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# Thermomechanical Fatigue Behavior of SiC/Ti-24Al-11Nb in Air and Argon Environments

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# THERMOMECHANICAL FATIGUE BEHAVIOR OF SiC/Ti-24Al-11Nb IN AIR AND ARGON ENVIRONMENTS

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## SUMMARY

A series of tension-tension, load-controlled thermomechanical fatigue tests were conducted on a titanium aluminide composite in both laboratory air and a flowing argon environment. Results from these tests show that the environment plays an increasingly important role as applied stress levels are decreased. Differences in damage mechanisms between the two environments were observed which correspond to observed variations in TMF lives.

## INTRODUCTION

Fiber-reinforced titanium aluminides have been identified as an alternative to conventional super-alloys for applications that require a low density material that maintains its structural integrity at elevated temperatures. However, there is some concern in using composites for high temperature applications where thermomechanical loadings occur. Mismatch of physical properties between the fiber and matrix of composite materials can lead to failure mechanisms that do not occur in monolithic materials. It is believed that differences in creep resistance, coefficient of thermal expansion (CTE), and tensile properties between the fiber and matrix will be detrimental to the thermomechanical fatigue (TMF) life. In addition, oxidation may play an important role in the TMF life of titanium alloy matrix composites, since proposed composite service-temperatures are higher than the maximum service-temperature of the unreinforced titanium matrix alloys. Also, the composite's weak fiber/matrix interfaces are more vulnerable to oxidation than the matrix and can act as a conduit making the internal regions of the composite's matrix more susceptible to oxidation.

## Background

Several recent studies have shown how the environment influences the mechanical properties of several metal matrix composites and their Ti-alloy matrices (refs. 1 to 6). Mahulikar and coworkers (ref. 1) compared the effects of prior heat treatment in air and in vacuum on the room temperature tensile and fatigue crack propagation behavior of the B<sub>4</sub>C-B/Ti-6Al-4V (atomic percent) composite. They concluded that these properties were adversely affected by the heat treatment in air because the fiber/matrix interface degraded; heat treatments in vacuum had little effect on these properties.

Russ (ref. 2) investigated how prior thermal cycling in air affects the tensile properties of SiC/Ti-24Al-11Nb (atomic percent). He found significant tensile strength degradation after a small number of thermal cycles. However, Brindley et al. (ref. 3) conducted a study similar to Russ', but with the thermal cycling performed in vacuum. This resulted in little degradation of tensile properties for specimens subjected to less than 10 000 thermal cycles. Specimens that experienced more than 10 000 thermal cycles had reduced tensile strength. Brindley et al. speculated that extensive surface matrix

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cracks normal to the loading direction and degradation of the weak interface due to the CTE mismatch caused this decrease.

Brindley and Bartolotta (ref. 4) observed longer isothermal low cycle fatigue (LCF) lives for tests conducted on SiC/Ti-24Al-11Nb in an inert environment compared to tests in air at temperatures of 425 and 815 °C. LCF lives were 10 times longer in the inert environment than in air for the 425 °C tests, which suggests that even at a relatively low temperature, environmental attack plays an important role in high temperature fatigue behavior. Gayda et al. (ref. 5) also showed that environment has a large effect on the isothermal and nonisothermal fatigue response of a SiC/Ti-15V-3Al-3Cr-3Sn (weight percent) composite. Results of these investigations suggest that the degradation of high temperature mechanical properties in Ti-matrix composites is due to environmental attack of either the matrix or the fiber/matrix interface.

In this study, the fatigue resistance of SiC/Ti-24Al-11Nb under thermomechanical fatigue loadings in both air and inert environments were investigated. Results from a series of both in-phase and out-of-phase TMF tests conducted in laboratory air using a temperature range of 215 to 815 °C are presented. TMF test results of a smaller temperature range of 425 to 815 °C are also presented along with companion inert environment test results.

## EXPERIMENTAL DETAILS

The material used in this study was a SiC/Ti-24Al-11Nb composite with a fiber volume,  $V_f$ , ranging between 22 to 28 vol % (fig. 1). The SiC/Ti-24Al-11Nb composite was fabricated by means of a powder cloth technique (ref. 7) that ensures full consolidation of the matrix, complete bonding between matrix and fibers, and low oxygen content in the composite. The fibers used were 140- $\mu$ m-diameter, double carbon-coated, SCS-6 SiC fibers. The matrix material, Ti-24Al-11Nb, was obtained as prealloyed powder. As shown in figure 1, the matrix of the consolidated composite was comprised of equiaxed  $\alpha_2$  (Ti<sub>3</sub>Al) that is partially surrounded by disordered  $\beta$  phase that is relatively more ductile than the  $\alpha_2$ . The fiber/matrix interface consists of an outer  $\beta$ -depleted zone and an inner fiber/matrix reaction zone that surrounds the C-rich coating of the fiber.

