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ORIGINAL ARTICLE

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# Evaluation of dynamic modulus measurement for C/C-SiC composites at different temperatures

**Sandrine Hönig**[1] | **Dietmar Koch**[1] | **Steffen Weber**[2] | **Simon Etzold**[3] | **Thorsten Tonnesen**[3] | **Rainer Telle**[3] | **Nicolas Traon**[4]

[1]German Aerospace Center (DLR): Institute of Structures and Design (BT), Stuttgart, Germany

[2]Krebs & Riedel Schleifscheibenfabrik GmbH & Co. KG, Bad Karlshafen, Germany

[3]RWTH Aachen University, Institute of Mineral Engineering, Aachen, Germany

[4]European Patent Office (EPO), The Hague, Netherland

**Correspondence**
Sandrine Hönig, German Aerospace Center (DLR): Institute of Structures and Design (BT), Stuttgart, Germany.
Email: sandrine.hoenig@dlr.de

**Abstract**

The determination of elastic properties at application temperature is fundamental for the design of fibre reinforced ceramic composite components. An attractive method to characterize the flexural modulus at room and high temperature under specific atmosphere is the nondestructive Resonant Frequency Damping Analysis (RFDA). The objective of this paper was to evaluate and validate the modulus measurement via RFDA for orthotropic C/C-SiC composites at the application temperature. At room temperature flexural moduli of C/C-SiC with 0/90° reinforcement were measured under quasi-static 4-point bending loads and compared with dynamic moduli measured via RFDA longitudinally to fibre direction. The dynamic modulus of C/C-SiC was then measured via RFDA up to 1250°C under flowing inert gas and showed an increase with temperature which fitted with literature values. The measured fundamental frequencies were finally compared to those resulting from numerical modal analyses. Dynamic and quasi-static flexural moduli are comparable and the numerical analyses proved that bending modes are correctly modeled by means of dynamic modulus measured via RFDA. The nondestructive RFDA as well as the numerical modeling approach are suitable for evaluation of C/C-SiC and may be transferred to other fibre reinforced ceramic composite materials.

**KEYWORDS**

bending, ceramic matrix composites, finite element analysis, flexural modulus, modeling/model, Resonant Frequency Damping Analysis

## 1 | INTRODUCTION

The design of fiber reinforced ceramic composite materials requires an accurate assessment of thermo-mechanical properties in the temperature range of application. Conventionally, tensile tests and bending tests are coupled at low and elevated temperature to determine the elastic behavior of the material under quasi-static conditions and to estimate the flexural modulus of the tested sample. The comparison of elastic properties assessed through destructive and nondestructive measurement techniques as well as micro-CT based computational modeling was already studied by Weglewsky et al. on metal-ceramic composites.[1] This study could prove both discrepancies between the results obtained through destructive (3-point bending test) and nondestructive (amongst others Resonant Frequency Damping Analysis [RFDA]) techniques of measurement, and the similar elastic properties between RFDA measurements and the micro-CT based computational modeling.

Since the 1950s, dynamic procedures for flexural modulus assessment have been appreciated as nondestructive approach of elastic property determination.[2,3] The measurement techniques are based on the treatment of the acoustic signal emitted by the specimen due to slight mechanical excitation. The emitted resonant frequency strongly depends on the stiffness of the material. According to the nature of sample excitation or acoustic signal acquisition, different techniques of measurements can provide a trustful assessment of elastic properties. RFDA is particularly valued for very fast acquisition and processing and moreover for the accurate estimation of flexural modulus, shear modulus, and Poisson's ratio.[4,5] This measurement technique is based on the treatment of the resulting acoustic signal after mechanical excitation of the sample with the help of a light mechanical impulse.[5,6]

In order to enhance numerical modeling via Finite Element Method (FEM), the consideration of the flexural modulus of the developed material sample at application temperature is even more profitable. Therefore, RFDA turns out to be a beneficial experimental procedure as both, room temperature and high temperature measurements can be performed under specific atmospheric conditions.[7]

Ceramic matrix composites (CMC) combine the advantages of high thermal shock resistance as well as damage tolerance tailored by the fibre architecture and an appropriate fibre-matrix interface. Their lower density compared to metallic alloy used for high temperature application is also a key advantage. Since the middle 1980s, DLR has been developing a cost-efficient production route for CMC materials based on Liquid Silicon infiltration (LSI)[8,9]The resulting C/C-SiC composites allow thin-walled, complex components for example, for aerospace applications as well as thick plates for example, for brake discs.[10–15]

