

Failure Criteria Development for Dynamic High-Cycle Fatigue of Ceramic Matrix Composites

Howard F. Wolfe,* Michael P. Camden,† Larry W. Byrd,‡ Donald B. Paul,§ and Larry W. Simmons¶
U.S. Air Force Research Laboratory, Wright–Patterson Air Force Base, Ohio 45433-7006

and

Ran Y. Kim**

University of Dayton, Dayton, Ohio 45469-0168

Acoustic fatigue in aircraft is typically characterized by random, high-frequency excitation that results in fully reversed bending in panels. Because of the expense of testing panels in an acoustic environment, traditional methods have first tested small cantilevered coupons on electrodynamic shakers to determine the strain vs number of cycles to failure behavior. A number of variables have been studied to provide failure criteria for the specimens. Developing criteria are examined for a relatively new class of materials, ceramic matrix composites, which are characterized by having a brittle matrix, which has a stiffness that is a significant fraction of the fiber value. Also discussed are experimental techniques that reduce the scatter associated with such data.

Introduction

ACOUSTIC fatigue research has predicted the fatigue life of aircraft designs for the last several decades.¹ Full-scale aircraft structures and components have been studied analytically and experimentally in reverberant rooms and acoustic progressive wave tubes (APWT). The simulated random acoustic loading generated in these facilities is used to generate the endurance data, closely reproducing the strain-time history found in service.^{2,3} However, with the rising cost of acoustic testing, cantilevered beam and coupon specimen testing on a vibration table has evolved.⁴ These vibration excitation methods are useful in developing stress vs cycles-to-failure (S/N) curves and strain vs cycles-to-failure (ϵ/N) curves for new materials and structural configurations. Shakers have also been used to test joining methods such as rivets, adhesive bonding, and welding. One weakness of these tests is that the load condition is one-dimensional whereas the stress in a panel is typically two dimensional. The boundary condition at the clamp may also affect the data because most panels will not have truly fixed edges. Thus, corrections are still required to apply the beam data to predictions of panel life.⁵

Vibration-induced fatigue has typically been detected by a decrease in fundamental frequency of a cantilevered rectangular specimen. The instantaneous bending stress distribution for a rectangular beam is shown in Fig. 1. The maximum and minimum stresses over time occur at the top and bottom surfaces. The excitation originally used was a constant amplitude sine wave, but with the advent of modern shaker controllers was changed to a random excitation. Random excitation gives shorter fatigue lifetimes than sinusoidal excitation at the same rms level. A number of analytical methods have been proposed to convert the results from sine tests to equivalent random lifetimes. This is desirable because the random results tend to agree better with data collected from panels and aircraft in service. Unfortunately, from the standpoint of scientifically studying the underlying micromechanics of damage accu-

mulation, a random excitation is more difficult to use because the input power spectral distribution is only known in an average (rms) sense.

Although the fatigue testing of specimens subject to in-plane loadings has been widely used, there are many problems to be solved when developing life prediction methods for specimens tested in fully reversed bending. Traditional methods used to establish failure in isotropic materials such as metals did not provide an accurate means of defining failure for new, high-temperature composite materials. Also, unlike metals, ceramics do not yield plastically under high loading, and they are usually highly susceptible to flaws. The designer typically predicts the life of a part undergoing a variable load profile using some variation of Miner's rule, with allowances for other effects that are not present during coupon testing. These effects include multimodal effects, multidimensional principal stresses, oxidation, environment, etc.

Resonant Frequency Effects

The fundamental frequency f of a rectangular cantilever isotropic beam in a standard clamp is

$$f(\text{Hz}) = 1k\sqrt{EI/\rho AL^4} = 11h\sqrt{E/12\rho L^4} \quad (1)$$

where

- E = Young's modulus, psi
- I = second moment of area with respect to the centroidal axis as shown in Fig. (1)
- ρ = density, lbm/in.³
- A = area of the beam cross section
- L = length of the beam, in.
- h = depth of the beam, in.