Specimens were machined by wire electrodischarge machining (EDM) from several 150- by 50-mm plates that had a [0]<sub>g</sub> layup and a nominal thickness of 2.4 mm. After EDM, specimen surfaces were polished with 180-grit SiC paper to remove a 10- $\mu$ m cladding reaction with the Mo release sheets used during fabrication and to remove damage due to wire EDM. The specimen design used in this study incorporates a reduced parallel gage section to facilitate close control of the temperature profile. A large fillet radius (61 mm) was found to be necessary to minimize the number of failures occurring at or near the radii. Tabs were epoxied onto the specimens to give an overall thickness of 6 mm over the gripped section. The intent here was to closely match the requirements of the precisely aligned grips, thereby minimizing possible bending induced by the grips. The tabs featured a tapered end to help distribute through-thickness stresses resulting from high clamping forces.

The TMF tests were conducted under computer control on a closed-loop, servohydraulic test system equipped with water-cooled hydraulic grips. Specimens were directly heated by 10-kW induction heaters. Temperature was measured by three thermocouples spot welded along the test section of the specimen. A standard 12.7-mm gage length axial extensometer mounted on the edge of the specimen was used for strain measurement. All environmental tests were performed in a specially designed environmental chamber capable of providing either a 10<sup>-6</sup> torr vacuum or a Ti-gettered argon atmosphere (fig. 2).



For the air tests, the specimens were cooled by forced air. For the argon tests, specimens were cooled by argon flowing through the chamber at a rate of 15 l/min. Dynamic temperature profiles throughout all tests were maintained within  $\pm 8$  °C over the 12.7-mm gage section.

## EXPERIMENTAL PROCEDURE

Initial test procedures were similar for all experiments with the exception of the flowing argon tests. For these tests, the following chamber preparation procedure was followed before specimen heating:

- (1) The chamber was pumped down to  $10^{-4}$  torr.
- (2) It was back-filled twice with argon.
- (3) It was again pumped down to  $10^{-4}$  torr.
- (4) Finally, the argon was allowed to flow at 15 l/min.

For all tests, prior to the start of TMF loading, each specimen was thermally cycled for at least five times under zero load to thermally stabilize it and to measure the "thermal strain-temperature" response for later data reduction.

In-phase and out-of-phase tests (fig. 3) were first conducted in air on SiC/Ti-24Al-11Nb using a temperature range of 215 to 815 °C to evaluate the overall TMF resistance of this composite system. Additional comparison out-of-phase tests were conducted in air and flowing argon to study the environmental influence on the TMF behavior of SiC/Ti-24Al-11Nb. The temperature range of 425 to 815 °C was used because of cooling-rate limitations of the flowing argon environment.

All experiments were conducted under load-control with a load ratio,  $R_P$  ( $P_{\min}/P_{\max}$ ), of zero. Control wave forms for both mechanical loads and temperature were triangular with a constant cycle period of 3 min for each test. Failure was defined as complete specimen fracture.

## RESULTS AND DISCUSSION

### Fatigue Behavior in Air (215 to 815 °C)

The overall TMF resistance of SiC/Ti-24Al-11Nb in an air environment is summarized in table I and presented in figure 4. In this figure, TMF results of 215 to 815 °C in-phase and out-of-phase tests are plotted along with 425 and 815 °C isothermal LCF life lines. As seen in figure 4, the TMF lives were significantly lower than isothermal LCF lives. Similar trends were observed in previous investigations for SiC/Ti-alloys (refs. 8 and 9) with one exception. For the present study, it was observed that with increasing maximum applied stress levels, the 815 °C isothermal fatigue life line tends to converge with the out-of-phase TMF data.

As for out-of-phase and in-phase TMF life comparisons, the same trends are observed for SiC/Ti-24Al-11Nb as were seen by Castelli et al. for SiC/Ti-15V-3Al-3Cr-3Sn (ref. 8). The slope of the 215 to 815 °C in-phase life line is more shallow than the 215 to 815 °C out-of-phase line. The lines intersect at a stress level of 450 MPa. At maximum stresses above this point, in-phase TMF loading is more damaging, and at stresses below this point, the out-of-phase condition is more detrimental.

