FINAL REPORT SUBMITTED TO:

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION (NASA)
Lewis Research Center

Report Title:
"Composites Research in Support of the NASP Institute for Composites (NIC)"
NCC3-218

Period Covered:
June 1, 1991 through August 31, 1994

Submitted by:
T. Michael Knasel
Director for Research
Ohio Aerospace Institute
22800 Cedar Point Road
Cleveland, OH 44142
216/962-3040

November 29, 1994
NASA GRANTEE SUBCONTRACTOR  
NEW TECHNOLOGY REPORT  

NASA requires each research grantee, research contractor and research subcontractor to report new technology to the NASA Technology Utilization Office. The required reports and corresponding schedules for research grantee subcontractors are as follows:

<table>
<thead>
<tr>
<th>Title of Report</th>
<th>Form Number</th>
<th>Timetable</th>
</tr>
</thead>
<tbody>
<tr>
<td>Individual Disclosure</td>
<td>NASA 666A</td>
<td>The grantee subcontractor discloses each discovery of new technology individually, at the time of its discovery.</td>
</tr>
<tr>
<td>Interim Report</td>
<td>LeRC-GSNTR</td>
<td>For multi-year grant subcontracts, the subcontractor summarizes the previous year's disclosures on an annual basis. The first Interim New Technology (NT) Report is due exactly 12 months from the effective date of the subcontract.</td>
</tr>
<tr>
<td>Final Report</td>
<td>LeRC-GSNTR</td>
<td>The grantee subcontractor submits a cumulative summary of all disclosed discoveries. This Final NT Report is submitted immediately following the subcontract's technical period of performance.</td>
</tr>
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</table>

**Subcontractor Name and Address:**  
Massachusetts Institute of Technology  
Department of Materials Science and Engineering  
77 Massachusetts Ave.  
Cambridge, MA 02139

**Report Submitted by:**  
R. M. Pelloux  
(617) 253 - 3314

**NASA Grant Title:**  
NASTP Institute for Composites (NIC)

**NASA Grant Number:**  
NCC 3-218

**NASA Grant Monitor:**  
OAI Mike Knasel

**Subcontract Completion Date:**  
8/31/94

**Today's Date:**  
9/22/94

New technology may be either reportable items or subject inventions.

A reportable item is any invention or discovery, whether or not patentable, that was conceived or first actually reduced to practice during the performance of the contract or subcontract. Large business contractors and subcontractors must disclose reportable items as they are discovered and submit a noncumulative list of these new technology items on an annual basis [ref: Interim NT Report] and a cumulative list at the completion of the contract (or subcontract) period [ref: Final NT Report].

A subject invention is any invention or discovery, which is or may be patentable, that was conceived or first actually reduced to practice during the performance of the contract or subcontract. Small business contractors and subcontractors must, at a minimum, disclose subject inventions as they are discovered and submit a cumulative list of these new technology items on an annual basis [ref: Interim NT Report] and at the completion of the contract (or subcontract) period [ref: Final NT Report].

Grantees, small business contractors and subcontractor are only required to disclose and report patentable items (subject inventions). We request, however, that small business contractors and subcontractors disclose both patentable and nonpatentable (reportable) items, both of which are automatically evaluated for publication as NASA Tech Briefs and considered for NASA Tech Brief awards.

PLEASE COMPLETE THE REVERSE SIDE OF THIS FORM AND MAIL TO THE FOLLOWING ADDRESS:

NASA LEWIS RESEARCH CENTER  
ATTN: KATHY KERRIGAN  
TECHNOLOGY UTILIZATION OFFICE; MAIL STOP 7-3  
CLEVELAND, OHIO 44135

LeRC-GSNTR  
7/01/93
INSTRUCTIONS

This form may be used when reporting inventions, discoveries, improvements or innovations to NASA. Use of this report form is optional; provided, however, that whatever report format is used contain the essential information requested therein.

In completing each section, use whatever detail deemed appropriate for a "full and complete disclosure," as required by the New Technology or Property Rights in Inventions clause. For further guidance as to what constitutes a satisfactory report, please refer to NHB 2170.3, Documentation Guidelines for New Technology Reporting.

Available additional documentation which provides a full, detailed description should be attached, as well as any additional explanatory sheets where necessary.