The stress distribution for bending is linear with the maximum stress occurring on the outer fibers of the beam and zero stress on the neutral axis. Thus, if there are no flaws cracks will initiate on the outer surface of the coupon. Once a crack initiates, it will grow across and into the specimen at a rate that is dependent on the stress distribution at the crack tip. Once a crack forms, Eq. (1) will only be approximately correct but still indicates the variables that affect the fundamental frequency. One oversimplified way to estimate the effect of crack depth on the frequency would be to use Eq. (1). If the crack extends uniformly across the specimen, calculate the fundamental frequency using the depth h at the crack location. Also calculate a new density by holding the mass of the beam constant.

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*Aerospace Engineer, Air Vehicles Directorate, Associate Fellow AIAA.

†Aerospace Engineer, Air Vehicles Directorate.

‡Mechanical Engineer, Air Vehicles Directorate, Member AIAA.

§Chief Scientist, Air Vehicles Directorate, Associate Fellow AIAA.

¶Engineering Technician, Air Vehicles Directorate.

**Senior Research Engineer, Research Institute.

The equivalent density would be $\rho_2 = \rho_1(h_1/h_2)$. Substituting into Eq. (1) gives a formula for the fractional change in fundamental frequency as

$$\frac{\Delta f}{f} \left(\frac{h_1/\sqrt{\rho_1} - h_2/\sqrt{\rho_2}}{h_1/\sqrt{\rho_1}} \right) = 1 - \left(\frac{h_2}{h_1} \right)^{\frac{3}{2}} \tag{2}$$

Thus, a crack that is 6.8% of the original depth of the coupon will give a 10% decrease in the natural frequency using this simplified approach. This is a typical value of the decrease in fundamental frequency defined as failure for metals. Cracks may occur on both sides of the specimen, and so the actual crack depth on each side could be half this value, or 3.4%. For a specimen with an original thickness of 2.032 mm (0.08 in.) a crack depth of 3.4% corresponds to 0.0691 mm (0.0025 in.). Note that this ignores changes in Young’s modulus that may occur in a localized area on the outer fibers of the coupon and that the actual crack is not of uniform depth across the specimen. This simplified analysis assumes the loss of stiffness is proportional only to changes in the second moment of inertia. However, changes in modulus, for example, due to fiber breakage, could also cause a drop in stiffness. Thus, using a decrease in frequency is reasonably sensitive to damage in the specimen and provides an alternative to visual inspection for cracks. It has been used extensively and has become the standard method for establishing failure.

Currently there is no standard value to be used as the correct drop in fundamental frequency at which point one would indicate failure. Values chosen by different researchers range between 2 and 10% depending on the material, but this can sometimes result in an order of magnitude of the defined fatigue life because the slope of the S/N curve is typically very small. Note that the response to the random input will always have fluctuations that may be as high as 2%, and so some type of smoothing is needed. Figure 2 illustrates some variations in the recorded data. To further complicate the problem, it takes many hours of run time to accumulate high cycles ($> 10^7$). As an example, to accumulate 10^8 cycles on a ceramic matrix composite (CMC) material specimen with a resonant frequency of 300 Hz, it takes 93 h of run time. With these long run times it can take months to develop a single (ϵ/N) curve. The inspections can also require a great deal of time, especially for new materials where the methods to determine crack initiation and the failure mechanisms are not known and have not been developed. Thus, it is critical that correct failure criteria be used for development of (ϵ/N) curves for CMC materials such as Blackglas™.

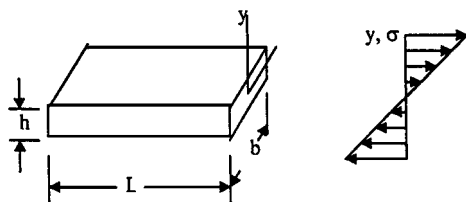


Fig. 1 Rectangular beam specimen and instantaneous bending stress distribution.

