



AFRL-RX-WP-TP-2011-4234

**FREQUENCY AND HOLD-TIME EFFECTS ON
DURABILITY OF MELT-INFILTRATED SiC/SiC
(PREPRINT)**

R. John

Metals Branch

Metals, Ceramics, and NDE Division

G. Ojard and R. Miller

Pratt & Whitney

Y. Gowayed

Auburn University

G. Morscher

University of Akron

U. Santhosh and J. Ahmad

Research Applications, Inc.

JULY 2011

Approved for public release; distribution unlimited.

See additional restrictions described on inside pages

STINFO COPY

**AIR FORCE RESEARCH LABORATORY
MATERIALS AND MANUFACTURING DIRECTORATE
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7750
AIR FORCE MATERIEL COMMAND
UNITED STATES AIR FORCE**

REPORT DOCUMENTATION PAGE					Form Approved OMB No. 0704-0188	
The public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.						
1. REPORT DATE (DD-MM-YY) July 2011		2. REPORT TYPE Journal Article Preprint		3. DATES COVERED (From - To) 01 July 2011 – 01 July 2011		
4. TITLE AND SUBTITLE FREQUENCY AND HOLD-TIME EFFECTS ON DURABILITY OF MELT-INFILTRATED SiC/SiC (PREPRINT)				5a. CONTRACT NUMBER In-house		
				5b. GRANT NUMBER		
				5c. PROGRAM ELEMENT NUMBER 62102F		
6. AUTHOR(S) R. John (AFRL/RXLM) G. Ojard and R. Miller (Pratt & Whitney) Y. Gawayed (Auburn University) G. Morscher (University of Akron) U. Santhosh and J. Ahmad (Research Applications, Inc.)				5d. PROJECT NUMBER 4347		
				5e. TASK NUMBER 20		
				5f. WORK UNIT NUMBER LN101100		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Metals Branch (AFRL/RXLM) Metals, Ceramics, and NDE Division Air Force Research Laboratory, Materials and Manufacturing Directorate Wright-Patterson Air Force Base, OH 45433-7750 Air Force Materiel Command, United States Air Force				8. PERFORMING ORGANIZATION REPORT NUMBER AFRL-RX-WP-TP-2011-4234		
Pratt & Whitney East Hartford, CT						
Auburn University Auburn, AL						
University of Akron Akron, OH						
Research Applications, Inc. 11772 Sorrento Valley Road, Suite 260 San Diego, CA 92121-1085						
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Air Force Research Laboratory Materials and Manufacturing Directorate Wright-Patterson Air Force Base, OH 45433-7750 Air Force Materiel Command United States Air Force				10. SPONSORING/MONITORING AGENCY ACRONYM(S) AFRL/RXLM		
				11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S) AFRL-RX-WP-TP-2011-4234		
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited.						
13. SUPPLEMENTARY NOTES PAO Case Number: 88ABW 2010-6652; Clearance Date: 21 Dec 2010. Paper presented at 35th International Conference and Exposition on Advanced Ceramics and Composites, Daytona Beach, Daytona, Florida, January 2011 and submitted as a journal article to the <i>Journal: Composites Science & Technology</i> .						
14. ABSTRACT With the growing interest in ceramic matrix composites for multiple applications, the response of the material to service conditions needs to be understood. A range of durability assessment was undertaken on Melt Infiltrated (MI) SiC/SiC Ceramic Matrix Composite. MI SiC/SiC was tested under 30 Hz fatigue, 1 Hz fatigue, dwell fatigue (2 hour hold cycle) and creep loading. The applied stresses ranged from the micro-cracking initiation point to well above the saturation stress of the material. Test temperatures included room, 815 °C and 1204 °C. The effects of fatigue loading frequency and dwell (hold) time on the durability of the composite are discussed.						
15. SUBJECT TERMS ceramic matrix composites (CMCs), melt infiltrated (MI) SiC/SiC, dwell fatigue, creep loading						
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT: SAR	18. NUMBER OF PAGES 16	19a. NAME OF RESPONSIBLE PERSON (Monitor) Reji John 19b. TELEPHONE NUMBER (Include Area Code) N/A	
a. REPORT Unclassified	b. ABSTRACT Unclassified	c. THIS PAGE Unclassified				

