BENDING FRACTURE BEHAVIOR OF 3D-WOVEN SiC/SiC COMPOSITES WITH TRANSPERSION COOLING STRUCTURE CHARACTERIZED BY AE WAVELET ANALYSIS

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Abstract

The fracture behavior of the SiC/SiC composites was examined by 4-point bending tests. The AE signals detected during the tests were processed by AE wavelet analysis, which extracted the first and second major frequencies from continuous wavelet transform (CWT) diagram. Consequently, the two-frequency combination analysis can detect local frequency components and characterize a wavelet diagram well. A history of these major frequencies was correlated to initiation and propagation of micro-damages, such as cracks in SiC matrix, crack propagation along thickness and fiber buckling. Although other micro-damages, such as fiber breaks and pull-out of fiber, have not been correlated to the AE signals, the results suggest that the two-frequency combination analysis based on CWT has great potential to characterize the micro-damages of the composites.

Introduction

SiC/SiC composites have been studied in order to increase the temperature capability of jet engine components [1, 2]. An increase in the temperature capability would raise the gas temperature and reduce the need of air cooling, both of which would improve the efficiency of a jet engine. However, active cooling is still needed for the composites to preserve structural strength, since the temperature capability of the composites is lower than the gas temperature (e.g., 1600°C at turbine inlet). In order to develop a simple cooling structure with high efficiency, a transpiration-cooling structure was created in the composites by decreasing a volume fraction of SiC matrix, which was fabricated by four cycles of matrix densification process, “polymer impregnation and pyrolysis (PIP)” [3]. In order to investigate the PIP-cycle effect on the fracture mechanism, flexural loading and unloading test was carried out in a previous study [4]. However, the observed fracture behavior could not be correlated to the captured AE signals by examining AE parameters, such as amplitude and energy. In order to fully understand the bending fracture behavior and monitor micro-damage evolution under long-term evaluation (e.g., thermal cycling test, fatigue test), better correlations between captured AE signals and micro-damages are needed.

Many authors have studied frequency analysis based on frequency domain (FT) or time-frequency domain (WT) in order to investigate correlations between micro-damages and AE signals [5-9]. Many publications on the frequency analysis dealt with CFRPs or GFRPs, which were fabricated by materials of quite different properties. As the SiC/SiC composites are fabricated by materials with similar properties (e.g., chemical compositions and mechanical property), the frequency analysis with a capability to detect minor differences in AE waveforms is required. In recent studies, the discrete wavelet transform (DWT) has often been applied to characterize the AE waveform. The DWT decomposes a signal to several scale levels that correspond to frequency range. However, it appears that the scale level of the DWT may give only a rough estimation for characterizing the AE waveforms emitted from the SiC/SiC composites. Thus, this
study applied the continuous wavelet transform (CWT), which can analyze frequency at a finer scale, for the analysis of the AE waveforms of the composites.

The purpose of this study is to correlate the micro-damages of the SiC/SiC composites to the AE waveforms by analyzing the first and second major frequencies utilizing the continuous wavelet transform (CWT).

Materials and Experimental Procedures

Materials and Specimens

The SiC/SiC composites used in this study were made of Tyranno™ ZMI SiC (Si-C-O-Zr) fiber of Ube Industries, Ltd. and SiC matrix. The composites have the 3D-orthogonally woven structure as schematically shown in Fig. 1. The 3D-woven architecture consists of three layers of X bundles, four layers of Y bundles and Z bundles. Figure 2 shows a cross-sectional view of YZ plane along “aa”. The material composition near surface is also depicted in the figure. Tensile side is not flat because of the woven structure, which results in a notch-like geometry with stress concentration. The location of the notch-like geometry indicated by a gray arrow is called a “corner” in this paper.

First, a carbon interface layer derived from the chemical vapor infiltration (CVI) process was coated on the fibers, then the SiC matrix was infiltrated using methyl-trichlorosilane by a CVI process and from polycarbosilane by several cycles of PIP process. The porosity was controlled by varying the number of PIP cycles. The specimens were fabricated with 4 cycles of PIP. After the PIP treatments, the specimens were cut and coated with a thin layer of CVI-SiC. The volume fraction of fiber, matrix and porosity are 41%, 34% and 25%, respectively. The dimensions of the specimen were 50 mm length x 4 mm (approx.) width x 2 mm (approx.) thickness. The width corresponds to the size of two pitches of the textures.

Fig. 1. Schematic diagram of SiC/SiC weave (Fiber volume ratio: X:Y:Z=1:1:0.2, pitch=2 mm).

Experimental Procedures

Four-point bending tests were carried out with inner span of 10 mm and outer span of 30 mm at room temperature in air. Loading was applied by a universal testing machine (Instron, 5505) in Z-direction under a constant crosshead speed of 0.5 mm/min so that the tensile stress was applied
along X-direction. Deformation of the sample was measured parallel to the Z-axis by the displacement transducer and recorded by a PC-based data recorder (Kyowa, UCAM-500A).

