total tensile elongations are different, as shown in the first figure. The additional molecular structure in Ultem, as shown in the second figure, is responsible for the difference. The radiation homolytically cleaves the aromatic ether bond in both polymers. The broken ether bond self-mends in Kapton. In Ultem, however, the radiation also causes hydrogen abstraction at nonaromatic sites. The abstracted hydrogen bonds to the phenyl radical at the site of the broken ether bond, thereby inhibiting self-mending. The polymer chains are thus free to crosslink, which is the cause of the large decrease in the elongation of Ultem. Similar studies have been made for the other mechanical and electrical properties of the three polymeric systems. The interpretations of the molecular data have led to consistent explanations of the radiation effects on those properties.

Significance: The identification of the molecular changes which cause the radiation-generated changes in the functional properties can suggest polymeric structures that are more radiation durable.

Future Plans: Similar studies on additional advanced polymers and studies on threshold and dose rate effects will be conducted.

Figure 33(a).
EFFECTS OF ELECTRON RADIATION ON TENSILE ELONGATION OF POLYMER FILMS

150

100

50

0

Total dose, gigarad

Elongation-to-failure, %

Kapton  ○
Ultem  ●

Figure 33(b).
ELECTRON RADIATION EFFECTS ON MOLECULAR STRUCTURES

ULTEM

KAPTON

Figure 33(c).
INNOVATIVE FABRICATION OF COMPOSITE STRUCTURES

Marvin B. Dow
Applied Materials Branch
Ext. 2850 April 1987
RTOP 505-63-01

Research Objective: To demonstrate the feasibility of pultrusion as an automated fabrication process for composite aircraft structures.

Approach: Develop tooling and techniques for pultruding stiffened panels incorporating damage tolerant design features. Pultrude a stiffened panel using thermoset-matrix composites and perform structural tests.

Accomplishment: The fabrication development is focused on pultruding graphite-epoxy plies to produce a stiffened panel of the dimensions shown in the first figure. The panel design incorporates structural efficiency and damage tolerance features such as "soft skin" and crack stoppers. These design requirements result in a complex arrangement of plies within the panel, shown in the second figure. Region 1 is the basic soft-skin layup with a high percentage of ±45° plies. Region 2 are the "J" section stiffeners which feature 0° plies for strength. Region 3 are 0° crack stopper planks buried in the skin beneath the stiffeners. Region 4 are overlay plies which provide continuous attachments between stiffeners and the skin.

To provide the required design features and to accomodate the pultrusion process, dry AS4 graphite fabrics are slit to width, knitted as preplied elements, and wound on creels. Dry fabric from the creels is pulled through a pressurized tank which wets the fibers with Shell 9310 epoxy resin. From the wetting tank, the fabric elements are pulled through a curing die which shapes and cures the panel in a single operation. The panel is oven post-cured for 2 hours at 350°F.

A pultruded panel recently fabricated in this program is shown in the third figure. The fabrication was performed by Goldsworthy Engineering, Incorporated, under subcontract to Lockheed-California Company. The plan was to pultrude 30 feet of the stiffened panel, but an accumulation of problems forced a halt after 10 feet was pultruded. A 6-foot length of the panel has been delivered to Langley for testing.

Significance: One of the greatest challenges facing the aircraft industry is to reduce the acquisition costs for composite structures to a level below that of metal structures. The pultrusion process provides a means to automate the fabrication of composites and thereby reduce the costs. Results obtained in this investigation should help establish pultrusion as an acceptable process for fabricating the complex structures required in aircraft applications.

Future Plans: Other pultruded panels will be fabricated after improvements are made in the wetting tank and the pultrusion die. Structural tests will be performed to verify the structural quality of pultruded graphite-epoxy panels.