FREQUENCY AND HOLD-TIME EFFECTS ON DURABILITY OF MELT-INFILTRATED SiC/SiC

Ojard, G.², Gawayed, Y.³, Morscher, G.⁴, Santhosh, U.^{5,6}, Ahmad, J.⁵, Miller, R.² and John, R.¹

¹ Air Force Research Laboratory, AFRL/RXLM, Wright-Patterson AFB, OH

² Pratt & Whitney, East Hartford, CT

³ Auburn University, Auburn, AL

⁴ University of Akron, Akron, OH

⁵ Research Applications, Inc., San Diego, CA

⁶ Structural Analytics, Inc., Carlsbad, CA

ABSTRACT

With the growing interest in ceramic matrix composites for multiple applications, the response of the material to service conditions needs to be understood. A range of durability assessment was undertaken on Melt Infiltrated (MI) SiC/SiC Ceramic Matrix Composite. MI SiC/SiC was tested under 30 Hz fatigue, 1 Hz fatigue, dwell fatigue (2 hour hold cycle) and creep loading. The applied stresses ranged from the micro-cracking initiation point to well above the saturation stress of the material. Test temperatures included room, 815°C and 1204°C. The effects of fatigue loading frequency and dwell (hold) time on the durability of the composite are discussed.

INTRODUCTION

For any material to be considered for insertion into real applications, the material needs to be fully characterized. This is especially true for Ceramic Matrix Composites (CMC) that are being considered for applications in the extreme environments of gas turbine engines. The full extent of the material performance throughout a range of temperatures and durability should be explored. This has been shown by the authors by looking at long term “creep” type testing and residual properties and fatigue testing [1-4]. This is usually considered to be important for long term applications such as industrial gas turbines [5-6]. This work expands the past assessment of this material where durability and the time the material spent at temperature was the focus of the investigation [4].

The MI SiC/SiC system was chosen as the material for this effort based on the past work of the authors as well as the vast data that exists on the material system [7-8]. This material was developed during the Enabling Propulsion Materials Program led by NASA-Glenn Research Center.

PROCEDURE

Material Description

The Melt Infiltrated SiC/SiC CMC system chosen for this study was initially developed under the Enabling Propulsion Materials Program (EPM) and is still under further refinement at NASA-Glenn Research Center (GRC) [7-8]. The material description and microstructure has been documented earlier by the authors [1-4].

Mechanical and Durability Testing

Testing for this effort ranged from standard tensile testing per ASTM C1275 (room temperature) and ASTM C1349 (elevated temperature) to durability testing. The creep and dwell fatigue testing was performed per ASTM C1337. The dwell fatigue testing conditions included 2 hour hold period followed by unloading of the sample to a load ratio (R) of 0.05. The fatigue testing was done per ASTM C1360 under load controlled conditions at 1 Hz or 30 Hz using a sine wave with R of 0.05, 0.5 and 0.8. During the creep testing, either total strain or creep strain was recorded. During the dwell fatigue and fatigue testing, total strain was recorded.

RESULTS

Tensile Tests

The tensile behavior of MI SiC/SiC has been reported previously [1,4]. The results are summarized in Figure 1 showing the initiation of micro-cracking stress, proportional limit and crack saturation stress [4]. As can be seen in Figure 1, the micro-cracking, proportional limit and crack saturation stress are not sensitive to temperature. In contrast, the ultimate tensile strength does decrease for the testing done at 1204°C.

Creep/Dwell Fatigue Testing

Creep and dwell fatigue testing (2 hour hold, R = 0.05) were performed over a range of times, temperature and stresses. For the testing done, there were a varying range of run-out times set for the testing. When the time for the testing was complete, the test was stopped and the sample removed. The results of this testing is summarized in Figure 2. For this series of testing, the only failures observed were for the creep tests performed at 1204°C. These failures corresponded to either a high stress level or long test duration.