In figure 5, typical fracture surfaces of out-of-phase and in-phase specimens are shown. The in-phase SiC/Ti-24Al-11Nb specimens exhibited little or no matrix fatigue cracking with the fracture surface dominated by fiber pullout and ductile matrix failure similar to a tensile failure. The fibers in the pullout areas for the in-phase specimens were quite long, suggesting that the fiber-matrix interfaces were

degraded. These observations were not surprising, since specimens during in-phase TMF cycling are subjected to peak loads when the temperature is at its maximum (fig. 3). In this case, the matrix tends to shed its load (via stress relaxation or plastic flow) onto neighboring fibers, increasing the stress levels experienced by these fibers (ref. 11). Thus, it is consistent to find that in-phase TMF damage is dominated by the fiber strength.

Out-of-phase specimens had matrix fatigue cracks throughout the test section, typically initiating at the surface, with a few initiating at the fiber-matrix interface (fig. 6). An overall view of the fracture surfaces of the out-of-phase specimens revealed a ring of matrix fatigue cracks around the outer edge with a region of ductile matrix failure in the center. The amount of fiber pullout was limited, and pullout lengths were shorter than those in the in-phase specimens. Due to the phasing between load and temperature histories (fig. 3), the matrix during out-of-phase TMF experiences larger stress and strain ranges, making it a more conducive condition for matrix fatigue cracking. During out-of-phase tests, the matrix is subjected to higher tensile stresses at minimum temperatures than during in-phase TMF (ref. 11). This is due to both temperature-load phasing and the CTE mismatch between fiber and matrix (ref. 11). At the lower temperature portion of an out-of-phase cycle, the matrix wants to contract but the fiber limits this shortening since it has the lower CTE and a higher stiffness. This low temperature tensile stress caused by the CTE mismatch combines with the externally applied load resulting in the development of even higher tensile matrix stresses. The observed differences in TMF failure mechanisms between out-of-phase and in-phase conditions along with similar observations previously reported for SiC/Ti-15V-3Al-3Cr-3Sn (ref. 8) confirm this hypothesis.

#### Air Versus Argon Fatigue Behavior (425 to 815 °C)

Results from the 425 to 815 °C TMF tests are summarized in table II and presented in figure 7. In this figure, TMF lives of tests conducted in laboratory air are represented by open symbols and closed symbols denote the argon test results. Life lines for the 215 to 815 °C TMF tests (air environment) are also shown for comparison purposes. Comparing only the air tests in figure 7, the 425 to 815 °C TMF tests (with one exception) tend to exhibit longer lives than the 215 to 815 °C tests. These differences in life tend to increase as maximum applied stress levels are decreased. The stress level where the out-of-phase and in-phase 425 to 815 °C TMF life lines intersect followed the same trend as the 215 to 815 °C data and increased to approximately 525 MPa. The increase in TMF resistance with the decrease in temperature range (i.e., in this case 215 to 815 °C to 425 to 815 °C) was expected, since a decrease in temperature range is associated with decreases in thermal mismatch stresses and strains. However, note that as applied mechanical stress levels are increased, the out-of-phase TMF lives tend to converge, suggesting that the mechanical stresses are dominating over the thermal stresses.

Four sets of out-of-phase TMF tests were conducted in both laboratory air and flowing argon environments. Stress levels for these tests ranged between 827 and 414 MPa. Note that all of these tests failed with the exception of the 414 MPa flowing argon test which was interrupted after 25 140 cycles (52 days). The role of environment on the out-of-phase TMF lives of SiC/Ti-24Al-11Nb is best illustrated in figure 8. Here, it is shown that for a high maximum applied stress the difference in TMF life is small (i.e., within a factor of 2 in life). On the contrary, the difference in life for the smaller applied stresses is significant, with the inert environment TMF lives an order of magnitude longer than those conducted in air for the same test conditions. These observations suggest that as the applied stresses increase, the mechanical load effects tend to dominate the out-of-phase TMF damage behavior over the environmental effects.

A comparison of the typical mechanical maximum strain response for out-of-phase TMF behavior of SiC/Ti-24Al-11Nb in air and argon is shown in figure 9. For the high-stress tests, the maximum



mechanical strain response is similar in both air and argon. Note that the maximum strain increases (ratcheting) for both test environments and that the rate and extent of this ratcheting are the same. The low-stress tests for both environments tend to have similar mechanical strain response until the air test fails. At that point, the maximum mechanical strain for the argon tests starts to increase. This ratcheting implies that the specimen's compliance changes dramatically as additional damage accumulates (i.e., crack propagation, fiber debonding, fiber fracturing, or any combination of these mechanisms). The full understanding of this phenomena will require additional research.