Figure 34(a).
PULTRUDED PANEL DIMENSIONS

(All dimensions in inches)

Figure 34(b).
"J" PANEL STRUCTURE

Figure 34(c).
PULTRUDED COMPOSITE WING PANEL

Figure 34(a)

78°

21.80°
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**Figure 36.**
B₄C PARTICULATES SHOW PROMISE TO IMPROVE PROPERTIES IN ALUMINUM MATRIX COMPOSITES

William D. Brewer
Metallic Materials Branch
Ext. 4193 January 1987
RTOP 505-63-01

Research Objective: To develop 2XXX aluminum matrix composites with improved properties for high temperature aircraft structures applications.

Approach: Determine the effects of reinforcing phase, alloy chemistry, and primary and secondary processing on composite properties.

Accomplishment: Aluminum matrix materials reinforced with SiC whiskers have been shown to have properties that make them attractive for a variety of aircraft structural applications. At present, however, the whiskers are relatively expensive, about 15 percent more dense than aluminum, and have an aspect ratio sufficiently high (2:1 to 5:1 in final composite form) to result in composites with some anisotropy in properties. Boron carbide particulate reinforcement has the potential to overcome all of these shortcomings. The figure shows the strength, modulus, and strain to failure of 2124 Al/SiC and 2124 Al/B₄C composite extrusions for different heat treatments and orientation (with respect to the extrusion direction). Heat treatment has little effect on either composite, whereas the orientation effects are obviously greater for the SiC whiskers than for the B₄C particulates. In general, the properties of the two composites are about the same. However, the B₄C is about 25 percent less dense than the SiC; and thus, for the same reinforcement volume fraction, the specific properties (density adjusted) of the B₄C composite are somewhat better.

Significance: Because the B₄C is less costly, less dense, and results in composites with properties at least as good as SiC whiskers, significant payoffs in terms of structural efficiency and cost may be realized by using the B₄C reinforcements.

Future Plans: Composites processed with higher toughness alloys for the matrix and improved uniformity of distribution for the reinforcements investigated will be evaluated in terms of damage tolerance and durability.

Figure 37(a).
MECHANICAL PROPERTIES OF 2124/SiC AND 2124/B₄C COMPOSITE EXTRUSIONS

Figure 37(b).
MATERIAL PROPERTY VERIFICATION OF LaRC PROCESSED PM ALUMINUM ALLOYS

O. R. Singleton
PRC/Kentron, Inc.
Ext. 2006 March 1987
RTOP 505-63-01

Research Objective: To develop advanced aluminum alloys with improved strength and toughness properties for high temperature applications on high-speed civil transports.

Approach: Synthesize in-house research quantities of rapidly solidified, zirconium bearing PM Al alloy sheet using small consolidated billets prepared from powder supplied to LaRC from a CALAC-Alcoa contract. Compare material properties of LaRC produced sheet to those obtained from sheet produced from larger billets.

Accomplishment: In an initial phase, 31 billets of PM 2124-Zr aluminum alloy were produced from supplied powder, of which 27 were fabricated to sheet. Of these sheets, eleven contained the high 0.6% Zr used in the CALAC-Alcoa sheet material. Billet consolidation parameters of time, temperature, and vacuum were varied to achieve the desired material properties. As shown in the graph, the longitudinal and transverse tensile properties evaluated are nearly identical to those obtained on the contract study. These properties demonstrate that sheet material produced on a small laboratory scale can be utilized for advanced alloy synthesis studies.

Significance: A small in-house research facility will permit inexpensive and fast screening of innovative rapid solidification powder metallurgy aluminum alloy compositions. Rapid solidification appears to offer the most practical means to develop aluminum materials for use at temperatures of 600°F and above. Such aluminum alloys are not only lighter, but also potentially less expensive, than competing titanium alloys.

Future Plans: After completion of the material property sheet validation program, the methodology and equipment will be used to fabricate innovative P/M aluminum alloy sheet containing silicide additions. These aluminum alloys with potential for use at 600°F and above have densities under 2.9 g/cc. The research aluminum alloy powder has been purchased to specification through commercial suppliers.