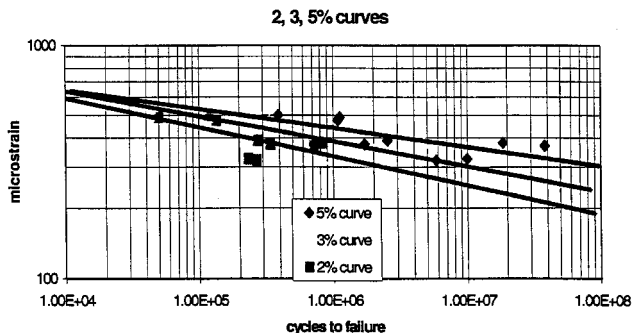


Fig. 2 Curves of 2, 3, and 5% for ceramic matrix composite coupons at room temperature.

CMC Fatigue Curve Development

Single rectangular cantilevered beam coupons were used initially in these fatigue tests. The testing arrangement for these earlier tests with the rectangular clamping of the beam is shown in Fig. 3. The beam was mounted on the shaker head with the motion perpendicular to the beam. These beam specimens require rigid clamping devices to provide the necessary bending moment at the clamped boundary. The clamping devices should also have sufficient stiffness to make the torsional and fixture resonant frequencies higher than the coupon bending resonant frequencies of interest. A more detailed description of the test coupons and the test setup is found in Ref. 6. The input excitation bandwidth was centered around the fundamental frequency of the beam. It was wide enough to include changes in the frequency while damage accumulated. Early data obtained for CMC materials were very limited and based on failure criteria used for metals. The cycles to failure were determined from the product of the time to failure and the resonant frequency. Failure was determined by a sharp downward change in the resonant frequency of the CMC cantilevered beams.

The resonant frequency in testing composite coupons usually shows as a small reduction during the initial fatigue test. The time required for this to take place is referred to as settling time. This is usually followed by a time when the frequency is constant or drops slowly. The frequency used in calculating the cycles was the reduced frequency immediately after the settling time. A sharp reduction in frequency usually follows after some time has elapsed, indicating crack initiation and some crack propagation. The time it takes for the small reduction in frequency is usually short and does not affect the time to failure significantly. When it is not short, the change in frequency method is not as accurate.

A typical method of defining failure in metals is to determine the time for a sharp resonant frequency reduction. A shift of about 10% is generally used. At this time the crack had initiated and started to propagate. The cracks were usually large enough to see without magnification and enhancement. The failure times for the CMC coupons were determined from a plot of percent frequency shift vs cycles to failure (F/N) as shown in Fig. 4. The entire time record can

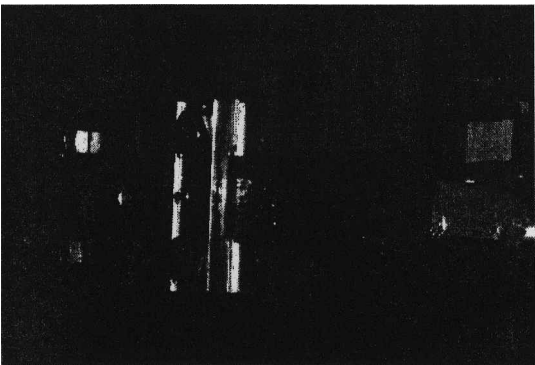


Fig. 3 Test setup using a conventional clamp.

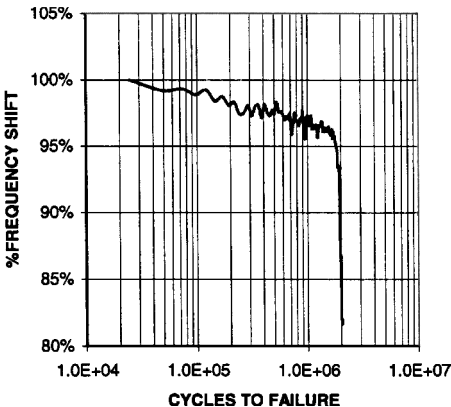


Fig. 4 Typical F/N curve.