1. TITLE Elevated Temperature Tensile and Creep Deformation of a SiC Fiber-Reinforced Titanium Metal Matrix Composite

2. INNOVATOR(S) (Name and Social Security No.) *

   R. M. Pelloux – SS 534 42 3580
   Rita Thurston – SS 357 68 9567

3. EMPLOYER (Organization and division)

   Massachusetts Institute of Technology
   Dept. of Materials Science and Engineering

4. ADDRESS (Place of Performance)

   77 Massachusetts Ave.
   Cambridge, MA 02139

SECTION I – DESCRIPTION OF THE PROBLEM THAT MOTIVATED THE TECHNOLOGY DEVELOPMENT (Enter A. – General Description of Problem Objective; B. – Key or Unique Problem Characteristics; C. – Past History/Prior Techniques; D. – Limitations of Prior Techniques)

The main goal was to measure the elevated temperature tensile and creep properties of a SiC fiber-reinforced titanium metal matrix composite.
C. Test data results are given in attached report and in MS thesis.
September 30, 1994
Research Assistant: Rina J. Thurston
Professor R. M. Reif
Department of Materials Science and Engineering
Massachusetts Institute of Technology

and GE Aircraft Engines, Lynn, MA
Additional assistance provided by: Ishion Corporation, Canton, MA

Directorate, Wright-Patterson AFB, OH
of High Temperature Composites (HTC) at the Wright Laboratory Materials
Research sponsored by the National Institute for Mechanics and Life Prediction

TITANIUM METAL MATRIX COMPOSITE
DEFORMATION OF A SiC FIBER-REINFORCED
ELEVATED TEMPERATURE TENSILE AND CREEP
Relative Capabilities of Several Conventional Materials and Advanced Materials
Outline of Presentation

Research Objectives •
Material •
Experimental Results •
Monotonic Tensile Behavior and Strain Rate Sensitivity (1)
Creep Behavior (2)
Creep Tests with DCPD Technique (3)
Conclusions •
Recommendations for Future Work •
and life prediction techniques

- Generate mechanical property data for analytical models and design

- High temperature composites

- Evaluate test methodologies and equipment for evaluation of

- Including damage accumulation and failure mechanisms,

- Behavior of continuous fiber-reinforced metal matrix composites,

- Develop a comprehensive understanding of the tensile and creep

Research Objectives
SCS-9/Beta 21S Composite

Beta 21S: Ti-15Mo-2.7Nb-3Al-0.2Si  
(weight percent)

SCS-9: SiC fiber

Composite: 0.24 fiber volume fraction

Cross-Weaving Ribbon  
Woven Fiber Mats  
Rolled Foils  

Stack Foils and Fiber Mats  
Encapsulate, Degas, HIP Consolidate  
Consolidated Composite Panel
Cross Sections of SCS-9 Fiber

and Fiber-Matrix Interface
Histogram of SCS-9 Fiber Tensile Strength
Test Equipment

Tensile Behavior
NOTE: ALL DIMENSIONS IN INCHES

TO EXCEED 0.010
TAB MISMATCH NOT

1.000 ± 0.030
0.250 ± 0.100

0.400 ± 0.100

0.400 ± 0.010

6.000 ± 0.100

0.001, 0.01, 0.1 mm/mm/min.

- 482, 650, 815°C
- RT, 482, 650, 815°C

Strain Rate Sensitivity Tests

Constant Strain Rate Tests

Tensile Test Parameters and Specimen Geometry
Stress vs. Strain Curves

0°C, 21°C, 32°C, 54°C, 98°C
(90) SCS-9/Beta 21S Stress-Strain Curves

![Stress-Strain Curves Graph]

- 23°C
- 482°C
- 630°C
- 815°C
(0) SCS-9/Beta 21S RT and 815°C Stress-Strain Curves

---

**Graph Description:**
- **Axes:**
  - Y-axis: Stress (MPa)
  - X-axis: Strain (mm)
- **Lines:**
  - E = 110 GPa
  - E = 159 GPa
  - 25°C, 815°C
- **Legend:**
  - Random Fiber Texture
(0/90)_s SCS-9/Beta 21S RT and 815°C Stress-Strain Curves

- At 23°C, the stress-strain curve shows a gradual increase with strain, indicating a linear elastic behavior.
- At 815°C, the stress-strain curve shows a steeper increase, indicating a lower modulus of elasticity.
- At both temperatures, 90° fibers debond from the matrix followed closely by random fracture of 0° fibers, which is indicated by the curve's slope changes.