Fatigue Testing – 1 Hz and 30 Hz

In addition to the above work, a series of 1 Hz and 30 Hz fatigue tests were done at 1204°C at R of 0.05. The results of this testing are shown in Figure 3. The run-out (or discontinued) condition for the 1 Hz testing was set at 400,000 cycles while the 30 Hz testing run-out was achieved at a value greater than 40,000,000 cycles. The effect of R on fatigue behavior at 30 Hz and at 1204°C is shown in Figure 4.

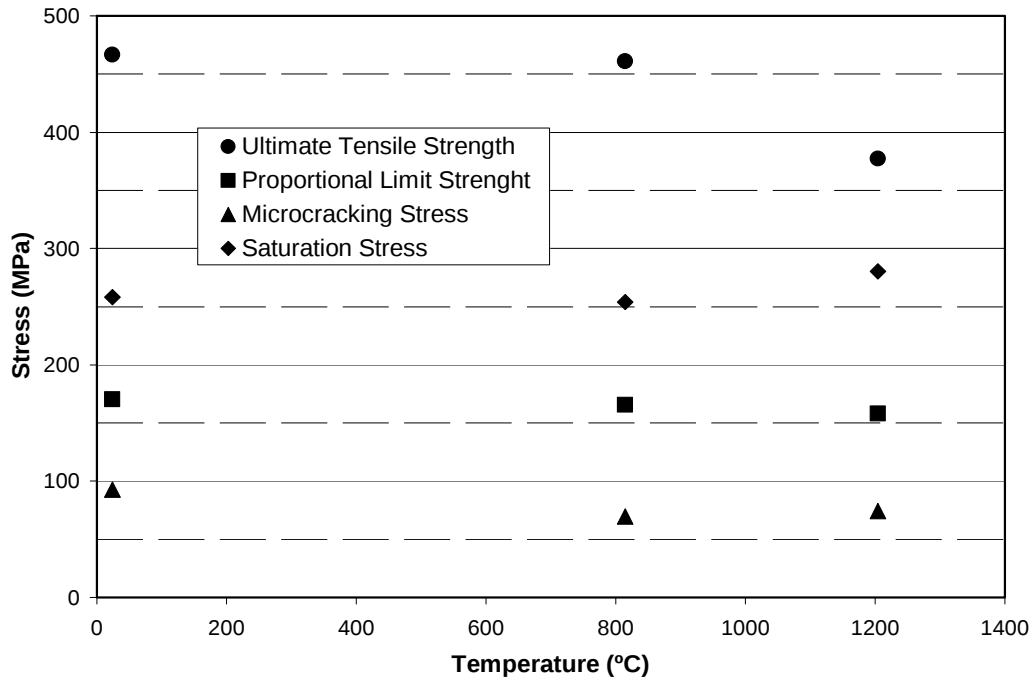


Figure 1. Key Stress Points from Stress-Strain Curves for MI SiC/SiC versus Temperature

