Microstructural and Defect Characterization in Ceramic Composites Using an Ultrasonic Guided Wave Scan System

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ABSTRACT. In this study, an ultrasonic guided wave scan system was used to characterize various microstructural and flaw conditions in two types of ceramic matrix composites, SiC/SiC and C/SiC. Rather than attempting to isolate specific lamb wave modes to use for characterization (as is desired for many types of guided wave inspection problems), the guided wave scan system utilizes the total (multi-mode) ultrasonic response in its inspection analysis. Several time- and frequency-domain parameters are calculated from the ultrasonic guided wave signal at each scan location to form images. Microstructural and defect conditions examined include delamination, density variation, cracking, and pre/post-infiltration. Results are compared with thermographic imaging methods. Although the guided wave technique is commonly used so scanning can be eliminated, applying the technique in the scanning mode allows a more precise characterization of defect conditions.

INTRODUCTION

Ceramic matrix composites (CMCs) are being developed for advanced aerospace propulsion applications in order to save weight, to improve reuse capability, and to increase performance. However, mechanical and environmental loads applied to CMCs can cause degradation in the form of discrete flaws and distributed micro-damage that plays a significant role in reduction of desirable physical properties. Categories of microdamage include fiber/matrix debonding [interface failure], matrix microcracking, fiber fracture and buckling, oxidation, and second phase formation. A recent study of the durability of a ceramic matrix composite, C/SiC, discussed the requirement for improved nondestructive evaluation (NDE) methods for monitoring degradation in these materials. Distributed micro-damage in CMCs has proven difficult to characterize nondestructively due to the complex micro- and macro-structure of these materials. This article discusses the use of an ultrasonic guided wave scan system to characterize various microstructural and flaw conditions in SiC/SiC and C/SiC ceramic matrix composite samples.

Ultrasonic guided wave inspection is generally thought of as an attractive alternative to scanning because guided waves can be excited at one location of a structure by a single transducer or line of transducers with returning or received echoes indicating

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the presence of defects [2–6]. This type of inspection has seen success detecting flaws and material degradation in many types of materials and components, and in some applications over significant distances. The guided wave signal in raw form is very complex (many dispersive and interfering modes present traveling at different velocities) with significant coherent noise that cannot be averaged out. As a result, guided wave methods seem to be most successful for flaw detection when tuning for a minimally- or non-dispersive mode of ultrasound, at a particular excitation frequency, in one direction, in order to control coherent noise [2,5]. The guided wave method discussed in this article takes a different approach by utilizing the total (multi-mode) ultrasonic response, doing so in a scanning configuration, and employing specialized signal processing routines to extract parameters of the time- and frequency-domain signals [4,6,7]. These parameters have proven to be sensitive to changes in microstructural conditions [4,6,7] and to the presence of defects [8], and appear promising at monitoring degradation in CMCs [9].

ULTRASONIC MULTI-MODE GUIDED WAVE SCAN METHOD

Measurement

The multi-mode guided wave method is a single-sided technique with the basic measurement employing separated sending and receiving transducers at normal incidence to the sample similar to acousto-ultrasonic interrogation (figure 1) [4,6]. Ultrasonic guided waves through the material thickness are generated when the ultrasonic wavelength is on the order of the thickness of the material being interrogated. The key factor in multi-mode ultrasonic guided wave interrogation is that the relationship between the ultrasonic wavelength ($\lambda$) and material thickness (h) results in a diffuse field of multiple plate wave modes [4].

![FIGURE 1. Schematic of multi-mode guided wave experimental setup. S = distance between sending and receiving transducers.](image)

Although guided wave techniques are commonly used so scanning can be eliminated, applying the techniques in the scanning mode likely allows a more precise characterization (location, size, shape) of defect conditions. For example, although a simple guided wave method may determine whether a defect does or does not exist, a guided wave scanning technique may be able to image the same defect and allow more accurate identification of type and more accurate size estimation [7,8]. The scan setup is shown in figure 2.

![FIGURE 2. Scan setup.](image)

Depending on the situation, there may be several further advantages of using guided wave scanning over conventional ultrasonic methods. These include:
Guided wave scanning can be performed directionally allowing correlations to be made between ultrasonic parameters and directionally-dependent material properties (such as for unidirectional composites or to test the premise of non-directionality of properties).

The sample under test does not have to be immersed in fluid (as for most conventional ultrasonic characterization).

Other positive features of guided wave scanning include:

- Guided wave scanning is potentially applicable to components with mildly curved surfaces.
- Guided wave scanning is potentially more versatile in characterizing local modulus changes than is the resonant frequency method [10] since the resonant frequency methods require nodal excitation and generation and are thus not applicable for scanning.