The graph illustrates the mechanical behavior of the composite material under different temperatures, highlighting the transition from a linear elastic to a less elastic behavior at high temperatures.

Key Points:
- Stress (MPa) vs. Strain (mm/mm)
- 23°C and 815°C curves
- Fiber behavior at different temperatures
(90). SCS-9/Beta 21S RT and 815°C Stress-Strain Curves

![Stress-Strain Curves Graph]

- **23°C**
  - Matrix Yielding
  - Fiber/Matrix Debonding
  - Fiber/Matrix Debonding Followed Closely by Matrix Yielding

- **815°C**

Stress (MPa) vs. Strain (mm/mm)
Temperature Dependence of the UTS of SCS-9/Beta 21S Composite Layups
Temperature Dependence of the Elastic Modulus of SCS-9/Beta 21S Composite Layups
Decrease in Proportional Limit With Temperature
Specific UTS of (0)₄ Composites and Two Nickel-base Alloys
Specific UTS of (0/90)ₙ Composites and Two Nickel-base Alloys

![Graph showing specific UTS vs temperature for different composites and alloys.](image)
Specific UTS of \((90)_4\) Composites and Two Nickel-base Alloys

![Graph showing specific UTS of (90)_4 composites and two nickel-base alloys as a function of temperature.](image-url)

- \((90)_4\) SCS-9/Beta 21S \(v_f = 0.24\)
- \((90)_4\) SCS-6/Ti-15-3 \(v_f = 0.33\)
- \((90)_4\) SCS-6/Ti-14-21 \(v_f = 0.35\)
- Inconel 718
- Hastelloy X
and Nickel-base Superalloys

Specific Elastic Modulus of (0)_4 Composites
Specific Elastic Modulus of (0/90)\textsubscript{s} Composites and Nickel-base Superalloys
and Nickel-base Superalloys
Specific Elastic Modulus of (90) Composite
Stress-Strain Curves Obtained from Tensile Tests Conducted at 650°C With Instantaneous Changes in Strain Rate
Creep Test Parameters and Specimen Geometry

Test Parameters

(0)_4

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Stress Range (MPa)</th>
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</thead>
<tbody>
<tr>
<td>650°C</td>
<td>552, 586, 655</td>
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<tr>
<td>815°C</td>
<td>276</td>
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</table>

(0/90)_3

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Stress Range (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>650°C</td>
<td>276, 345, 414</td>
</tr>
<tr>
<td>815°C</td>
<td>103</td>
</tr>
</tbody>
</table>

(90)_4

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Stress Range (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>650°C</td>
<td>34, 69, 138</td>
</tr>
<tr>
<td>815°C</td>
<td>21, 34</td>
</tr>
</tbody>
</table>

NOTE: ALL DIMENSIONS IN INCHES
Creep Curves for \((0)_4\) SCS-9/Beta 21S
Creep Curves for (0/90) SCS-9/Beta 21S

- 650°C/276 MPa (40 ksi)
- 650°C/345 MPa (50 ksi)
- 650°C/414 MPa (60 ksi)
- 815°C/103 MPa (15 ksi)

Time (hr.)

Creep Strain (%)
Creep Curves for (90º) SCS-9/Beta 21S
Total Strain for (0), SCS-9/Beta 21S

\[ \varepsilon_c = \frac{\sigma_c}{E_f \nu_f} \]

- **Graph Description**
  - **Y-axis**: Total Strain (%)
  - **X-axis**: Time (hr.)
  - **Legend**:
    - 650°C/552 MPa (80 ksi)
    - 650°C/552 MPa (80 ksi) Intact
    - 650°C/586 MPa (85 ksi)
    - 650°C/655 MPa (95 ksi)
    - 815°C/276 MPa (40 ksi)
  - **Data Points**: Intact Specimen
The image shows a graph titled "Highlight (0) SCS-9/Beta 215 Creep Curves." The graph plots creep strain (%) on the y-axis against time (h) on the x-axis. There are various data points indicated by different symbols, each representing different conditions or data sets. The graph includes a legend with symbols corresponding to these data points, but the specific details or conditions are not legible in the image provided.
Highlight (0/90), SCS-9/Beta 21S Creep Curves

![Creep Curves Graph](image-url)
Creep Rate Dependence on Applied Stress
Larson - Miller Plot
Test Equipment

Creep Tests with DCPD Technique

3-zone slip round test furnace
Double knife-edge alignment components
ATS wedge coupling grips with flat face inserts
ATS 2410 lever arm creep tester with automatic data collection