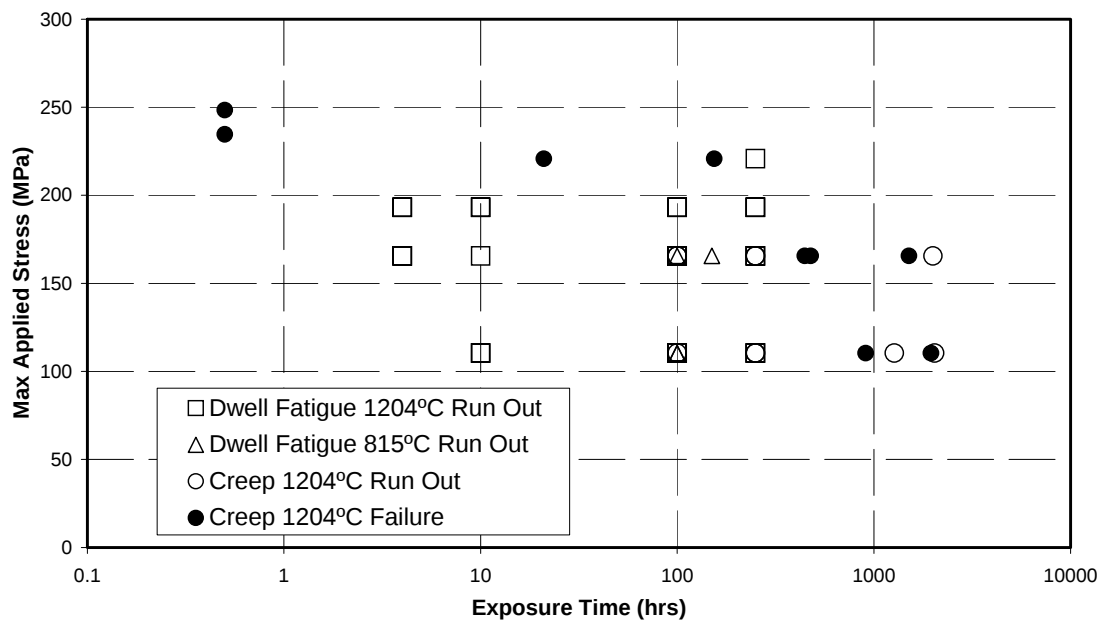


Figure 2. Summary of Creep and Dwell Fatigue Testing at 1204°C

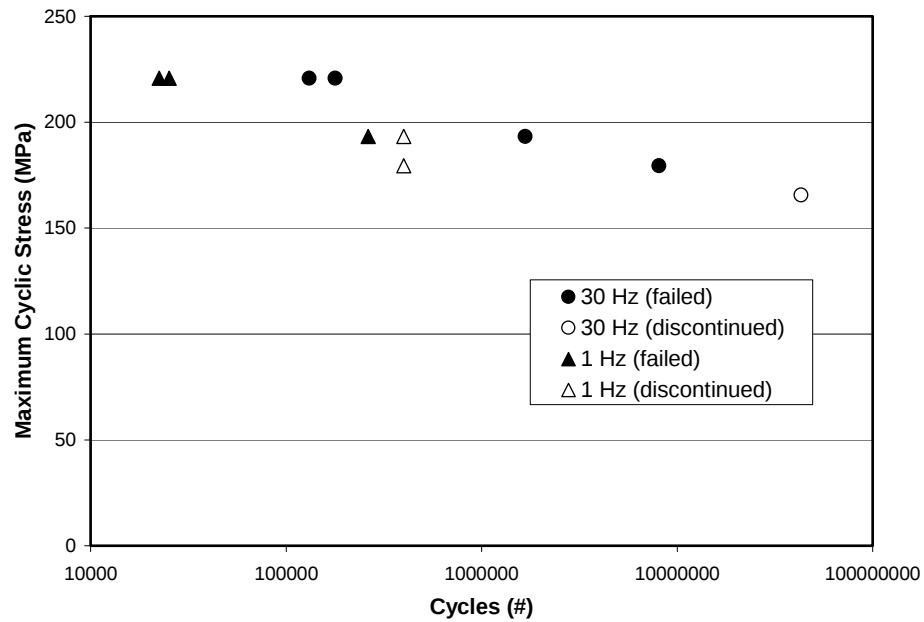


Figure 3. Summary of Fatigue Testing at 1204°C (R = 0.05)