Conventionally the flexural modulus of the C/C-SiC materials is experimentally characterized via quasi-static bending tests. At high temperatures the mechanical tests must be performed in inert atmosphere in order to prevent oxidation[16] Some typical flexural properties of C/C-SiC materials are given in.[8,9,17,18]Mechanical testing requires at least 5 specimens and application of strain gauges for measuring flexural modulus according to DIN EN standard. At high temperatures—the main application field for C/C-SiC materials—the complex flexural modulus estimation can be done with the help of high temperature strain gauges, laser or mechanical extensometer and cross head displacement evaluation. Therefore publications about flexural modulus characterization via quasi-static method at high temperatures are rare.[8,16,19–21]

Different to quasi-static mechanical testing the RFDA method by IMCE (Integrated Material Control Engineering, Belgium) applied at the Institute of Mineral Engineering of the RWTH Aachen is of great advantage for analyzing the flexural modulus at high temperatures. With densities of about 1.9-2.0 g/cm$^3$ C/C-SiC composites[8] are suitable for the RFDA method as these densities allow a local impulse excitation with low damping and without a global shift of the sample. Maile et al. measured the flexural modulus of one C/C-SiC material (0/90°) via RFDA at room temperature and reported only a graphical information[18] The present work is filling this gap with detailed RFDA evaluation at room and high temperature.

Wanner et al. determined the flexural modulus of reinforced C/C composites at different temperatures via resonant beam technique[22] Nevertheless, the correlation between the material properties and the eigenfrequency was not reported. With the help of numerical analyses, this dependency is quantified in this paper.

The objective of this paper is to evaluate and validate the flexural modulus measurement method via RFDA for orthotropic C/C-SiC composites at the application temperature. Flexural moduli under 4-point bending load of C/C-SiC were characterized at room temperature via RFDA and via quasi-static testing. The dynamic modulus of the C/C-SiC material was also characterized up to 1250°C via RFDA in argon atmosphere and compared to literature values. The first bending mode was then numerically analyzed and the frequencies resulting from finite element analyses were compared to those measured experimentally via the RFDA method.

For simplification reason in this paper the flexural modulus is called modulus.

## 2 | MATERIALS, EXPERIMENTAL METHODS, AND NUMERICAL ANALYSIS

### 2.1 | C/C-SiC material

For the manufacturing of C/C-SiC material, stacked sized Tenax® HTA prepregs (type 40/200 tex, 3k, style 462 Kö 2/2, woven fabrics 0/90° of carbon fibres with a JK60 phenolic resin) were cured under vacuum and consolidated in an autoclave. The green body was then pyrolysed under inert atmosphere; a C/C porous preform was created. Finally LSI led to the orthotropic C/C-SiC composite. A typical feature of the microstructure is a block formation of C/C surrounded by SiSiC layers. This allows block fibre pull-out effects under mechanical loading, and thus a high damage tolerance.

All specimens characterized via RFDA and quasi-static bending testing method were machined from one single plate called PH2110. At room temperature fewer specimen are required for RFDA compared to the quasi-static testing: since the standard test method for dynamic modulus ASTM C1548-02 does not provide any information about the minimum number samples, the authors decided to test 3 specimens as specified by the standard DIN EN 843-2 for monolithic ceramics. The specimens 1-3 were firstly experimentally analyzed via nondestructive RFDA method and then tested in quasi-static 4-point bending mode at room temperature. The specimens 4 and 6 were analyzed via RFDA technique up to temperature of respectively 900°C and 1250°C in argon atmosphere. At high temperature the testing methodology is evaluated on 2 different specimen geometries, no statistical evaluation will be done. Open porosities and densities of C/C-SiC were measured by Archimedes method according to DIN EN 993-1. The specimen dimensions, weights, open porosities and densities are listed in Table 1. The density values listed in this table are used for the estimation of the dynamic moduli via RFDA as well as material input data for the numerical analyses.

The coefficient of thermal expansion (CTE) of the sample, required for the RFDA at high temperatures, was measured along fiber direction following the standard DIN EN 1159-1. For numerical analysis the transverse CTE is also required; values at 100°C and 1500°C are found in[9] The longitudinal and transverse CTE values are described within the numerical model description chapter (Figure 3).