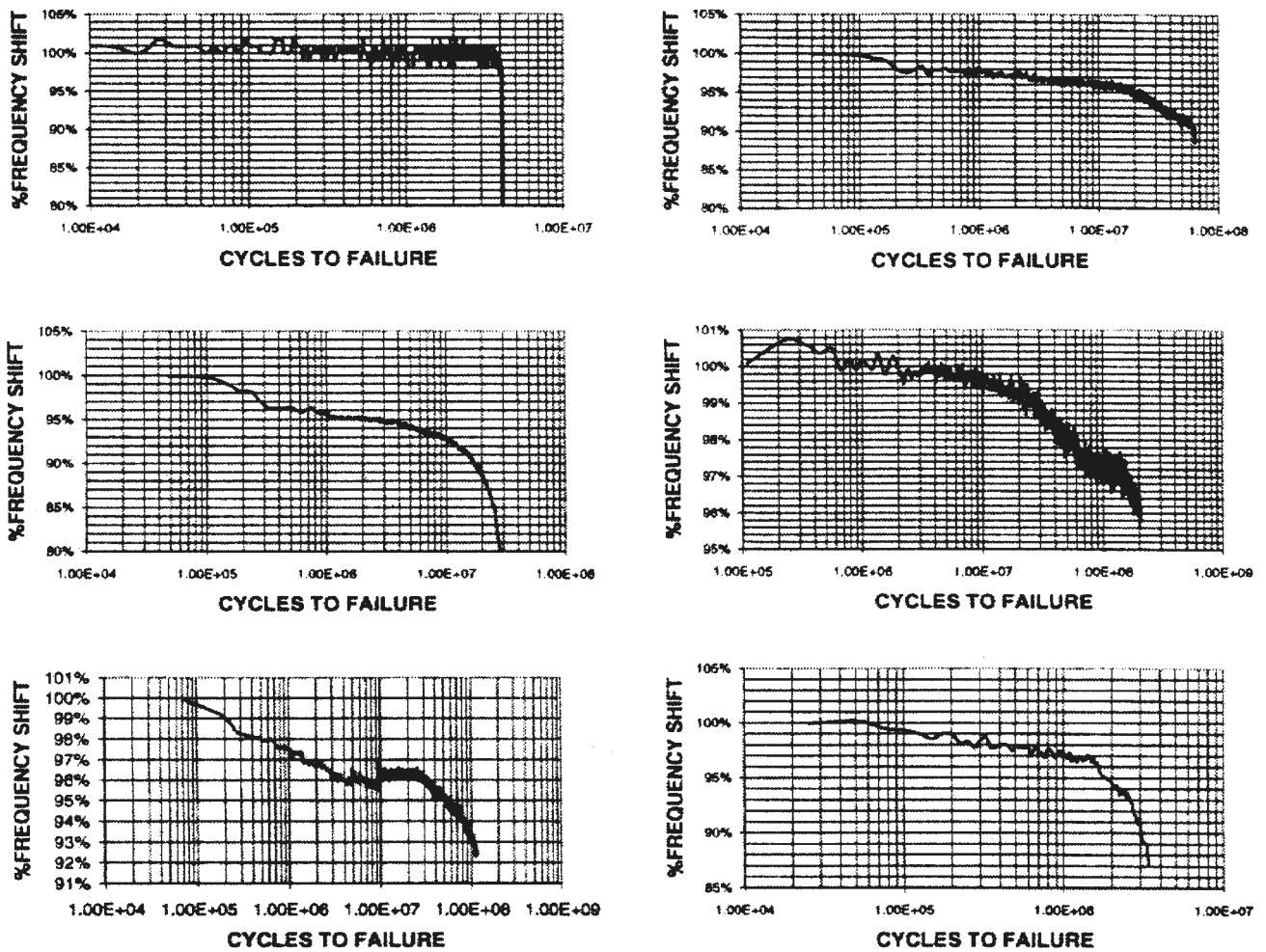


Fig. 5 Compilation of six F/N curves developed from typical CMC specimens.