**Ultrasonic Signal Analysis**

As mentioned above, the guided wave scan system utilizes the total (multi-mode) ultrasonic response in its inspection analysis. The analysis is based upon the premise of internal absorption and scattering mechanisms being primarily responsible for ultrasonic attenuation with minimal damping due to air, fixturing, and specimen edges. Under these conditions, the morphology of the waveform at the receiving transducer is that of a short duration rise (dominated by early plate wave modes L-11 [first symmetric {longitudinal}...
mode] and L₁₂ [first anti-symmetric {shear} mode]) followed by an exponentially-decaying portion. A “model” waveform is shown in figure 3.

Analysis is performed in several “domains” in this investigation. First, different parameters associated with the amplitude-based time-domain waveform are calculated. These include centroid mean time and time domain skew factor. The power spectral density (PSD) (frequency domain) of the time-domain signal is calculated and resulting parameters such as Zeroth moment and PSD centroid are obtained. Mathematical expressions for both sets of parameters are provided in [2,5]. Briefly, these parameters are described as follows. Centroid mean time can be thought of as the time in the raw waveform demarcating the location of energy balance. TD skew factor denotes the tilt or shift of the raw waveform towards or away from the beginning of the time interval. The properties of TD Skew Factor are that it varies between +1 and -1. When it is greater than zero the waveform is skewed toward the beginning of the time interval, as with an exponentially decaying signal. The closer to +1 the time domain skew factor is, the greater the skew towards the beginning of the time interval. Zeroth moment is the area under the PSD and is a measure of energy density. Centroid of the PSD can be thought of as the frequency in the PSD demarcating the location of energy balance. Many other parameters of the time- and frequency-domain waveforms, and their envelopes, are being investigated in terms of their effectiveness for differentiation of material state.

Additionally, the waveform can be described in terms of energy density (|φ(t)|²) as a function of time and frequency (amplitude response squared equals energy response) containing frequency dependence since the frequency components of the waveform may change as a function of time. The exponential decaying portion of the waveform can then be expressed by [4]:

\[
|φ(t)|² = K(φ) \exp(-β(t))
\]

where β(t) is the frequency-dependent ultrasonic decay rate which describes wave decay over time, and K(φ) is the frequency-dependent initial energy density. β(t) is analogous to the frequency-dependent attenuation coefficient α(t) which describes wave decay over distance. However, since there is no precise wave path length that is easily defined, the current method for calculation of β(t) requires first dividing the time domain wave into time partitions, obtaining power spectral density (PSD) of each partitioned portion of the time domain waveform (using the short-time fourier transform), calculating the area under each PSD curve to obtain energy density (zeroth moment) of each PSD, plotting the energy density values versus the midpoint of each time partition, and obtaining the best exponential fit of this curve from its peak to obtain K(φ) and β(t). The step of obtaining PSD of each partitioned portion allows one to view how the frequency components of the waveform change over time and is obtained using the short-time fourier transform.
EXPERIMENTAL

In this investigation, several ceramic matrix composite specimens were examined using the guided wave scan system. The samples included Silicon Carbide fiber/Silicon Carbide matrix (SiC/SiC) samples containing delamination and regular pattern of density variation, SiC/SiC samples prior to and after silicon infiltration of the carbon (C) perform, and C/SiC creep tensile samples undergoing interrupted creep tensile testing. Broadband ultrasonic transducers were employed with center frequencies ranging from 1 to 3.5 MHz (both sender and receiver were of the same frequency). Ultrasound was coupled to the material via elastic coupling pads. Analog-to-digital sampling rates used were 10 MHz to 25 MHz. Scan increments varied between 1 and 5 mm. Each sample was scanned 2 to 4 times to characterize reproducibility [8]. As described in the previous section, images constructed included those calculated from centroid mean time, time-domain skew factor, zeroth moment and centroid of the PSD, frequency-dependent ultrasonic decay rate and frequency-dependent energy density initial value. Thermographic NDE utilizing flash lamps and a state-of-the-art high speed camera was used to obtain images of the samples for comparison with the ultrasonic guided wave scan images.

RESULTS

The SiC/SiC sample containing delamination had the delamination most easily discriminated in the centroid mean time and zeroth moment images (whitish areas in figures 4 and 5). Time-domain waveforms associated with the delaminated area and a non-delaminated area are shown in figure 4b. The shift in centroid mean time away from the origin is quite apparent for the delamination when viewing the waveforms. Power spectral densities associated with the delaminated area and a non-delaminated area are shown in figure 5b. The increase in the area under the power spectral density (zeroth moment) is quite apparent for the delaminated area. Thermographic images also revealed the delamination.

The SiC/SiC sample containing the regular pattern of density variation had the density variation most easily discriminated in the centroid mean time (“dots” represent lower density areas) (figure 6) and time domain skew factor images (not shown due to space considerations). In this case the change in the morphology of the time domain waveform from a high to a lower density area is much more subtle and one cannot rely on visual discrimination of the waveforms as was possible for the delamination. Instead, one

![FIGURE 4. a) Centroid mean time image of SiC/SiC sample. Whitish area in lower left of image is delaminated area. b) Time-domain waveforms associated with delaminated and non-delaminated area.](image-url)
must rely solely on the image. Thermographic images also revealed the density variation. Approximately 10% thickness variation corresponding to the spots of density variation was noted which could affect the ultrasonic results. However, similar ultrasonic results were observed for a plate containing the regular density variation but no appreciable thickness variation [7].