Figure 38(a).
MECHANICAL PROPERTIES OF ALCOA AND LaRC CONSOLIDATED ALUMINUM P/M 2124 + 0.6% Zr SHEET

Billet wt = 100 lbs  Billet wt = 1 lb

Strengthen, ksi

Alcoa          LaRC

Long  Trans   Long  Trans

UTS  YS  UTS  YS  UTS  YS  UTS  YS

15

5

0

Elongation, percent
IMPROVED AGING CHARACTERISTICS BY MINOR ALLOYING ADDITIONS IN Al-Li ALLOYS

Linda B. Blackburn
Metallic Materials Branch
Ext. 4581 May 1987
RTOP 505-63-01

Research Objective: To develop Al-Cu-Li-Zr-X alloys that can achieve high strength levels through direct heat treatment without requiring cold deformation prior to aging.

Approach: Add minor alloying additions of Cd, In, and Sn to a baseline alloy similar in composition to the 2090 Al-Li alloy, in an attempt to promote the precipitation of strengthening phases such as T1 and m. Evaluate the effect of each addition on the aging response of each alloy at several aging temperatures. Determine the mechanical properties of peak-aged specimens and correlate microstructural features with the observed mechanical properties.

Accomplishment: Thermal analyses were conducted to identify optimum solution heat treatment temperatures in order to minimize the number of coarse constituent particles and maximize the amount of solute in solid solution. Aging response, as shown in the figure, indicates the In-bearing alloy resulted in significantly higher hardness than the other three alloys when aged at 160°C. Tensile tests conducted on specimens peak aged at 160°C also indicated the In-bearing alloy achieved the highest strengths. Charpy specimens tested in slow bend indicate the fracture toughness of the In-bearing alloy approaches that of baseline alloy and exceeds that of the Cd- and Sn-bearing alloys. TEM examination of the alloy microstructures indicates an increased density and homogeneity of the strengthening precipitates in the In-bearing alloy.

Significance: The ability to strengthen Al-Cu-Zr alloys through heat treatment alone makes it possible to consider forming the alloy in the solution treated condition before cold deformation or final aging. The material in this condition would have higher formability. The material should also be amenable to superplastic forming which would not be possible if cold deformation prior to aging were necessary.

Future Plans: The fracture toughness of each alloy will be more precisely determined and microstructural examinations will continue in order to determine the nature of the effect of minor alloying additions on the precipitation mechanisms of the strengthening phases. Mechanical property determination will be continued and sheet material will be prepared for conducting superplastic formability studies.

Figure 39(a).
ALLEVIATION OF CAVITATION IN SUPERPLASTICALLY FORMED 7475 ALUMINUM ALLOY USING POST-FORMING PRESSURE

Thomas T. Bales
Metallic Materials Branch
Ext. 3405 November 1986
RTOP 505-63-01

Research Objective: To develop the processing methodology to suppress cavitation in aluminum alloys resulting from the large strains associated with superplastic forming (SPF).

Approach: Conduct experimental studies to determine the feasibility of using post-forming pressure to heal cavitation resulting from SPF. Conduct metallographic analyses to determine the effect of various post-SPF pressures on the extent of void content.

Accomplishment: Superplasticity is the ability of selected metal alloys to undergo large strains (500-1000 percent) at elevated temperature prior to localized thinning or fracture. Processes have been developed for superplastically forming complex structural shapes in a single operation and result in significant cost savings compared to forming the same components using conventional means. When 7475 aluminum alloy sheet material is superplastically formed, internal porosity or cavitation is generated due to the metallurgical nature of the alloy. Methods to suppress or alleviate cavitation must be developed to realize the full potential offered by superplastic forming for reducing the cost of aerospace structural components. The figure shows the effects of post-forming pressure on cavitation. Specimens were superplastically formed at a temperature of 960°F using argon gas at a pressure of 100 psi. Following forming of the sheet material into the tool cavity, the post-forming gas pressure was increased and maintained for 1-1/2 hours. Several levels of post-forming pressure were investigated. Following exposure to the prescribed pressure-time profiles, metallurgical specimens were prepared and examined using a light microscope. A comparative image analyzer was used to determine the amount of porosity. As shown on the figure, the cavitation was reduced from approximately 2-1/2 percent for specimens subjected to a post-forming pressure of 125 psi to less than 1/4 percent for specimens exposed to 425 psi.