## 2.2 | Experimental methods

### 2.2.1 | RFDA at room temperature

In a first part of the study, RFDA was performed at room temperature to measure the flexural modulus of C/C-SiC materials. Three samples were measured to estimate a representative mean value or possible scatter of the moduli. The dynamic modulus of each sample was determined through RFDA according to ASTM C1548-02 with help of an IMCE (Belgium) testing device (RFDA system 23).

Once the specimen was positioned on the sample holder as shown on Figure 1, it was excited by a slight mechanical impulse in the middle of its lower surface. The acoustic response was sensed by a microphone and processed according to the frequency and attenuation rate detection. A Fast Fourier Transformation was carried out by the corresponding software and a corresponding graph was created based on the amplitude of the measured frequencies. Every frequency that exceeds an adjustable threshold value is taken into account for the calculation of the elastic properties.

**TABLE 1** Sample properties, open porosity and densities of the characterised C/C-SiC

| Plate name | Specimen number | Dimensions Length [mm] | Width [mm] | Thickness [mm] | Weight [g] | Open porosity [Vol.%] | Density [g/cm³] |
|---|---|---|---|---|---|---|---|
| PH2110 | 1 | 150.1 | 24.8 | 3.0 | 20.9[c] | 0.9[a] | 1.90[a] |
| | 2 | | 25.1 | 3.1 | 21.1[c] | | 1.91[a] |
| | 3 | | 25.2 | 3.1 | 21.3[c] | | 1.91[a] |
| | 4 | | 25.0 | 3.1 | 21.0[d] | — | 1.81[b] |
| | 6 | 76.4 | 33.2 | 3.1 | 14.2[d] | — | 1.81[b] |

[a]Open porosity and bulk density measured by Archimedes method (DIN EN 993-1).
[b]Geometric density (room temperature weight over volume).
[c]Dried weight.
[d]Weight measured at room temperature and room humidity.

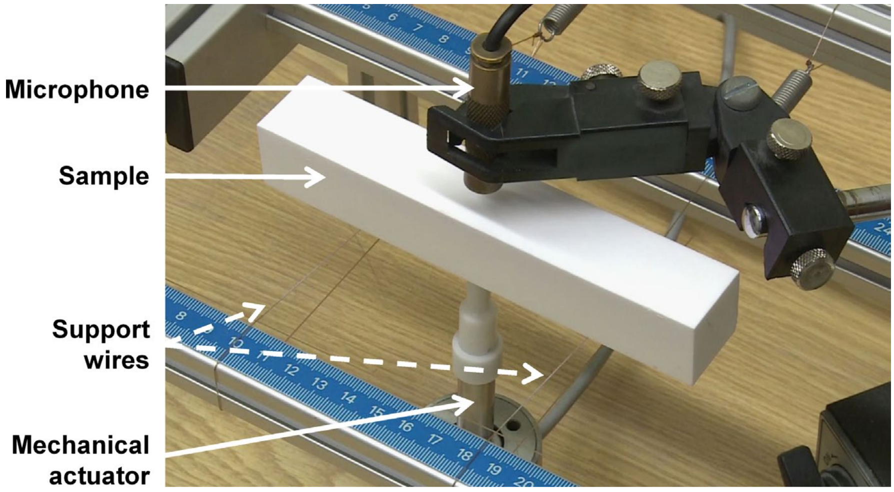


**FIGURE 1** RFDA facility for room temperature investigation. RFDA, Resonant Frequency Damping Analysis

For samples with square cross-sections excited at the flexural mode of vibration, the dynamic flexural modulus ($E$) is calculated using the following equation:

$$E = 0.9465 \cdot \left( \frac{m \cdot f_f^2}{b} \right) \cdot \left( \frac{l}{h} \right)^3 \cdot T \tag{1}$$

The parameter $m$ is the weight of the specimen (g), $f_f$ is the flexural resonant frequency of the specimen (Hz), $h$ is the specimen thickness in excitation direction, $l$ is the specimen length, $b$ is the specimen width (mm) and $T$ is a geometrical correction factor that depends on the aspect ratio of the specimen (depending on $l$ and $h$) and on the Poisson's ratio (supposed to equal to 0.20 in this study for each specimen). Three measurements were carried out on each sample to control the reproducibility of the measurement and to provide representative mean value of modulus.

