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# Modal acoustic emission of damage accumulation in C/C–SiC composites with different fiber architectures

Fabian Breede<sup>a,\*</sup>, Dietmar Koch<sup>a</sup>, Emmanuel Maillet<sup>b</sup>, Gregory N. Morscher<sup>b</sup>

<sup>a</sup>Institute of Structures and Design, German Aerospace Center (DLR), Pfaffenwaldring 38-40, 70569 Stuttgart, Germany <sup>b</sup>Mechanical Engineering Department, The University of Akron, Akron, OH 44325-3903, USA

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#### Abstract

The stress-dependent material behavior of carbon fiber reinforced ceramic matrix composites (C/C–SiC) was investigated with modal acoustic emission (AE) technique. AE observation is a promising health monitoring method which provides accurate location and identification of the recorded damage-related AE events. For a better understanding and identification of different damage mechanisms C/C–SiC composites with different fiber orientations were studied. Load/unload/reload tensile tests were performed and measurements were made over the entire stress range in order to determine the stress-dependence of acoustic activity for increasing damage states. In general it was found that in C/C–SiC composites a significant damage-related increase in AE energy was observed close to the ultimate tensile stress. Also, fiber architecture dependent differences during damage accumulation in terms of total number of AE events and their typical AE energy could be determined and correlated to different damage mechanisms.

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#### 1. Introduction

Carbon fiber reinforced ceramic matrix composite (CMC) materials are favorable for thermostructural components due to their excellent specific mechanical properties, high thermal conductivity and thermoshock resistance. Carbon fiber reinforced silicon carbide (C/SiC) composites have made it into several high-temperature aerospace applications, like nozzle extensions, combustion chamber components and thermal protection panels for re-entry [1,2]. The Institute of Structures and Design of the German Aerospace Center (DLR) has been continuously working on advanced C/C–SiC composites for future thermal protection and space propulsion systems [3,4]. The damage behavior of these materials must be known to allow a proper design of structural components. The somewhat non-linear stress–strain characteristic in CMCs when loaded can result from different energy dissipating effects, like

\*Corresponding author. Tel.: +49 711 6862 618; fax: +49 711 6862 227. *E-mail address:* fabian.breede@dlr.de (F. Breede). laminar- and inter-laminar matrix cracks, crack opening and closure, crack propagation and deflection, interfacial sliding (friction) and single fiber breaks or fiber bundle breaks. All of these effects create acoustic waves which can be digitized and analyzed using the modal acoustic emission (AE) technique. In the recent past the modal acoustic emission approach has been used to investigate the stress-dependent damage accumulation in several ceramic matrix composite materials and has proven to correlate individual AE events to specific damage sources [5–13].

In this study, the stress-dependent damage accumulation of DLR's C/C–SiC composites was evaluated for the first time applying the acoustic emission technique. Load/unload/reload hysteresis tensile tests were performed on C/C–SiC materials with a fiber orientation of  $\pm~15^{\circ},~\pm~45^{\circ}$  and 0/90°, respectively. Modal AE was used to record damage-related acoustic events and then to determine their location. Also an attempt was made to correlate waveform properties as well as the energy character of AE events to their probable physical damage sources.

#### 2. Material and testing procedure

In this study C/C-SiC composites<sup>1</sup> were tested [14,15]. Two C/C-SiC panels, one with a  $0/90^{\circ}$  and one with a  $+15^{\circ}$  fiber architecture, were manufactured using the liquid silicon infiltration (LSI) process (Table 1) [16]. The 0/90° composite was composed of 14 HTA<sup>2</sup> carbon fiber plies of plain weave fabrics (Style 98150). The  $+15^{\circ}$  composite consisted of 13 layers of plain weave fiber cloth which were fabricated by braiding technique<sup>3</sup> using T800HB<sup>4</sup> carbon fibers. Each fabric ply was pre-impregnated with a phenolic carbon precursor. In the first processing step the pre-impregnated fiber preforms were cured to a CFRP green body panel by warm pressing technique. During the following carbonization process the polymer matrix of the composite was converted to amorphous carbon forming a highly porous carbon/carbon preform. In this state carbon fibers were embedded in characteristic dense carbon matrix blocks. The carbon fibers were thus protected from degradation during the final liquid silicon infiltration. The silicon carbide matrix was then built up by the chemical reaction of the liquid silicon, that is infiltrated by capillary forces, and the accessible carbon. Here, silicon carbide is formed around each carbon/carbon block. Fig. 1 shows a typical micrograph of an as-received C/C-SiC composite with a 0/90° fiber architecture. The fiber volume fraction was about 60%. The test samples were cut from the 3 mm thick panels into dog-bone and straight-sided tensile test specimens. The length of the dog-bone specimens was 220 mm, the width at the end was 15 mm and the width of the gage section was 10 mm. The gage section length was 60 mm. The straightsided specimens had a length of 150 mm and a width of 10 mm. Load/unload/reload hysteresis tensile tests with 8-9 loops were performed with an electromechanical universal testing machine<sup>5</sup> at a rate of 2 kN/min. During the tests glass fiber reinforced epoxy tabs (2 mm thick) were used in the grip section (hydraulic grips). Strain measurements were made using a clip-on extensometer<sup>6</sup> with a gage length of 25.4 mm and a range of 2% strain.