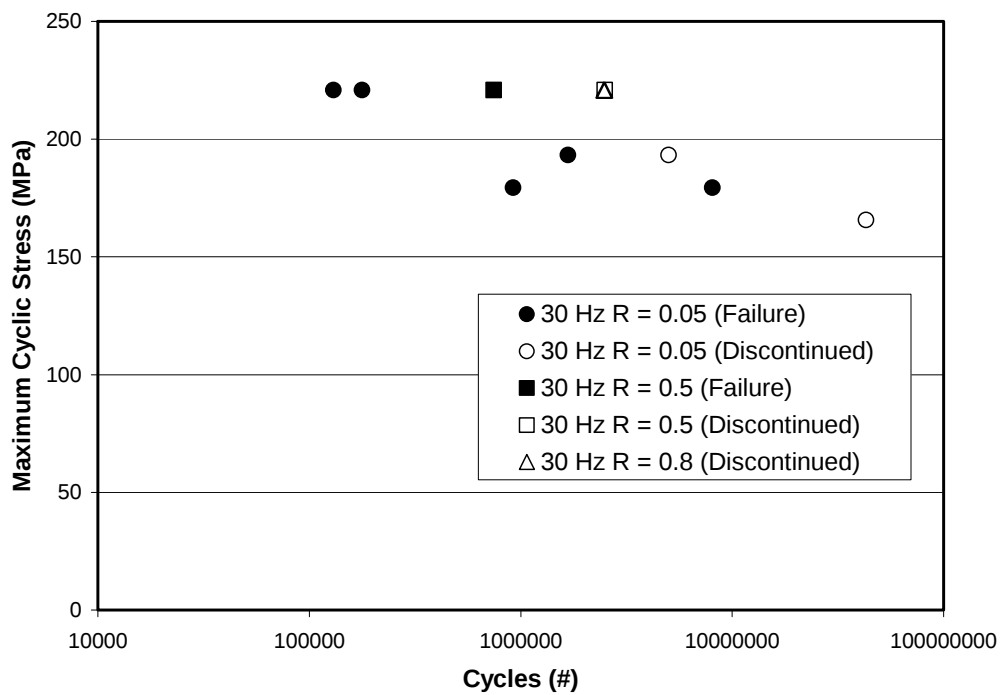


Figure 4. Summary of Fatigue Testing at 1204°C with Differing Load Ratios (R).

Residual Tensile Tests

From the series of creep and dwell fatigue (2 hour hold) testing, there were multiple samples where the testing was stopped at several different exposure temperatures and times. From these samples, a series of residual tensile tests were conducted. Since the evolved strain was recorded during testing as well as the strain from the residual tensile test was known, the data was plotted as the evolved strain during exposure versus the retained strain seen in the tensile test. This is shown in Figure 5. This work has been presented elsewhere by the authors [9]. The data falls near a constant 0.5% strain line as shown in Figure 5. Past published work by the authors has shown that the as-received strain capability is 0.5% (tensile) and failed creep tests achieve near 0.5% strain [9]. Figure 5 shows that the total strain accumulated either by creep, tensile or combination of both tests sums to 0.5% giving guidance on how to use the material.