For comparison of the results, the geometric setup was kept constant. The node distance was set depending on the sample length (0.224 × length of the edge of the sample) to impose the mechanical boundary condition for unaffected assessment of the flexural resonant frequency. The distance between the microphone and the samples was kept to 5 mm. The excitation was delivered with help of an automatic hammer. To avoid any undesirable effects and to minimize eventual nonlinearity acoustic response, the intensity of the impulse excitation was kept constant[23]

### 2.2.2 | RFDA at high temperatures

In a second part of the study, RFDA measurements were carried out at elevated temperatures up to 1250°C according to the same standard ASTM C1548-02 with a testing device also developed by IMCE (Belgium), RFDA HT1750 illustrated on Figure 2. This device combines the measurement principle previously described under room temperature conditions with a heating furnace up to maximum temperature 1750°C. RFDA measurements were performed on the C/C-SiC materials up to 1250°C with a heating and cooling rate of 5 K/min. In order to achieve a suitable temperature profile, the measurements were performed each 30 seconds. In order to limit the sample damaging through oxidation during the test, the experiment was carried out while the furnace chamber was purged with argon.

The dynamic modulus was corrected according to the following equation by taking into consideration the CTE:

$$E_T = E_{RT} \left( \frac{f_T}{f_{RT}} \right)^2 \cdot \left( \frac{1}{1 + \alpha . \Delta T} \right) \tag{2}$$

Parameters $E_T$ and $E_{RT}$ respectively represent the flexural modulus of the material at temperature $T$ and room temperature $RT$ (GPa). Parameters $f_T$ and $f_{RT}$ are the flexural resonant frequencies of the material at temperature $T$ and room temperature $RT$ (Hz), respectively. The coefficient $\alpha$ corresponds to the CTE of the specimen ($K^{-1}$) in the temperature range $\Delta T = T - RT$ (K).

### 2.2.3 | Four-point bending test at room temperature

Four-point bending tests according to DIN EN 658-3 were carried out at room temperature on C/C-SiC samples in a Zwick 1494 testing facility. For the quasi-static tests, longitudinal strain gauges were glued on the middle of the tensile loaded face of each sample. The test provides the flexural modulus, the flexural strength and the flexural failure strain for C/C-SiC specimen 1-3. Specimen dimensions are listed in Table 1.

## 2.3 | Numerical modal analysis: model description

All the orthotropic C/C-SiC samples experimentally characterized via RFDA were numerically modeled with the help of the commercial FEM software ANSYS. The first bending mode of each sample was studied via a modal analysis. The resulting eigenfrequency was then compared to the frequency measured experimentally via RFDA method.

The samples were considered as perfect rectangular cuboid and built up on the measured averaged dimensions listed in Table 1**.** The blocks were homogeneously meshed with quadratic 20-node elements. The mesh size was chosen so that the thickness of each sample was divided into 4 elements; this was precise enough for modeling the first bending mode. The z direction is defined as the out-of-plane direction, the x direction is the length direction of the sample and the y direction is the width direction.

Contrary to the RFDA method, an inverse approach has to be conducted here: the in-plane dynamic modulus determined via RFDA Equation (1) was used as input for the numerical analysis. The fundamental frequencies of the first bending modes were then numerically estimated via modal analysis and compared to experimental frequencies.

The C/C-SiC material was defined as linear elastic with orthotropic properties. The density ρ, the longitudinal dynamic moduli $E_x$ and $E_y$ (RFDA) as well as the in-plane longitudinal CTE were experimentally measured in this study. The unknown mechanical material properties at room temperature were defined via literature values[17] or hypothetical values at elevated temperature.