In figure 10, the fracture surfaces of out-of-phase specimens tested in air and argon in the high-stress, low-life regime are compared. Just as the mechanical and life response were similar, the fracture surfaces have comparable features. In both cases, little matrix fatigue was observed, with the majority of the fracture surface having features characteristic of tensile overload. The fact that the damage modes and TMF mechanical response are so alike implies that for the higher stresses the mechanical and thermal stresses are dominant, whereas the environmental influence in these brief tests is a second-order effect because of their shorter high-temperature exposure times.

The environmental degradation is more evident in the specimens that experienced the lower applied stresses. In these specimens, differences in the damage mechanisms are as broad as the variation in their TMF lives. As shown in figure 11, the air environment generated more surface crack initiation sites than the argon. This could be due to the oxygen embrittlement of the matrix as seen in reference 6. Also there were low amounts of fiber pullout in the air-tested specimens. This implies reduced fiber bridging of the matrix cracks since fiber pullout tends to be an indicator of crack bridging. The argon specimens displayed fewer crack initiation sites, but the matrix fatigue cracks were longer. Fiber pullout was more pronounced in the argon tests, implying more fiber crack bridging was taking place. Also fiber pullout lengths of the argon test specimens were significantly longer than the air-tested specimens, indicating that fiber debonding was more prevalent in the argon tests. This was probably due to the fact that the argon tests had significantly longer TMF lives than the air tests. Therefore, the fiber/matrix interface of the argon specimens experienced more multiaxial loadings which resulted in more fiber debonding than in the air tested specimens.

An attempt was made to study the environment's influence on the in-phase TMF behavior of SiC/Ti-24Al-11Nb. At first, two tests (one in laboratory air and one in flowing argon) were conducted at a high stress level of 550 MPa. TMF lives of 4 and 251 cycles were obtained for the air and flowing argon tests respectively. A repeat test was conducted at the 550 MPa stress level in laboratory air. This time, a TMF life of 185 cycles was obtained. This result was not surprising, because in-phase TMF damage is predominately fiber controlled and the SCS-6 SiC fiber is known for its statistical variation in strength both between and within fiber lots (ref. 12). This variation has been shown to influence the composite's properties (ref. 12). In reference 12, composite tensile properties were found to depend on whether the specimen contained either predominately "weak fibers" or predominately "strong fibers." Likewise, in-phase TMF lives of a composite may vary depending on the ratio of "weak fibers-to-strong fibers" and their relative locations to each other in the composite.

Another set of in-phase tests in both environments were conducted for a stress level of 480 MPa. Here, the air test failed at 8449 cycles while the argon test was interrupted after 10 570 cycles (due to test system time limitations) with no indication of impending failure. As mentioned above, it is premature to comment about the environmental influence on in-phase TMF. Also note, that for equivalent applied stress ranges, the more life limiting TMF condition for lives above 1000 cycles is out-of-phase (fig. 7). Since we are uncertain as to whether the in-phase TMF lives were influence by the statistical nature of the SiC fiber or the environment for high stresses, and low stress level comparisons are inconclusive, a definitive statement about the environmental influence on in-phase TMF cannot be made at the

present time. To further pursue this issue, an investigation is warranted using specimens whose fiber strengths are well known and closely matched.

In closing, designers should be made aware of the effect of environmental degradation on the TMF behavior of Ti matrix composites. If these composites are to be considered for high temperature aerospace applications, the designers should then be prepared to either prescribe the use of a protective coating or decreased the maximum use temperatures, to a level where this degradation can be tolerated.

## SUMMARY AND CONCLUSIONS

(1) In general, the in-phase and out-of-phase TMF behavior of SiC/Ti-24Al-11Nb is similar to that previously observed for other Ti-matrix composites. The in-phase and out-of-phase TMF life lines intersect. In-phase lives are shorter at high stresses and out-of-phase lives are shorter at low stresses. In the "low stress-long life" regime (fig. 7), the 215 to 815 °C TMF tests have shorter lives than the 425 to 815 °C TMF tests. This is attributed to the greater CTE mismatch stresses and strains associated with the larger temperature range tests.