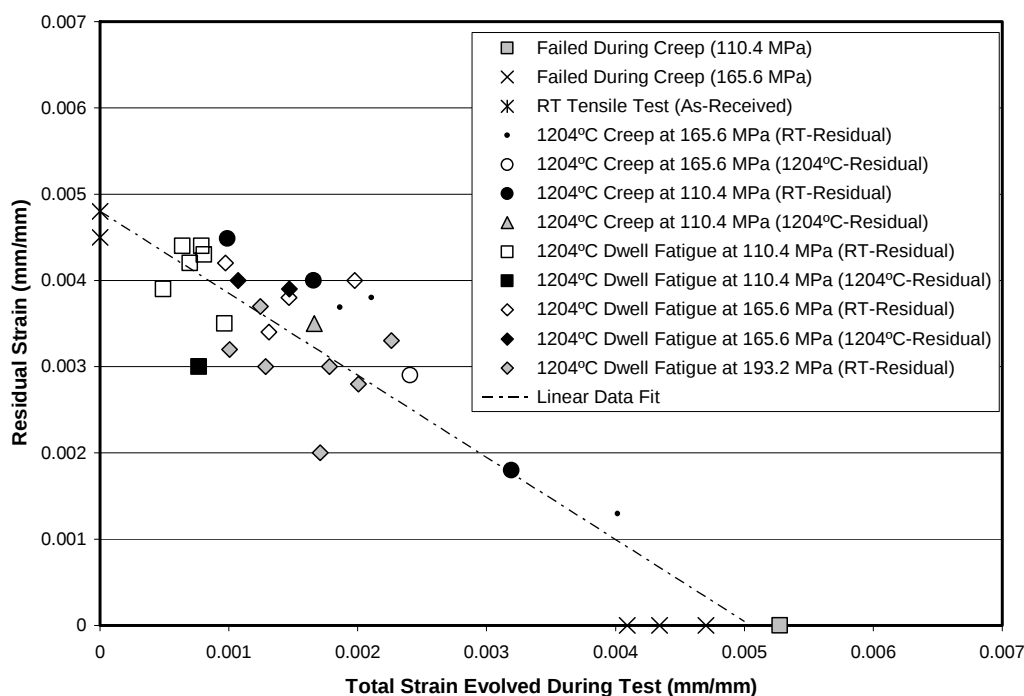


Figure 5. Residual Tensile Results plotted as Evolved Strain vs. Residual Strain

DISCUSSION & CONCLUSIONS

As noted above, a wide range of durability testing was performed across a variety of tests. This raises the question about which is the best way to compare the data. Some researchers looked at the cycles to failure when looking at frequency effects [10,11]. Other researches looked at the loading rate between tests [12]. Since the range of frequencies tested only varied by an order of magnitude, it was decided to look at time to failure for comparison and not cycles to failure. This type of analysis excludes samples that were discontinued early in the analysis as there is no way to project where they would fail in a direct comparison. This is done for all the creep and fatigue tests (dwell fatigue, 1 Hz and 30 Hz) where R was 0.05 as shown in Figure 6. Note that there were no dwell fatigue failures in this testing series. Figure 6 also includes the

key points from the stress-strain curve showing that most of the testing was done between the proportional limit and the saturation stress for the tests run at 1204°C.

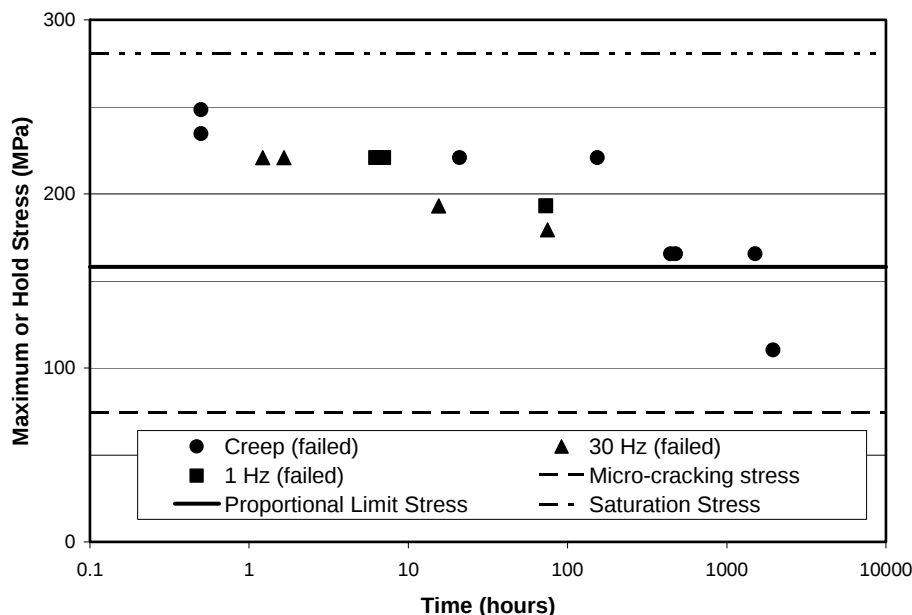


Figure 6. Failed Creep and Fatigue Data Points (1204°C)

A quick review of Figure 6 can lead to the conclusion that the failure is dependent on the time at temperature [4]. This is easily the case when looking at creep tests closer to the micro-cracking stress (long term) and saturation stress (short term). With the current work on R of 0.5 and 0.8, this conclusion can be challenged. This can be seen in Figure 4 where the 30 Hz data points are plotted with differing R ratios. As the R ratio is increased (meaning less cycling between stress levels), longer lifetimes are achieved. For the 220.8 MPa stress level experiments, there were no failures for the R = 0.8 tests (as shown in Figure 4). As R is being increased, the test is becoming more and more like a hold test (creep or dwell fatigue (2 hour hold)).

With the limited 30 Hz testing that was done with different R ratios and no R ratio testing done at 1 Hz, it would appear that analysis would be limited and no additional insight could be achieved. As an alternative, residual tensile testing done on these samples can be used. While the curve in Figure 5 shows that the overall breadth of the data falls on an almost constant strain line, the data does segregate when comparing the dwell fatigue (2 hour hold) versus the creep data. As can be seen in Figure 7, the dwell fatigue data is mostly below the constant strain line while the creep data falls above. This is reinforced when looking at a curve fit of the data (See Figure 8) where the creep data has the appearance of a constant line parallel to the previous lines shown in Figures 5 and 7. The dwell fatigue (2 hour hold) data has a much lower slope and appears to be greatly affected by the load un-load during the test.