Acoustic emission was monitored during the tests with a Fracture Wave Detector<sup>7</sup> acquisition system. Three wide-band transducers (Sensor Model B1025, 50–2000 kHz, 9.2 mm diameter) were mounted to the face of the tensile specimen (middle, +30 mm, -30 mm) to record acoustic emission events. The tensile test set-up and mounting of the measurement equipment is shown in Fig. 2. The AE sensors were connected to a pre-amplifier (Model PA-20) which was connected to the acquisition system (Model FM1, 4-channel, 20 MHz, 16 bit). The preamplifier was set at 12 dB for the

regular tensile test and 18 dB for the boron fiber breaks. The filter signal and the filter trigger were not amplified. Each AE signal consisted of 1024 points (including 256 points of pretrigger) which were digitized at a sampling rate of 10 MHz. The load and the strain were also recorded by the acquisition system. The post-test analysis was conducted with the Wave Explorer software and MS Excel.

The test procedure for each test was as follows. Before loading the tensile specimen boron fiber<sup>8</sup> breaks were performed on the face of the fully equipped tensile bar just outside of the top and bottom AE sensors. These manually-generated acoustic events were used to determine the speed of sound of the undamaged sample [5]. The specimens were then mechanically loaded, unloaded to zero stress and reloaded where the maximum stress level was progressively increased until failure. AE activity was continuously recorded during the tensile tests. In order to correlate acoustic emission activity to the composite's mechanical behavior, only AE events should be considered for the analysis which were generated between the top and the bottom sensors. Only AE events within the tensile bar gage length (60 mm) could be used for location determination and are related to the recorded strain measurements. In this study a typical 3-sensor configuration was used which allowed an easy sub-division into three general location regions according to which sensor was triggered first. Here "sensor 1 events" are referred to longitudinal locations  $x \ge +15$  mm, "sensor 2 events"  $x \le -15$  mm and "sensor 3 events" are between  $-15 \text{ mm} \le x \le +15 \text{ mm}$ , where x=0 is the middle of the gage length. In general an adequate filtering procedure has to be applied to discard false events, e.g. events that were triggered by electro-magnetic interferences.

Some of the test samples were observed by scanning electron microscopy (SEM) in order to evaluate if typical damage mechanisms are detectable within the composite comparing "as-received" and loaded (damaged) specimens. The SEM specimens were cut from the tensile test bars in longitudinal direction and polished.

#### 3. Results and discussion

#### 3.1. Speed of sound determination

In order to determine the location of the observed AE events the velocity of the extensional wave component  $C_e$  must be known. The theoretical speed of sound of an extensional wave  $C_{e,theo}$  can be calculated from the classical plate theory [17]

$$C_{e,theo} = \sqrt{\frac{E}{\rho \cdot (1 - \nu^2)}} \approx \sqrt{\frac{E}{\rho}}$$
 (1)

 $C_{e,theo}$  is a function of the longitudinal elastic modulus E, the material density  $\rho$  and Poisson's ratio  $\nu$ , which only has a minor influence, that allows a simplification of the above equation.

<sup>&</sup>lt;sup>1</sup>Manufactured at the Institute of Structure and Design (DLR), Stuttgart, Germany.

<sup>&</sup>lt;sup>2</sup>Toho Tenax Co. Ltd., Japan.

<sup>&</sup>lt;sup>3</sup>Fiber preforms fabricated by the Institute of Aircraft Design, The University of Stuttgart, Germany.

<sup>&</sup>lt;sup>4</sup>Toray Carbon Fibers Europe S.A. (CFE), France.

<sup>&</sup>lt;sup>5</sup>Criterion Model C43, MTS Systems Corporation, Eden Prairie, MN, USA.

<sup>&</sup>lt;sup>6</sup>Model 632.27E-30, MTS Systems Corporation.

<sup>&</sup>lt;sup>7</sup>Digital Wave Corporation, Centennial, CO, USA.

<sup>&</sup>lt;sup>8</sup>Boron fiber source – Specialty Materials, Lowell, MA, USA.

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