be plotted on a log scale that compresses the time record. Figure 5 shows a compilation of six different F/N curves developed from CMC specimens. All six specimens are CMC specimens; however, their resulting F/N curves vary dramatically. The time to failure for the top left F/N curve is clear. The resonant frequency fell off very quickly, indicating that crack initiation and some propagation had taken place. The time to failure is not clear for the remaining samples shown in Fig. 5. The settling time and the quick falloff of resonant frequency was not apparent. Both the reduced frequency and the time to failure were unclear. Visual inspections of the metal coupons tested in the past usually determined that a crack found in the material was long enough to be found without magnification when a 10% reduction in the resonant frequency was reached. A (ε/N) curve using a percent shift in frequency was developed for a CMC sample using a 2, 3, and 5% shift in the starting resonant frequency. The starting frequency was the reduced frequency after the settling point. This is the traditional way of establishing the time to failure in metals. As can be seen in Fig. 2, a slight change in the percent shift in frequency used to define failure results in a very different fatigue life. The slopes of the (ε/N) curves are small compared with those obtained for aluminum and titanium alloys. This means that much smaller changes in the rms strain result in a much larger change in the cycles to failure.

Damage Detection

Visual inspections of the metal coupons usually determined that a crack found in the material was long enough to be found without magnification and that a 10% reduction in the resonant frequency was reached. Failure in the CMC coupons was determined by the change in resonant frequency long before a crack could be observed visually. Cracks in most of the test specimens were not completely across the beam. The beams were fractured with a small pressure

applied by hand. The fractured surfaces were jagged. Micrographs of fractured surfaces were taken and will be discussed in detail in the next section. The edges of the beam were examined after each inspection interval for cracking. This was carried out to determine if the crack initiated at the edge of the beam; however, a visual inspection was unable to determine this. The type of failure or mode of failure should be consistent to produce accurate lifetime prediction techniques.

In the case of composites, defining failure is not as straightforward as it is with isotropic materials. There is a much broader range in the material response as it comes from the manufacturer, and this leads to more scatter in the data. Another problem with composites is that edge defects from cutting and moisture adsorption can cause premature failure. The composite modulus depends on the volume fraction of the fibers, the magnitudes of the matrix and fiber moduli, fiber orientation, and manufacturing flaws such as voids or cracks. For ceramic matrix composites there are often microcracks from the manufacturing process. The fiber is often coated with a lower strength material to provide fiber slippage to improve toughness, although this will decrease the strength. Current studies are underway to look at the crack structure, damping, and changes in the torsional fundamental frequency as damage accumulates. Using a fixture that holds four specimens simultaneously will do this. This should provide an indicator on the scatter to be found in the material. Also, by removing individual specimens at set intervals for evaluation, damage accumulation can be investigated.

CMC Fatigue Curve Interpretation

As was mentioned earlier, the number of cycles to failure is determined from a plot of fundamental frequency as a function of the number of applied cycles at a given rms strain level. With composites, however, the point of failure is more difficult to characterize

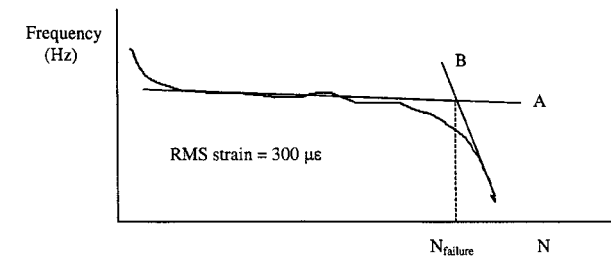


Fig. 6 Defining a failure point from an F/N curve.

from this plot. This results in a large amount of scatter in the data around the derived S/N curve. This paper will discuss some possible ways to interpret the F/N curve and reduce scatter.

One possible technique to interpret the F/N curve is to calculate the coefficient of determination r^2 value of curve fits to ε/N data using the assumed standard power law behavior, using different values of frequency decrease to define failure. The curve fit with an r^2 value closest to 1 shows the least scatter in the data and, thus, that frequency reduction would be used. The problem with this approach is that it assumes that a power law with constant coefficients describes the behavior. It is known that low-cycle fatigue will show different values for the curve fit parameters than those typically found in high-cycle fatigue, and so this may be forcing a form to the data that is inappropriate. It also does not address the problem of the visual interpretation that is used to decide when the settling period has ended. Two other possible ways to reduce the data to remove some of the visual interpretation of the F/N curve are presented next.