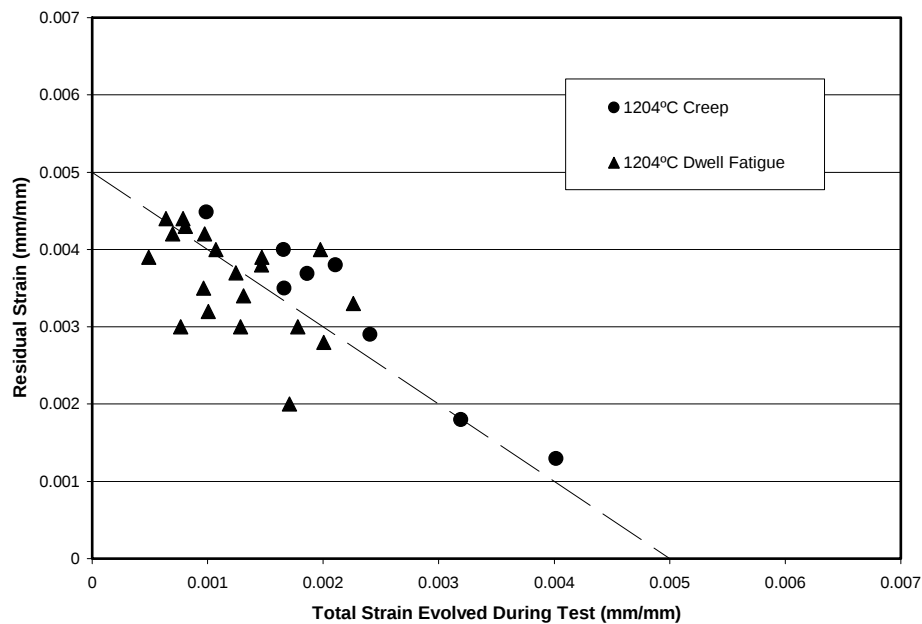


Figure 7. 1204°C Creep and Dwell Fatigue Data from Figure 5

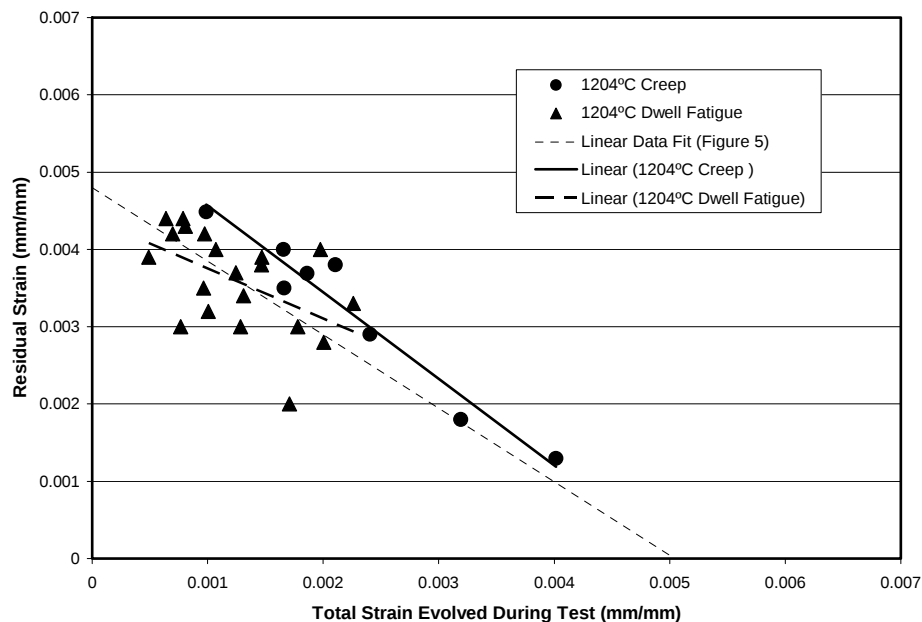


Figure 8. Curve Fits of Data from Figure 7

The results shown in Figures 7 and 8 shows that periodic load un-load cycles degrades the material performance, as indicated by the reduction in failure strain of the dwell fatigue specimens compared against the creep samples (with no unloading occurring). This is consistent with the 30 Hz data shown in Figure 4 where having a higher R is not as detrimental as the lower R on the material performance. The higher R ratio results in less stress cycling meaning that the

cracks formed during the test are not being opened to as great of an extent in the low R ratio baseline tests. This allows any oxide formed in the crack to remain allowing some protection. This is then consistent with the R ratio of 0.8 tests that did not fail during the test.