(2) Damage mechanisms of the in-phase and out-of-phase specimens showed trends similar to previous SiC/Ti-alloy studies as well. The in-phase specimens displayed little or no matrix fatigue cracking with matrix failure via tensile overload and extensive fiber pullout on the majority of the fracture surfaces. Out-of-phase specimens exhibited extensive matrix fatigue cracks around the outer edge of the fracture surface and matrix failure via tensile overload in the central region.

(3) Based on the present study, it is not apparent how the environment influences the in-phase TMF behavior of SiC/Ti-24Al-11Nb. This is due to the inherent nature of in-phase damage mechanisms which are associated with fiber failures and the statistical variation of the SiC fiber properties. A further investigation is warranted using specimens whose fiber strengths are more uniformed.

(4) For out-of-phase TMF, the environment plays an important role in governing damage mechanisms and hence life of the lower applied stress specimens. In this region, the air lives were over an order of magnitude shorter than the argon lives. In general, the lower stressed air specimens exhibited more crack initiation sites, shorter matrix cracks, and less fiber crack bridging, than comparable specimens tested in argon.

(5) As the maximum applied stresses of out-of-phase TMF tests were increased, there was little difference in life and damage mechanisms between the air and argon environments. The decreased dominance of an environmental effect is attributed to the decreased exposure time of the "high stress-low life" tests.

## ACKNOWLEDGEMENTS

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TABLE I.—SUMMARY OF 215 to 815 °C TMF DATA  
FOR SiC/Ti-24Al-11Nb

[Air environment.]

Specimen I.D.	Maximum stress, MPa	Fiber volume, percent	CTE, $10^{-6}$ m/m/°C	Cycles to failure, $N_f$
215 to 815 °C Out-of-phase				
90-07917-1	826	27.9	7.838	245
90-0797-1	689	22.1	8.468	286
89-0772-2	414	27.8	7.504	546
90-0795-1	276	23.2	8.255	1958
215 to 815 °C In-phase				
90-0798-1	690	25.3	8.376	4
90-0797-2	551	21.7	8.474	25
90-0794-3	415	22.4	8.258	14 405

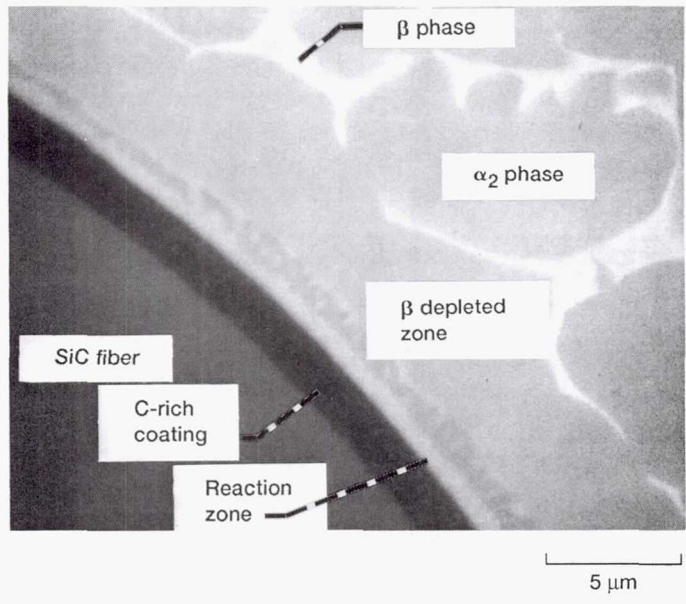
TABLE II.—SUMMARY OF 425 TO 815 °C TMF DATA FOR SiC/Ti-24Al-11Nb

[Air environment versus flowing argon.]

Specimen I.D.	Maximum stress, MPa	Fiber volume, percent	CTE, $10^{-6}$ m/m/°C	Cycles to failure, $N_f$
425 to 815 °C Out-of-phase (flowing argon)				
90-0798-3	827	24.6	8.246	520
90-0797-3	690	22.2	8.154	1114
90-0794-1	551	22.3	8.215	2737
90-0799-3	414	27.9	7.859	25 140 <sup>1</sup>
425 to 815 °C Out-of-phase (laboratory air)				
90-0799-2	825	27.4	8.208	268
90-0798-2	689	26.5	7.974	329
89-0772-3	552	27.8	7.749	467
90-0795-3	424	23.6	8.183	1359
425 to 815 °C In-phase (flowing argon)				
91-0972-6	550	24.1	7.287	251
91-0972-4	480	24.3	7.527	10 571 <sup>1</sup>
425 to 815 °C In-phase (laboratory air)				
91-0971-1	550	24.0	8.790	4
91-0972-3	550	25.4	8.431	185
91-0971-6	520	24.6	8.321	7587
90-0796-3	480	24.6	8.138	8449

<sup>1</sup>Specimen did not fail.