The second method, which is shown in Fig. 6, would be to define the failure point to occur at the intersection of two straight lines. The first line A is constructed to be tangent to the curve in the midphase, that is, after the settling period has appeared to have finished and before the sharp decrease that signifies failure. The second curve B is tangent at a point where the fundamental frequency has decreased by a set amount from its highest value. This method has an advantage of simplicity, and it should give relatively consistent results when used by different people, although it is still somewhat dependent on the judgment of the individual. It is also dependent on where lines A and B are drawn.

The third method calculates the curvature of a smoothed line through the data points and then uses the point where the normalized curvature starts its final monotonic increase as failure initiation. The curvature is the reciprocal of the radius of curvature of a given line. A small radius of curvature results in a large curvature. Thus, data that are flat have a curvature of zero, and the region where a large drop is found has a large curvature. This method is similar to looking at the second derivative because for the flat region there is little difference between the curvature and the second derivative. Two quantities were studied, the curvature and the rate of change of the curvature. The advantage of this method is that the information during the early part of the test, that is, the settling period, is not needed to define the failure criteria.

The curvature $K(x)$ in a line $y = f(x)$ is given mathematically as

$$K(x) = \frac{|f''(x)|}{[1 + (f'(x))^2]^{\frac{3}{2}}} \tag{3}$$

where $f'(x)$ and $f''(x)$ are the first and second derivatives. The rate of change of the curvature is the first derivative or the slope of the curvature and is

$$K'(x) = \frac{(1 + (f'(x))^2)f'''(x) - 3f'(x)(f''(x))^2}{[1 + (f'(x))^2]^{\frac{5}{2}}} \tag{4}$$

An example is a sixth-order polynomial fit using the least-squares method. This resulted in a curve fit of the form

$$f(x) = \sum_{i=0}^6 a_i x^i \tag{5}$$

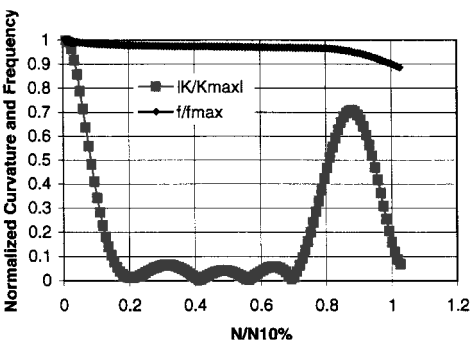


Fig. 7 Normalized curvature and normalized frequency for a CMC coupon.

where a_i are the constants determined from the data.

The derivatives are given by

$$\begin{aligned} f'(x) &= \sum_{i=1}^6 i a_i x^{i-1}, & f''(x) &= \sum_{i=2}^6 i(i-1) a_i x^{i-2} \\ f'''(x) &= \sum_{i=3}^6 i(i-1)(i-2) a_i x^{i-3} \end{aligned} \tag{6}$$

The curvature and its slope were normalized by dividing by the maximum value of each quantity. This always occurred at the end of each data set. This is inconsistent in that some of the runs had data with a much larger percent decrease in frequency than others. A decision would have to be made whether to use a settling period or the initial frequency, but it is expected the result would be somewhat insensitive to either one. A study is underway to see which method will give the least scatter in the (ε/N) curve. Also scaled for study was the number of cycles. This was done by using the number of cycles to failure as determined by the curvature or curvature slope as the reference value. Figure 7 illustrates the absolute value of the normalized curvature and normalized frequency response of a CMC specimen. As can be seen, the largest curvature occurs during the initial settling period and at failure. $N10\%$ is the number of cycles that result in a 10% decrease in fundamental frequency.

The design of the fixture can significantly affect the time to failure. If inspections are planned that require the removal of the test specimen from its fixture, then the fixture should be designed so that the specimens can be easily removed. A considerable amount of time can be saved if failure can be determined without removing the test specimen. Also, the method to determine failure in composite coupons needs to be automated to save time and to eliminate the human judgment factors.