This study has shown that there is a load ratio (R) effect on the fatigue behavior and no clear evidence of a frequency effect in MI SiC/SiC composites. We suggest that this work be followed with additional testing looking further into possible frequency effects in greater detail for this class of material.

ACKNOWLEDGMENTS

The Air Force Research Laboratory, Materials and Manufacturing Directorate (AFRL/RX) sponsored this work under contracts F33615-01-C-5234 and F33615-03-D-2354-D04.

REFERENCES

1. Y. Goward, G. Ojard, R. Miller, U. Santhosh, J. Ahmad, and R. John, "Correlation of Elastic Properties of Melt Infiltrated SiC/SiC Composites with In-Situ Properties of Constituent Phases", *Composites Science and Technology* 70 (2010) 435–441.
2. Morscher, G.N., Ojard, G., Miller, R., Goward, Y., Santhosh, U., Ahmad, J., and John, R., "Tensile Creep and Fatigue of Sylramic-iBN Melt-Infiltrated SiC Matrix Composites: Retained Properties, Damage Development, and Failure Mechanisms", *Composites Science and Technology* 68 (2008) 3305–3313.
3. Y. Goward, G. Ojard, J. Chen, G. Morscher, R. Miller, U. Santhosh, J. Ahmad and R. John, "A Study of the Time-Dependent Response of Sylramic-iBN Melt-Infiltrated SiC Matrix Composites Using Dwell-Fatigue Experiments", Submitted to the *Journal of Composites Science and Technology*
4. Ojard, G., Goward, Y., Morscher, G., Santhosh, U., Ahmad, J., Miller, R. and John, R., "Creep And Fatigue Behavior Of MI SiC/SiC Composites At Temperature", published in *Ceramic Engineering and Science Proceedings*, 2009.
5. Brewer, D., Ojard, G. and Gibler, M., "Ceramic Matrix Composite Combustor Liner Rig Test", ASME Turbo Expo 2000, Munich, Germany, May 8-11, 2000, ASME Paper 2000-GT-670.
6. Wedell, James K. and Ahluwalia, K.S., "Development of CVI SiC/SiC CFCCs for Industrial Applications" 39th International SAMPE Symposium April 11- 14, 1994, Anaheim California, Volume 2, pg. 2326.
7. Calomino, A., NASA-Glenn Research Center, personal communication.
8. J.A. DiCarlo, H-M. Yun, G.N. Morscher, and R.T. Bhatt, "SiC/SiC Composites for 1200°C and Above" *Handbook of Ceramic Composites*, Chapter 4; pp. 77-98 (Kluwer Academic; NY, NY: 2005)
9. Ojard, G., Calomino, A., Morscher, G., Goward, Y., Santhosh, U., Ahmad J., Miller, R. and John, R., "Post Creep/Dwell Fatigue Testing of MI SiC/SiC Composites", *American Ceramic Society, Ceramic Engineering and Science Proceedings*, pp. 135-143, 2008.
10. Mall, S. and Engesser, J.M., "Effects of Frequency on Fatigue Behavior of CVI C/SiC at Elevated Temperature", *Composites Science and Technology* 66 (2006) 863–874.

11. Staehler, J.M., Mall, S. and Zawada, L.P., "Frequency Dependence of High-Cycle Fatigue Behavior of CVI C/SiC at Room Temperature", *Composites Science and Technology* 63 (2003) 2121–2131.
12. Choi, S.R. and Gyekenyesi, J.P., "Load-rate Dependency of Ultimate Tensile Strength in Ceramic Matrix Composites at Elevated Temperatures", *International Journal of Fatigue* 27 (2005) 503–510.