Fibers . . . . .	Continuous SiC (SCS-6)
	$V_f = 24$ to $27\%$
Matrix . . . . .	Ti-24Al-11Nb (atomic percent)
Layup . . . . .	$[0]_8$
Fabrication . . . . .	Powder cloth technique

Figure 1.—Microstructure of the SiC/Ti-24Al-11Nb composite.

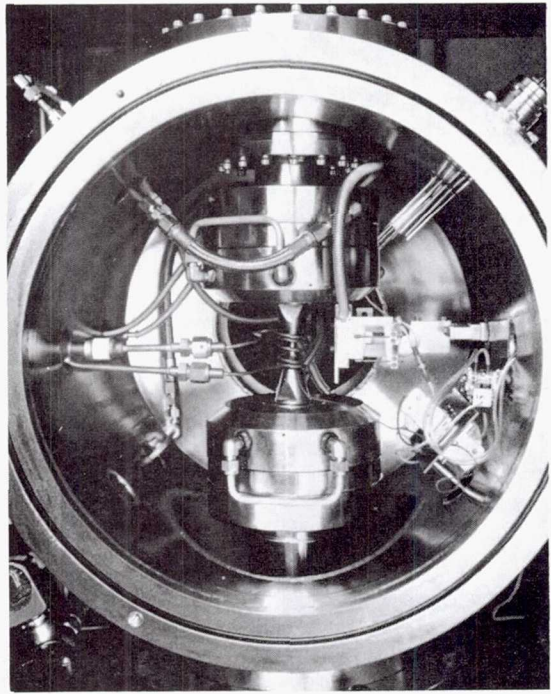


Figure 2.—Environmental test equipment.

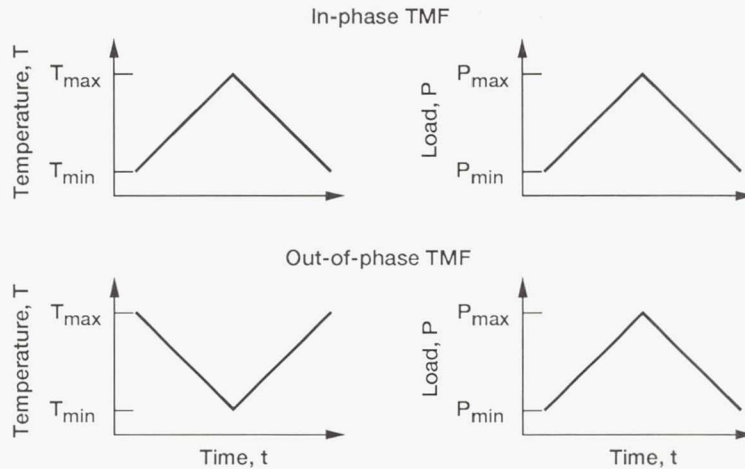


Figure 3.—In-phase and out-of-phase TMF loading history.

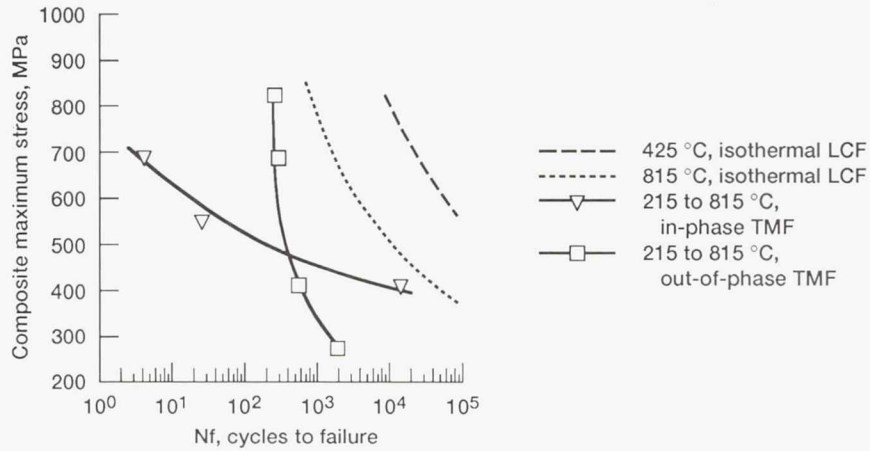


Figure 4.—Comparison of isothermal LCF and TMF lives for SiC/Ti-24Al-11Nb in laboratory air environment.