Significance: Results of this study indicate that post-forming pressure can be used to heal cavitation resulting from SPF. The technique is relatively simple and should extend the maximum forming strains for use in fabricating structural components.

Future Plans: Mechanical property tests are being conducted on specimens subjected to post-forming pressures to determine the effects of processing on material properties.
EFFECT OF POST-FORMING PRESSURE ON CAVITATION OF 7475 ALUMINUM ALLOY

960°F, 1-1/2 hours

Percent voids

Post-forming pressure, psi

Figure 40(b).
EMITTANCE/CATALYSIS COATINGS TO IMPROVE PERFORMANCE OF TITANIUM-ALUMINIDES

Ronald K. Clark and Terryl A Wallace
Metallic Materials Branch
Ext 4557 July 1987
RTOP 506-43-71

Research Objective: To develop high emittance-low catalysis-oxidation resistant coatings for advanced high temperature structural metallic materials.

Approach: Candidate coatings are selected/formulated to reduce catalysis and increase emittance. Current focus is on titanium-aluminide based material systems prepared with coatings using sputtering, physical vapor deposition, chemical vapor deposition, and slurry processes. Coated specimens are screened for emittance-oxidation performance. Promising candidate coatings are subjected to preliminary reentry simulation testing for 1 hour which includes about four 4 thermal cycles. The catalysis of specimens is evaluated from the temperature rise rate of the specimens on exposure to the test environment. Coatings that meet performance requirements for the research objective are tested for 5 hours. Post test analysis of coatings include spectral reflectance measurements and metallurgical examination including x-ray diffraction and scanning electron microscopy.

Accomplishment: A single titanium-aluminide alloy with three different coatings has been subjected to preliminary reentry simulation testing for 1 hour at a surface temperature of 1800°F. As shown on the figure, all three coatings reduced catalytic heating by 30 percent and increased the emittance to a favorable level (.8).

Significance: Minimum structural weight of NASP structures requires coatings that lower the vehicle surface temperature by re-radiation of energy (high emittance) and by reducing the catalytic heating resulting from gaseous recombination (low catalysis). Coating must also provide oxidation protection of the substrate materials which are susceptible to oxidation.

Future Plans: Post exposure specimens will be subjected to metallurgical analysis including SEM, XRD, and microprobe of the specimen surface and cross-section. Results of this evaluation will be input to NASP contractors for possible use in coating modification activities. Most outstanding candidate coatings will be tested for 5 hours at 1800°F in the LaRC HYMETS facility.

Figure 41(a).
EMITTANCE/CATALYSIS COATINGS IMPROVE PERFORMANCE OF TITANIUM–ALUMINIDE

Ti–14Al–19Nb–8Mo

**Before reentry test**

**After reentry test**

**Total normal emittance at 1800°F**

- Uncoated
- Pack aluminum
- MoSi₂-thick
- MoSi₂-2 med.

**Temperature rise rate F/sec**

- Uncoated
- Pack aluminum
- MoSi₂-thick
- MoSi₂-2 med.

Figure 41(b).
LIQUID INTERFACE DIFFUSION BONDING OF TITANIUM ALUMINIDES SHOWS PROMISE FOR ELEVATED TEMPERATURE APPLICATIONS TO 1700°F

R. Keith Bird and Eric K. Hoffman
Metallic Materials Branch
Ext. 2212 September 1987
RTOP 763-01-41

Research Objective: To develop advanced joining processes for fabricating Ti₆Al metal-matrix composite and RSR titanium honeycomb-core sandwich structure and demonstrate upper use temperatures through testing and analysis.