When developing (ε/N) curves for metals, the beams are clamped at one end with the clamp perpendicular to the long axis of the specimen. If this type of fixture is used for CMC specimens there is concern about edge effects causing premature and unrealistic failures. It is difficult, if not impossible, to cut CMC materials without causing any damage to the edges of the specimens. New clamping devices are planned for future tests to add to the accuracy of failure criteria. One clamping device that has recently been used is the half-sine clamp. This clamp is shaped like a half-sine wave and induces high stress concentrations into the center of the test specimen. This clamp can be seen in Fig. 8. It looks as if this clamp would cause premature failure of specimens due to the induced stress concentration at the tip. When testing two different types of CMCs the half-sine clamp showed an increase in life of 10 and 100 times over a conventional clamp. This provides a good example as to the damage induced into the edge of the CMCs when they are cut into specimens.⁶ Other methods are planned to detect the time of early damage. One method uses thermography to determine the temperature distribution that is related to the strain energy on the coupon surface. Another method uses an infrared differential thermography system to measure surface strain. This system has the capability

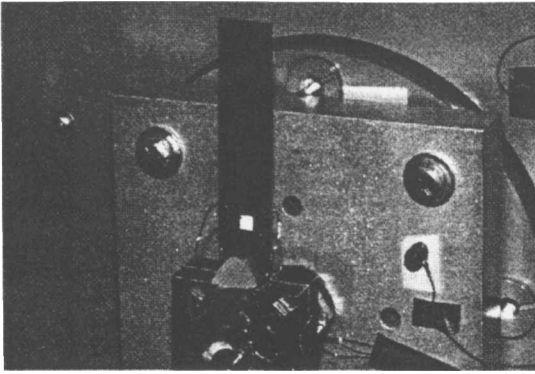


Fig. 8 Half-sine clamp.

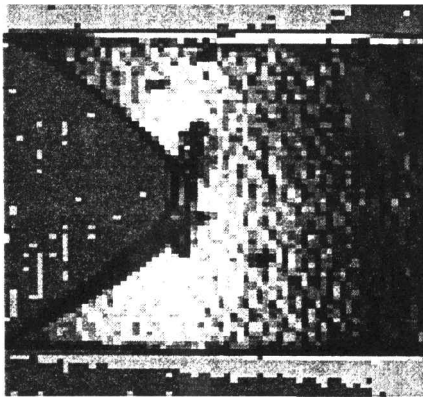


Fig. 9 Crack initiation at center of Blackglas coupon.

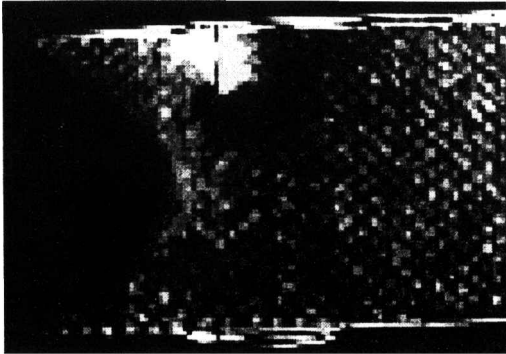


Fig. 10 Crack initiation at edge of Blackglas coupon.

to measure principal stresses of coupons and structures being excited with a sinusoidal input. It works based on the principal that materials heat up or cool down when they are loaded in tension or compression. Both methods are discussed in greater detail in Ref. 6. The liquid nitrogen cooled thermography system has a resolution of 0.001 K. A crack that was initiated in the center of the Blackglas coupon at the tip of the clamp is shown in Fig. 9. The crack started near the tip of the half-sine wave clamp and extended across the width of the coupon. Figure 10 shows a Blackglas coupon being tested in a half-sine clamp that failed due to edge effects. This shows the amount of damage that can be induced into a specimen when cutting it. Even with the high stress concentration at the tip of the clamp, for some specimens the edge effects still cause premature failure of the CMCs.

A fixture that holds four specimens was built to speed up the process of testing and also to allow damage accumulation to be studied by microscopic means. This is not a new idea; in the past, multiple specimens were loaded, and the number of cycles carried out until half the specimens failed was used as the life for the given

strain. The tests with the four specimens were not completed at the time this paper was written. It is hoped that it will allow data to be collected that can be used in micromechanical models. It also allows a means of estimating the variability in the material because all coupons are seeing the same load.