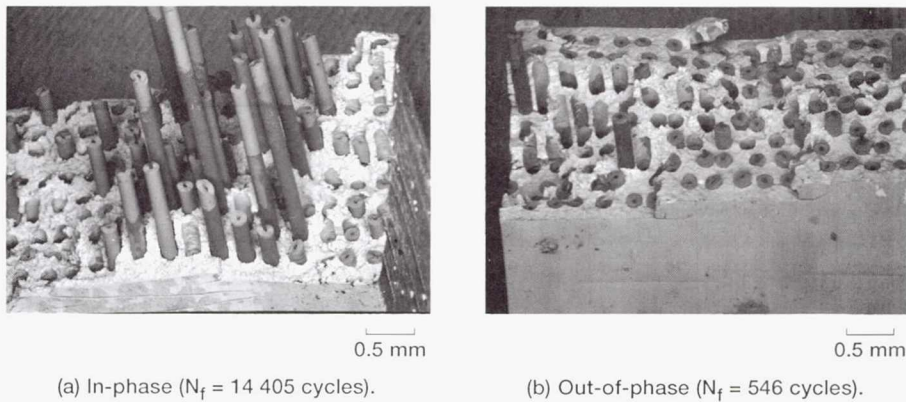


Figure 5.—Fracture surfaces of SiC/Ti-24Al-11Nb after TMF in air: (215-815 °C),  $\sigma_{max} = 415$  MPa.



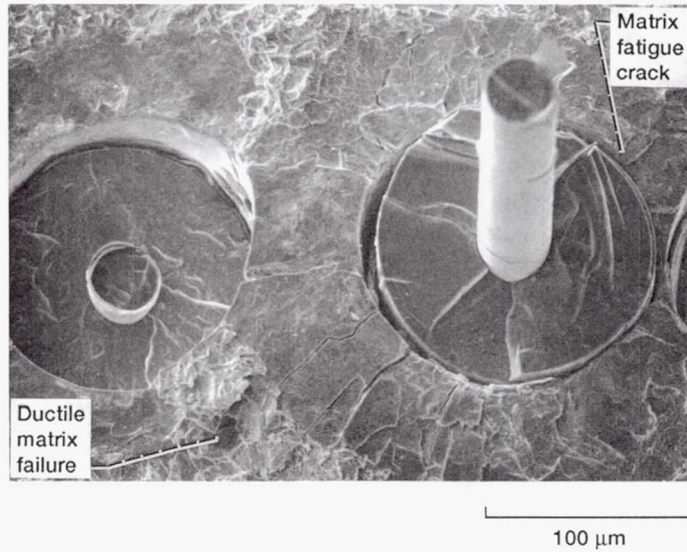


Figure 6.—Internal matrix fatigue cracks initiating at fiber/matrix interface.

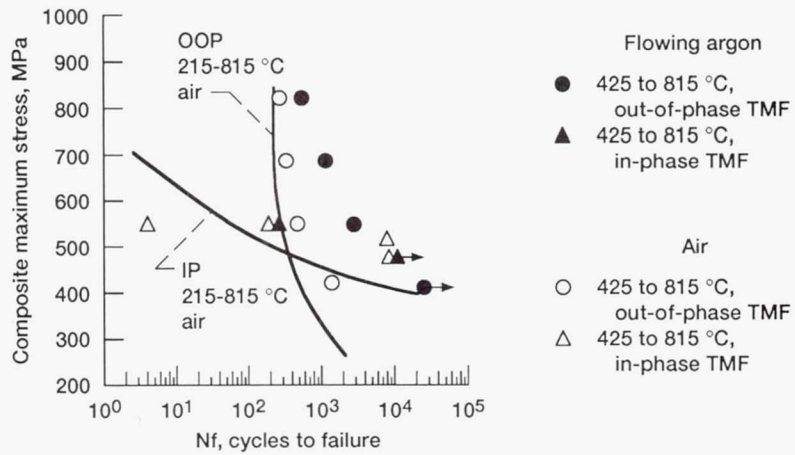


Figure 7.—Environmental effect on TMF lives of SiC/Ti-24Al-11Nb: air versus flowing argon.