Approach: Conduct in-house studies using ingot metallurgy (IM) titanium aluminate model materials to develop joining processes suitable for fabricating Ti₆Al composite sandwich structure. Utilize candidate liquid interface diffusion (LID) bonding materials and parameters to produce simple Ti₃Al - Ti₆Al butt joints. Optimize process parameters to achieve highest joint strength with minimum added weight. Measure joint strength over a range of elevated temperatures and determine the factors controlling the strength achieved. Analytical techniques include energy dispersive x-ray analysis and microprobe analysis for the determination of the composition of the material at the joint and scanning electron microscopy to determine the microstructural effects of the LID bonding process on the parent metal.

Accomplishment: Ti₃Al butt joints were fabricated using two different LID bonding material compositions. The joints were evaluated at elevated temperatures up to 1700°F. The figure shows the joint strength results. At the higher test temperatures the pure copper LID bonding material exhibits higher joint strengths than the copper/silver mixture. The LID bonding processes examined to date show promise for application at elevated temperatures, but further optimization is required.

Significance: Minimum structural weight of NASP structures requires the use of honeycomb-core sandwich panels with thin-gage Ti₃Al face sheets. The core/facade sheet joint must exhibit adequate mechanical properties in the temperature regimes of interest. In addition, the joining process must result in minimum added weight to the structure.

Future Plans: Additional LID bonding material compositions will be utilized to fabricate Ti₃Al butt joints for elevated temperature evaluation. The optimum LID processes will be used to produce honeycomb-core sandwich panels with Ti₃Al face sheets. These panels will be subjected to mechanical property evaluation at elevated temperatures.

Figure 42(a).
LID BONDED TiAl BUTT JOINT STRENGTH VS. TEMPERATURE

Figure 42(b).
POLYMERIC MATERIALS BRANCH

FY 88 PLANS

- Synthesis of improved polymers
  - Thermoplastics (homo and copolymers)
  - Thermosets
  - Blends and IPNs
  - Low CTE

- Expand composite work
  - Resin scale-up
  - Better quality prepreg (powder coating)
  - Optimize composite fabrication processes
  - Innovative fabrication techniques

- Structure/property relationships
  - Polymer synthesis (CTE, modulus, etc.)
  - Composite constituent relationships
  - Fiber-matrix interface
  - Adhesives
FATIGUE AND FRACTURE BRANCH

FY 88 PLANS

- Metals and MMC
  - Continue short crack cooperative programs
  - Begin to address crack growth in cryotankage alloys
  - Start elevated temperature fatigue and fracture testing SiC/TiAl MMC

- Composites
  - Develop viscoelastic micromechanics analysis
  - Emphasize fatigue of tough composites
  - Begin fatigue and fracture analysis of multidirectional composites

- Computational methods
  - Continue development of multi-grid method
  - Continue cooperative effort with computational structural mechanics group

- Shuttle support
  - Complete O-ring testing

Figure 44.
- Verify durability of Space Station tube materials and thermal control coatings.
- Develop advanced composites for precision segmented reflectors.
- Assess lifetime of SOA carbon-carbon composites in NASP mission environments.
- Develop sealants for refurbishment/repair of carbon-carbon coatings.
- Initiate development of in-house CVI/CVD capability.
- Composites for aircraft/rotorcraft panels.
- Develop multiaxial, multilayer weaving technology for net shape panels.
- Develop resin transfer molding and pultrusion concepts for woven/braided preforms.
METALLIC MATERIALS BRANCH

FY 88 PLANS

- Advanced aluminum alloy technology
  - PM alloys for higher temperature airframe applications
  - PM and aluminum-lithium alloys for cryogenic tank applications
- Processing and joining
  - Fabrication, testing, and evaluation of curved cap compression panel
  - Explore SPF feasibility for cryogenic tank application
- High temperature TiₓAl and AMMC metallics for hypersonics and NASP
  - Continue growth of in-house processing and characterization
  - New emphasis on high temperature thin gage metal matrix composites
  - Development of deposition techniques for thin gage MMC
The research program of the Materials Division is presented as FY 87 accomplishments and FY 88 plans. The accomplishments for each Branch are highlighted and plans are outlined. Publications of the Division are included by Branch. This material will be useful in program coordination with other government organizations, universities, and industry in areas of mutual interest.