For both C/C-SiC samples modeled at elevated temperatures, the modulus evaluated via RFDA (considering thermal correction via equation [2]) as well as the temperature dependent CTEs were used as material input properties. The transverse CTE at test temperature is

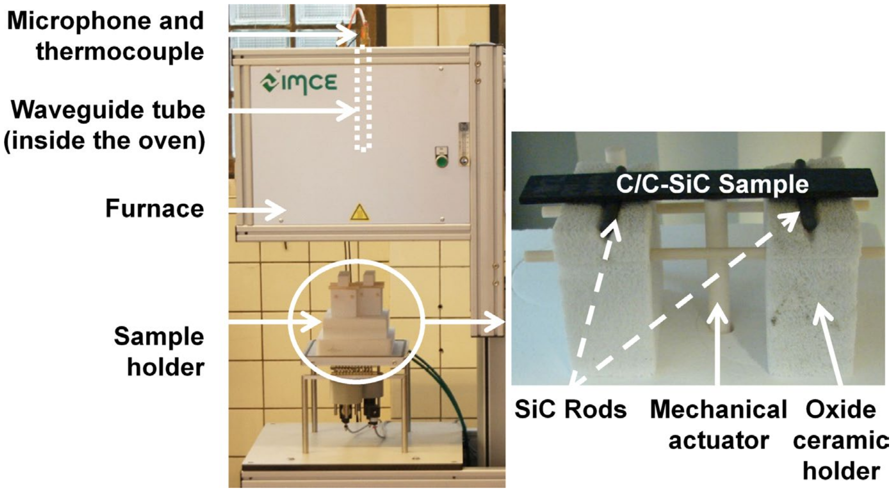


**FIGURE 2** RFDA facility for high temperature investigation. RFDA, Resonant Frequency Damping Analysis

also required for defining the numerical material model; it is calculated via linear extrapolation based on literature values. Krenkel[9] gives values of the out-of-plane CTE at 100°C and 1500°C of respectively 2.5.10$^{-6}$/C and 6.5.10$^{-6}$/C. Between these temperature points and for room temperature, a linear extrapolation is done. Out of the given temperature range, ANSYS sets the nearest CTE as constant value. The thermal variations of the other material properties from literature[17] were considered constant over the temperature. The material properties used in the FEM models are described in Table 2 and Figure 3.

Static structural analyses were firstly performed at room temperature and in 50 K steps between 200°C and 900°C or 1250°C for specimen 4 or 6 respectively, so that the thermal expansion was calculated. The applied load was a constant temperature over the whole sample with free mechanical boundary conditions. Modal analyses were then performed on each thermal loaded models, the frequency of the first longitudinal bending mode was calculated.

A parameter correlation study and a sensitivity analysis based on specimen 1 were then conducted. The objective was firstly to identify which material property mainly influences the fundamental frequency and secondly to quantify the level of this influence. This helps to evaluate the transfer of the isotropic based RFDA approach to the orthotropic C/C-SiC composites. Hajianmaleki showed in[24] that no simple analytical approach is available for dynamic analysis of laminate beams. On the contrary, numerical modeling can provide a quick overview of the main influencing material parameters.

# 3 | RESULTS AND DISCUSSION

## 3.1 | Experimental investigations

### 3.1.1 | Investigation at room temperature

The dynamic and quasi-static flexural moduli determined experimentally at room temperature for the C/C-SiC samples 1-3 are presented on Figure 4.

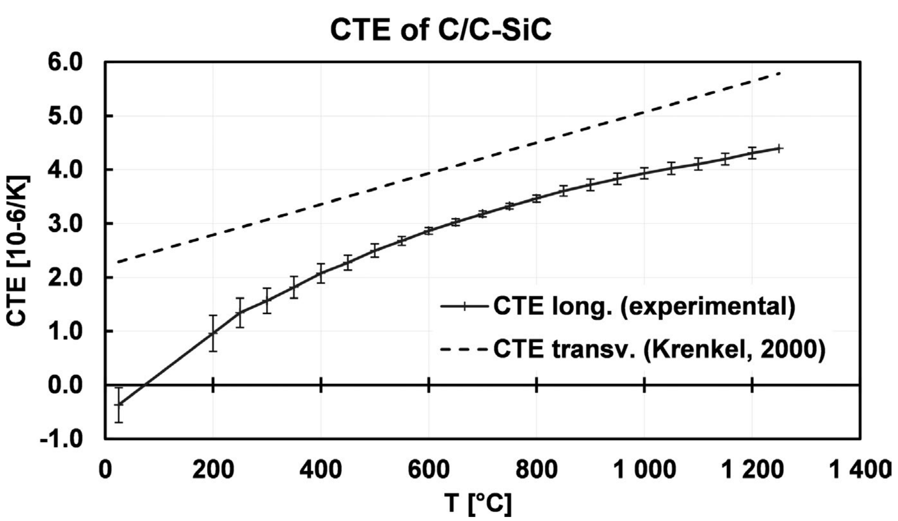


**FIGURE 3** CTE of C/C-SiC used in the FEM models at elevated temperatures. CTE, coefficient of thermal expansion

**TABLE 2** C/C-SiC material properties used in the FEM model (x, y, and z directions correspond respectively to the length, width and thickness directions of the flexural sample)