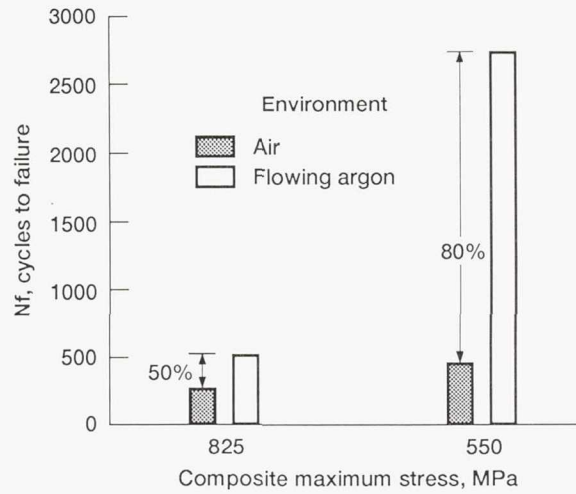


Figure 8.—Differences between air and flowing argon out-of-phase (425-815 °C) TMF lives with respect to maximum applied stress levels.

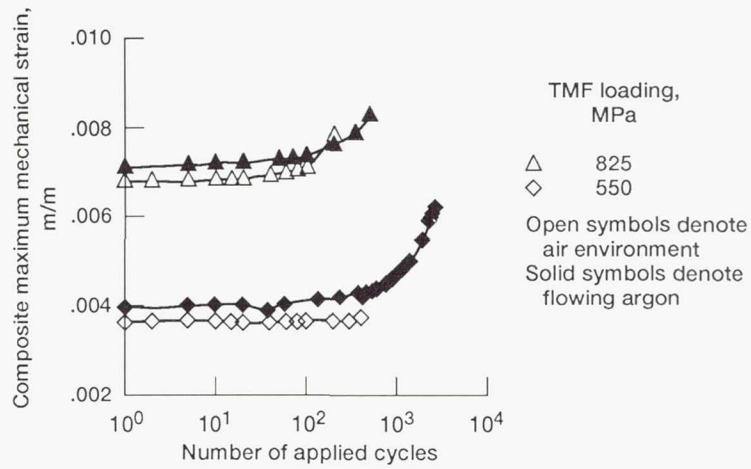
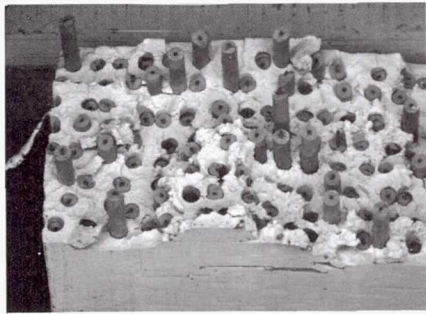


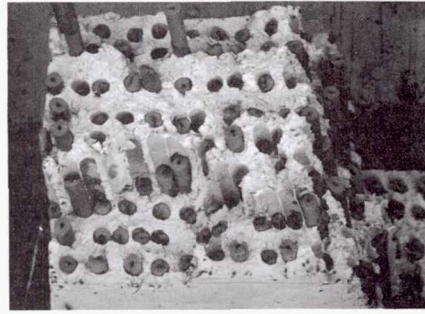
Figure 9.—Maximum mechanical strain response of SiC/Ti-24Al-11Nb under out-of-phase TMF loadings in air and argon. Note ratcheting.





0.5 mm

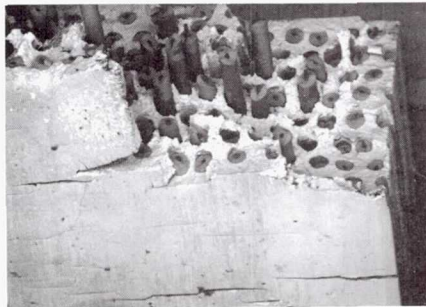
(a) Air ( $N_f = 268$  cycles).



0.5 mm

(b) Flowing argon ( $N_f = 520$  cycles).

Figure 10.—Fracture surfaces of SiC/Ti-24Al-11Nb after out-of-phase TMF in environments: (425-815 °C),  $\sigma_{\max} = 825$  MPa.



0.5 mm

(a) Air ( $N_f = 467$  cycles).



0.5 mm

(b) Flowing argon ( $N_f = 2737$  cycles).

Figure 11.—Fracture surfaces of SiC/Ti-24Al-11Nb after out-of-phase TMF in environments: (425-815 °C),  $\sigma_{\max} = 550$  MPa.

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