NOTE: ALL DIMENSIONS IN INCHES

- 0.0375 ± 0.001
- 0.00 ± 0.010
- 0.400 ± 0.001
- 0.175 ± 0.001
- 1.00 ± 0.010
- 0.750 ± 0.001
- 0.00 ± 0.010
- 6.000 ± 0.100
Assumptions:

\[ \frac{\partial}{\partial t} V(t) = (i) C (i) d(t) I = (i) A \]

Creep Tests with DCD Technique
Verifying Assumptions

650°C / No Load Tests
DCPD Curves for (0)_4 SCS-9/Beta 21S

650°C / 276 MPa
Normalized DCPD Curves for (0), SCS-9/Beta 21S

650°C / 276 MPa
Normalized DCPD Curves for (90)_4 SCS-9/Beta 21S

650°C / 21 MPa
Quantitative Correlation of DCPD Readings to Creep Strain

\[ \ln \left( \frac{V}{V_0} \right) = 2\ln \left( \frac{A_o}{A_t} \right) \]

\[ \varepsilon = \ln \left( \frac{A_o}{A_t} \right) = \frac{1}{2} \ln \left( \frac{V}{V_0} \right) \]

\[ \delta = \frac{\Delta L}{L} = \left( \frac{V}{V_0} \right)^{0.5} - 1 \times 100(\%) \]
Conclusions

• At 650°C, the UTS of (0)₄ and (0/90)₄ SCS-9/Beta 21S decreases by almost 50% from the room temperature values, indicating that operating temperatures should be less than 650°C to take advantage of the specific strength as well as the specific elastic modulus of these composite layups.

• The tensile properties of SiC fiber-reinforced titanium composites are dependent on the properties of the fiber reinforcement as well as the fiber volume fraction.

• Fracture mechanisms in (0)₄ SCS-9/Beta 21S are a combination of random fiber failure throughout the specimen and matrix microyielding around fractured fibers.

Failure of (90)₄ SCS-9/Beta 21S is dominated by the properties of the metal matrix.

Fracture mechanisms in (0/90)₄ SCS-9/Beta 21S are a combination of fiber-matrix debonding, random fiber fracture of the 0° fibers and matrix microyielding around debonded 90° fibers and fractured 0° fibers.
Conclusions

- The (0)„ and (0/90)s, SCS-9/Beta 21S composites are highly creep resistant when the applied stress on the 0° fibers is below the fracture strength of the fibers. At 650°C, the threshold stress levels of the 0° and (0/90)s, layups are approximately 552 MPa and 276 MPa, respectively. Below these stress levels, the creep rate approaches zero as the applied stress is transferred to the 0° fibers. Above these stress levels, the 0° fibers are loaded to fracture during transfer of the applied stress, which leads to high creep rates and a much shorter creep life.

- At 650°C and near-threshold stress levels, there is a small but steady accumulation of creep strain with time in the (0)„ and (0/90)s, composites. This is believed to be due to environmental degradation of the fiber and matrix near the specimen edges. Failure of all three composite layups in long-term creep tests in air is expected to initiate from the edge of the specimen due to environmental degradation.

- Fiber reinforcement parallel to the stress axis or at an angle less than 90° is necessary to enhance the tensile strength and creep resistance of the monolithic matrix material.
Conclusions

Deformation mechanisms differ significantly compared to monolithic materials due to the difference in creep changes in potential to creep deformation processes has to be approached in potential across the specimen face length. The lack of quantitatively releasing revealed that creep damage and deformation could be detected by the change in potential recordings of creep deformation of the three composite layups.

DC potential recordings of creep deformation of the three composite layups.

Strain rate sensitivity which increases with increasing temperature.

The (0/6) "SCS-9/β10A 21S" composites are practically insensitive to strain rate at 650°C and 815°C. The (90/0) composite exhibits significant strain rate during both tensile and creep tests.

The capacitive extensometer was capable of measuring the small strains in running constant load creep tests.

The servo-hydraulic test system is capable of instantaneously changing the
composites, including the effects of different creep loading cycles.

• Evaluate the creep recovery behavior of SiC fiber-reinforced titanium

• Grps.

Evaluate whether there is effective transfer of the applied stress from the matrix to the fibers in creep tests in which the fibers do not extend into the

• Effects and degree of environmental degradation.

Conduct creep rupture tests below threshold stress levels to determine the

Future Work