During the test, AE signals were monitored by two wide-band AE sensors (Fuji Ceramics, M5W), which were attached on both ends of the sample. The AE signals were amplified 20 dB and the threshold level was set to the value of 3.16 mV at the input of the preamplifiers. The AE signals were processed by an AE analyzer (PAC, PCI-2 AE system) with AEWin™ operating software. Recorded AE waveforms were post-processed by the CWT using a commercial software (Weisang, Flex Pro Professional with an option of spectral analysis). Morlet function was chosen as the mother wavelet with non-dimensional frequency \( \omega = 8 \) [10].

![CWT diagrams of AE waveforms detected during flexural loading, showing characteristic contour patterns in time-frequency domain.](image)

**Fig. 3.** CWT diagrams of AE waveforms detected during flexural loading, showing characteristic contour patterns in time-frequency domain.

**Results and Discussion**

*The First and Second Major Frequencies in CWT Diagram*

AE waveforms acquired in this study showed characteristic CWT diagrams in the time-frequency domain. As an example, some characteristic CWT diagrams obtained from the bending test are shown in Fig. 3. Each diagram has two or more characteristic peak frequencies. The sensor used has a slightly higher response between 0.2 and 0.3 MHz and flat frequency response beyond 0.5 MHz. Thus, a frequency near 0.3 MHz is detected as the dominant frequency in
most of the diagrams. Second characteristic peak frequency of the CWT diagrams often appears below or above the dominant frequency of ~0.3 MHz. It appears that a combination of several peak frequencies can possibly characterize and classify the CWT diagram. In this study, the 1st and 2nd major frequencies were utilized for this attempt.

Figure 4 shows the CWT diagram of an AE waveform, including cross-sectional views taken along “aa” and “bb” on the CWT diagram. In the CWT diagram, a pair of the frequencies, “FRQ1” and “FRQ2” (white arrows) are the two characteristic frequencies of this WT coefficient contour. The cross-sectional view along line “aa” is a time history of WT coefficient at the frequency “FRQ1”. Ni and Iwamoto [9] proposed that a WT-time curve at a specified frequency can identify the occurrence time and duration of the micro-fracture mode of single carbon-fiber composites. The specified frequency was selected from the peak on FFT plot of the examined AE signal. However, the WT-curve cannot fully characterize the CWT diagram, because the WT-curve contains information about only one frequency component. The WT-frequency curve taken from “bb” line of the CWT diagram exhibits a distribution of WT coefficient indicating “FRQ1” to be the major frequency. This curve does show that the frequency peak is also located near “FRQ2”, but it fails to properly account for the intensity at “FRQ2”, even though this is shown clearly in the CWT diagram. Thus, either WT-time or WT-frequency curve cannot fully characterize the CWT diagram.

![Figure 4](image-url)  
Fig. 4. Wavelet diagram with cross sectional views, taken along lines “aa” and “bb”.

Figure 5(a) shows a CWT diagram projected into WT-frequency plots of the same AE waveform (Fig. 4) for a comparison. The projected WT-frequency curves lost time information, but the frequency and intensity information of two major peaks are preserved. Figure 5(b) shows FFT of the same signal where the first major frequency in the projected WT-frequency curve is recognized only as a minor frequency in the FFT envelope due to the time averaging effect. Thus, FFT analysis fails to detect important frequency information. FFT results also indicate the
highest peak at 0.11 MHz. This can easily be mistaken as a characteristic major frequency of the AE waveform and implies that FFT-based methods must be used with caution.

Fig. 5. The major and second major frequencies in projected WT-frequency curve of the waveform shown in Fig. 4 (WT-frequency curve not time-averaged.) (a) Projected WT-frequency curve, (b) FFT spectrum.

Comparison between Projected WT Curve and FFT Spectrum

Figure 6 shows another example where FFT fails to detect the highest peak of the AE signal. Figure 6(a), (b), (c) and (d) show the WT diagram, AE signal, projected WT-frequency curve and FFT spectrum, respectively. The 1st and 2nd major frequencies are indicated by red circles and connected by a solid line in Fig. 6(a), (c) and (d). Figure 6(a) and (b) show that a wavefront of the AE signal has the first major frequency at 0.56 MHz and the second major frequency at 0.32 MHz, and that the frequency component of ~0.3 MHz has long duration. It appears that these two frequencies of the wavefront are the characteristic frequencies of the AE signal. The two frequencies can be clearly recognized by the projected WT frequency curve in Fig. 6(c). On the other hand, the two peak frequencies of the FFT spectrum in Fig. 6(d) were 0.33 and 0.29 MHz.