FIGURE 5. a) Zeroth Moment image of SiC/SiC sample. Whitish area in lower left of image is delaminated area. b) Power spectral densities associated with delaminated and non-delaminated area.

FIGURE 6. Centroid mean time image of SiC/SiC sample with regular pattern of density variation.

For the SiC/SiC sample prior to and after silicon infiltration of the carbon perform, the zeroth moment increased uniformly over the entire sample 100x after infiltration (from mean = 0.0036 V^2 to mean = 0.371 V^2). Centroid decreased uniformly over 50% after infiltration (from mean = 0.83 MHz to mean = 0.33 MHz). Typical power spectral densities clearly discriminate the two microstructural conditions by amplitude, energy density (zeroth moment), and frequency content (centroid) (figure 7). These results can be explained by the fact that the holes in the composite structure are being filled with material (Silicon) making the structure less attenuative. However, it must be noted that the surface condition also changed prior to and after infiltration which likely affected ultrasonic results.
Finally, the C/SiC creep-tensile-tested sample scanned had been removed from testing after 16 hours (testing conditions were temperature = 1200 °C, tensile stress = 10 Ksi, environment = air). For this sample, the zeroth moment and frequency-dependent ultrasonic decay initial energy clearly showed indications different from the rest of the sample at the X=10-11 cm location of the sample (whitish areas) (figure 8). These indications are likely of the crack or open space nature as carbon fibers disappear due to oxidation [1]. The critical points regarding these results include 1) these indications were not apparent by any means after 14 hours of creep-tensile testing which indicated major degradation likely occurred at the X=10-11 cm location between 14 and 16 hours of testing, 2) these indications were not apparent in optical or thermographic images after 16 hours but were apparent in the guided wave images noted, and 3) the sample failed at the location of the indication soon after being put back into the creep-tensile testing rig for another 2 hours of testing. The zeroth moment mean decreased an order of magnitude from 14 to 16 hours of creep tensile testing (~ 0.04 V² decreased to ~ 0.004 V²). A similar decrease was seen in ultrasonic decay initial energy mean from 14 to 16 hours (0.724 V² decreased to 0.083 V²).
CONCLUSIONS AND UPDATES

This article focused on surveying the ability of the multi-mode guided wave scan system for characterizing microstructural changes and defects in SiC/SiC and C/SiC ceramic matrix composites. Delamination, density variation, microstructural change due to silicon infiltration, and crack space indication, were revealed in images formed from several time- and frequency-domain parameters of the ultrasonic guided wave signal. The crack space indication revealed was not seen by other techniques and was the eventual failure location, both critical results. Zeroth moment and centroid overall seem to be the most consistently sensitive to defect and varying microstructural condition but other parameters such as time domain skew factor and frequency-dependent ultrasonic decay initial energy have provided promising results. Many other time- and frequency-domain parameters are being investigated. Which parameters prove most promising might be material and microstructural-condition dependent. Therefore, it may be critical to form and view many parameter images together as the scan proceeds which is a capability the guided wave scan system has. Theoretical modeling is needed to explain results seen and actual standards need to be developed to aid in the theoretical modeling.

A description of the initial version of the NASA GRC guided wave scan system was given in [7–9]. Since that period, several updates have occurred. First, a pci-based 25 MHz pulser-receiver card was added to the computer so that external pulser-receiver and preamplifier were no longer needed. The only instrument that currently resides outside of the computer (aside from the scanning hardware) is the motion controller/indexer. Second, the scan system has been assembled into a frame with the motion controller mounted on the frame for more compact and robust construction (figure 2). Third, a second receiving transducer has been added to allow guided wave velocities to be mapped (the results for which are to be described in a future article) (not shown). (The sending and receiving transducers can also be manually adjusted for distance between them with future plans to automate this capability.) Fourth, the software has been modified for the latter and also for greater flexibility in user choice of signal processing parameters and images to be calculated (ability to choose up to 18 out of a possible 30 time- and frequency-domain parameters to form images from as the scan proceeds). Fifth, the option now exists to analyze specific time portions of the time-domain waveform as well as tuning/filtering for specific frequency ranges of interest in the frequency domain. Sixth, the on-line ability to compare waveforms, power spectral densities, and ultrasonic decay curves at different locations of samples is possible. Seventh, precise control of load via automated air pressure valve adjustment has been added. These additions are pertinent to the eventual commercialization/technology transfer of the scan system and allow for a great number of future research and materials characterization opportunities. Future modifications will be implemented as needed (such as to accommodate mildly curved components and for tuning for specific modes of ultrasound, and for non-contact operation).
REFERENCES


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