Failure can also be detected by an increase in the structural damping. Increased damping is expected between damage initiation and crack propagation. Damping measurements were taken before the fatigue tests and during the fatigue tests of Blackglas. The transfer function was determined by dividing the vibrometer signal response by the accelerometers signal. The percent damping was measured using the one-half power point method on the transfer functions. Many of the damping measurements more than doubled as the fatigue crack initiated and progressed across the coupon. In some cases, the damping measurements actually decreased with more test time; however, failure could not be established reliably by a steady increase in structural damping.

Fracture Analysis of Failed Specimens

An additional part of understanding when failure occurs in CMCs involves the investigation of crack initiation and propagation through the use of both destructive and nondestructive techniques such as examination using a microscope and a scanning electron microscope (SEM). The specimens examined were failed under fully reversed bending, while being dynamically loaded, using a half-sine clamp. Damage being investigated includes microcracking in the matrix and interface, interfacial debonding, fiber breakage, and delaminations. Two specimens were examined using these and additional techniques. The first specimen (#10) was examined in the SEM. Micrographs showed considerable debonding at the fiber/matrix interface and scattered fiber pullouts. This damage can be seen in Figs. 11 and 12. The majority of the damage is formed by coalescence of fiber/matrix interface debonds. Horizontal cracks (intra- or interlaminar) were also observed in the central region

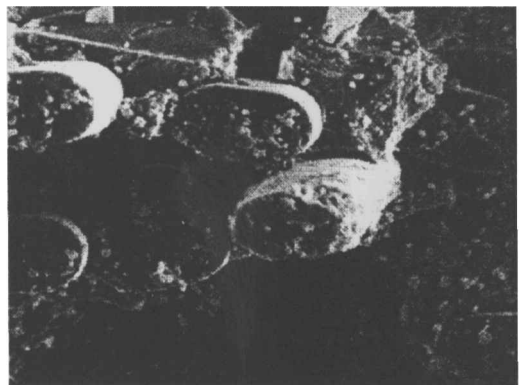


Fig. 11 Micrograph of fiber pullout and interfacial debonding.

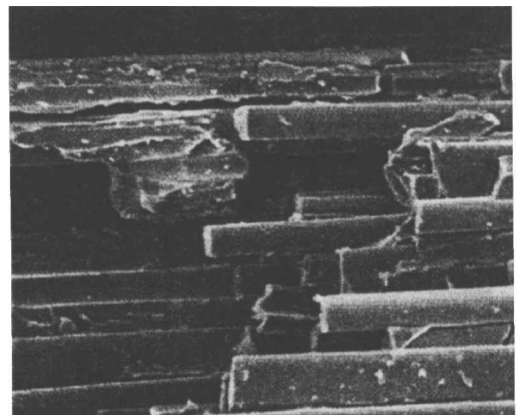


Fig. 12 Micrograph of fiber pullout and interfacial debonding.

where the neutral axis is located. This can be seen in Fig. 12. As expected, the fiber fracture surfaces indicated a dominant tensile failure mode.

Conclusions

The problems associated with generating ceramic matrix composite fatigue curves with cantilevered beam coupons require much more accuracy than those used for metal structures. The time to failure for the Blackglas coupons was determined from the 3% frequency vs cycle to failure curve. This was because the 3% curve fell between the 2 and 5% curves. Using the eyeball method, it was a good average of all of the data. The 3% change in the frequency response was used to determine failure time for Blackglas as opposed to the 10% shift typically used for metals. The material itself, the manufacturing quality, design of the beam, and design of the clamping devices affect the time to failure. Inspection techniques can also indirectly affect the time to failure in composite coupons due to subjective judgements required to determine failure; however, infrared and differential thermography systems provide promising inspection methods. Structural damping measurements were taken before the fatigue and during the fatigue tests. The damping mea-

surements did not improve the time to failure accuracy. The method to determine failure in composite coupons needs to be automated to save time and to eliminate the human judgment factors.

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