Thus, FFT analysis failed to detect the highest characteristic frequency at 0.56 MHz. Since the frequency component of ~0.3 MHz showed long duration, the frequency component of ~0.3 MHz was estimated to have high intensity due to the time-averaging effect of FFT analysis. This example again indicated that FFT analysis is likely to fail to detect the higher frequency component of AE waveforms, because high frequency components often exhibit very short duration due to attenuation.

Figure 7 shows one more example, in which FFT failed to detect the characteristic frequency of the AE waveform, even though FFT analysis detected a relatively high frequency component. Figure 7(c) indicates that the AE waveform has the first major frequency at 0.34 MHz and the second major frequency at 0.81 MHz. Since the second major frequency component of ~0.8
MHz shows relatively long duration, the FFT recognizes the frequency component of 0.79 MHz as a major frequency. Thus, FFT analysis failed to detect the major characteristic frequency of 0.34 MHz.

![Wavelet Transform Frequency Curve and FFT Spectrum](image)

Fig. 6. Comparison between projected WT-frequency curve and FFT spectrum, showing that FFT fails to detect the highest peak of AE signal. (a) WT diagram, (b) AE signal, (c) projected WT-frequency curve, (d) FFT spectrum.

**Mechanical Behavior and Microscopic Damages Observed after Bending Test**

Figure 8 shows the stress-displacement curve during the bending test. The curve showed several small fluctuations before the stress reached the maximum value and a drop of stress afterward. The final bending fracture as indicated by white arrow in Fig. 8 was followed by a sharp decrease in stress.

Figure 9(a) shows SEM micrographs of the most damaged cross section of the specimen used in the bending test. The horizontal direction is the longitudinal (X-) direction of the specimen, and the tension side is at the bottom. It can be seen in Fig. 9(a) that fiber buckling was observed in the upper and lower X-fiber bundles. Fiber buckling in the upper bundle was accompanied with interlaminar cracks between X- and Y-fiber bundles. Figure 9 (a) also indicates that the upper pore is significantly larger with almost no infiltration of PIP-SiC matrix. Thus, the upper X-fiber bundle was not supported rigidly enough to sustain the compressive stress during bending. As a result, the fiber buckled in the upper fiber bundle adjacent to the large pore. The fiber
buckling in the lower fiber bundle can possibly be attributed to the severely damaged upper fiber bundle. Unfortunately, the complexities of the fiber buckling on the lower side make it difficult to interpret.

Fig. 7. Comparison between projected WT-frequency curve and FFT spectrum, showing that FFT fails to detect the highest peak of AE signal. (a) WT diagram, (b) AE signal, (c) projected WT-frequency curve, (d) FFT spectrum.

Fig. 8. Stress-displacement curve during the bending test.
Figure 9(b) and (d) show the 2 corners where the two fiber bundles intersect. It appears that several cracks near corner in Fig. 9(b) were propagated along thickness direction and reached the pore. A crack shown by white arrows in Fig. 9 (d) was propagated into Y-fiber bundle shown by the gray arrow. A crack initiated from the pore is also observed at the corner as shown by slashed arrow in Fig. 9(c).
AE Wavelet Analysis

Figure 10(a) and (b) show the history of the first and second major frequencies, respectively, of the first hit AE signals obtained during the bending test. Relative values of the WT coefficients of the major frequency components are indicated by the circle diameter. In Fig. 10(a), the 1st major frequency components are distributed in a limited frequency band of 250 to 500 kHz. This range corresponds to the high sensitivity range of the AE sensor-specimen combination, identified by the through-transmission experiment using a pair of M5W sensors with tone-bursts. The 2nd major frequencies, shown in Fig. 10(b), are more widely distributed and frequency components over 500 kHz and below 250 kHz were also observed.

Fig. 10. Frequencies in CWT diagrams. (a) The 1st major frequency. (b) The 2nd major frequency.

The 1st and 2nd major frequency components are shown together in Fig. 11 with connecting link between the two frequencies, since a combination of the two major frequencies can be considered to represent a contour pattern shown in Fig. 3. A caption above the figure described the bending fracture process, which had been observed in the loading/unloading test [4]. The corner crack was generated in the CVI-SiC and PIP-SiC matrices on the tension-side surface in a stress range between 50 MPa and 100 MPa, which grew along thickness direction above 100 MPa and resulted in the bending fracture. The PIP-SiC crack was observed above 100 MPa in the matrix-rich region near cavity and on compressive-side surface. Accordingly, in a stress range from 50 to 100 MPa, the cracks in the both CVI-SiC and PIP-SiC matrices were only observed.

The two frequency components of low WT coefficient with widely distributed frequency range in 50-1050 kHz were observed throughout the test. The signature of the AE signal can be considered to correlate with the initiation and propagation of the crack in both CVI-SiC and PIP-SiC matrices, since only the AE signals with the signature were observed in the stress range of 50 - 100 MPa while these micro-damages were developed. In the stress range above 100 MPa, the AE signals with relatively large WT coefficient were detected.

Figure 12 shows the detailed plot of two peak frequencies against time with the axis extended to the final bending fracture, which shows the AE signals obtained over a stress of about 100 MPa. In order to focus on the characteristics of the damage process, only the combinations of frequency components with relatively large WT were enhanced by linking two frequency symbols by a thick solid line. Black arrows indicate the stress fluctuations that were observed. The
Fig. 11. Two-frequency combination and displacement during the bending test. The description above the figure showed the fracture process obtained by loading and unloading test using a same-lot specimen in the previous study [4].

Fig. 12. Detailed plot of the two-frequency combination and stress vs. time during the bending test over a stress level of about 100 MPa.
open arrows shows where the large drop in stress occurred. It appears that the two peak frequency combinations with large WT formed several characteristic groups, which are indicated by dotted line, called group A, B and C. The AE signals, which were detected above 100 MPa, should correlate to the damages, such as the corner-crack propagation along thickness, fiber buckling and interlaminar crack, while exempting matrix cracks near surface or around pores.

The group A contains frequency components with widely distributed and large WT coefficients. The frequency combination A_1 has the 1st major frequency of 340 kHz and the 2nd major frequency of 810 kHz. The frequency combination A_1 may correspond to the initial severe damage in the tensile-side X-fiber bundle due to the corner crack propagation. The micro-damage corresponding to the A_1 was accompanied with a stress fluctuation indicated by a black arrow. It appears that the stress fluctuation can be caused by micro-damages that permit deformation of composites to relax the stress, for example, a rapid propagation of CVI-SiC matrix crack along thickness or width direction, pull-out of fiber and fiber breakage. Since the pull-out of fiber and fiber breakage can possibly occur after well developed CVI-SiC matrix crack in the fiber bundle, the A_1 may correspond to the rapid propagation of CVI-SiC matrix crack along thickness or width direction. Although the combinations A_1~A_3 contains relatively high frequency component near 800 kHz, the 1st major frequencies of A_1, A_2 and A_3 were different. This may suggest that the A_1~A_3 correlate to different micro-damages. From the microscopic observation as shown in Fig. 9(b) and (d), the corner crack in the fiber bundle finally reached upper Y-fiber bundle or pore, which would enhance fiber pullout in the X-fiber bundle, and then result in the stress fluctuation. Since the frequency combination A_4 with low frequency component of 79 kHz accompanied by the stress fluctuation, the frequency combination of A_4 may correlate to the fiber pullout.

After the corner crack reached a pore or Y-fiber bundle, the crack then developed into Y-fiber bundle shown by gray arrow in Fig. 9(d) or another crack may be initiated at a corner in the pore as indicated by slashed arrow in Fig. 9(c). Since these crack propagation did not accompany fiber cracks, the AE signals corresponding to these crack propagation may contain few high frequency components. Thus, "Group B" with few high frequency components indicated in Fig. 12 may correlate to the crack propagation into Y-fiber bundle and the crack initiation at a pore corner.

"Group C" in Fig. 12 contains very low frequency combinations with significantly large WT. The AE signals of C_1 and C_2 were detected at the final bending fracture with the sharp drop in stress indicated by the white arrow. It appears that the final bending fracture was accompanied with the fiber buckling, because stress cannot be held any further if such fiber buckling occurred. Thus, the AE signals of C_1 and C_2, which were detected at the final bending fracture, can correlate to the fiber buckling. The common signature of C_1 and C_2 is that the 1st major frequencies were detected at significantly low value of 69 or 89 kHz. Another AE signal C_3, of which the 1st major frequency was 69 kHz, was observed in advance of final bending fracture at 140 s accompanying a little drop in stress indicated by the black arrow. This may indicate that the fiber buckling was initiated then. The pattern recognition of the combination will further lead to correlate the AE signals to more microscopic damages, such as SiC fiber breakage and pull-out of fiber. Further study will be carried out to find the correlation by the two-frequency combination analysis extracted from CWT.
Conclusions

The wavelet analysis of AE signals based on the two-frequency combination extracted from CWT diagram was applied to investigate the correlation between AE signals and micro-damages in the SiC/SiC composites with transpiration structure and the following conclusions were obtained.

1) Major frequency components that characterize the contour pattern of the CWT diagram are extracted with a projected WT-frequency curve and used in two-frequency combination analysis.
2) The AE signal with low WT coefficient with widely distributed frequency corresponds to the crack in the CVI- and PIP-SiC matrices.
3) The crack propagation in longitudinal fiber bundle along thickness direction may be correlated to a group of two-frequency combinations, which has high, middle and low frequency components.
4) The fiber buckling damage was correlated to two-frequency combination, which contains low frequency component.

References