| C/C-SiC sample | ρ [g/cm³] | $E_x = E_y$ [GPa] | $E_z$ [GPa] | $G_{xy}$ [GPa] | $G_{xz}$ [GPa] | $G_{yz}$ [GPa] | $\nu_{xy}$ [-] | $\nu_{xz}$ [-] | $\nu_{yz}$ [-] |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 1.90[a] | RFDA results | 20[c] | 5.1[c] | 6.6[c] | 6.6[c] | 0.01[c] | 0.1[c] | 0.1[c] |
| 2 | 1.91[a] | | | | | | | | |
| 3 | 1.91[a] | | | | | | | | |
| 4 | 1.81[b] | | | | | | | | |
| 6 | 1.81[b] | | | | | | | | |

[a]Bulk density measured by Archimedes method (DIN EN 993-1).
[b]Geometric density (room temperature weight over volume).
[c]From literature.[17]

The average quasi-static modulus is 63 GPa with a standard deviation of 3 GPa. This is in good agreement with the longitudinal modulus value of 65 GPa given in the recent literature[17] but higher than the modulus of 47 GPa given in.[8,9] All samples fail in tensile fracture mode. A typical stress-strain curve for the 4-point bending tests of the C/C-SiC material as well as a picture of the specimens after bending tests are shown on Figure 5. An almost linear-elastic behavior up to failure can be observed, which allows to use a linear elastic model in the numerical analyses.

The average dynamic modulus (RFDA) is 67 GPa with a standard deviation of 3 GPa. The dynamic modulus follows the same evolution than that of the quasi-static modulus. However the dynamic modulus always exceeds slightly the quasi-static modulus. Microcracks in the materials result in a reduced modulus when measured quasi-statically due to shear lag effects. The average difference between the dynamic and quasi-static modulus of each sample is 4 GPa which is close to the standard deviation of 3 GPa for both, quasi-static and dynamic modulus. Therefore the modulus values evaluated via quasi-static and RFDA technique are considered as comparable.

### 3.1.2 | Investigations up to 1250°C

Figure 6 shows the moduli measured experimentally at different temperatures via RFDA and 4-point bending methods for all C/C-SiC specimens of this study as well as moduli measured quasi-statically in literature.[8,9,21]

The dynamic elastic behavior of the specimen 4 and 6 are measured at temperatures respectively up to 900°C and 1250°C under argon flushing atmosphere. Since slight oxidation happens at the end of the heating cycle which alters the sample behavior, only the behavior during heating phase is discussed here. For a better visualisation in Figure 6 only the averaged modulus is plotted; the scatter is not shown.

Starting from dynamic modulus values at room temperature of 59 GPa and 64 GPa for specimen 4 and 6 respectively, which are in agreement with the averaged dynamic modulus 67 GPa of specimen 1-3, the modulus increases with

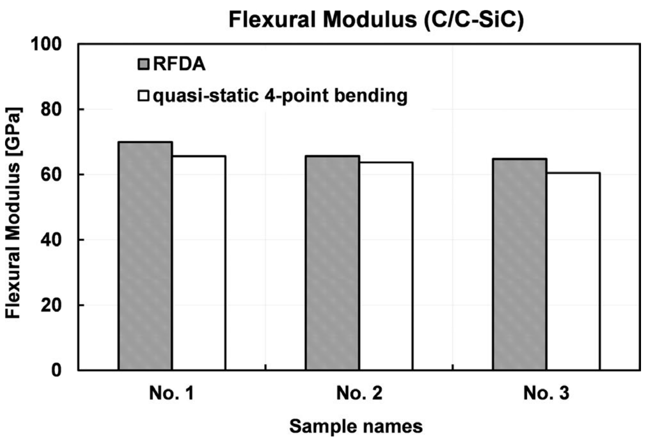


**FIGURE 4** Flexural moduli of C/C-SiC samples measured via RFDA and 4-point bending tests at room temperature. RFDA, Resonant Frequency Damping Analysis

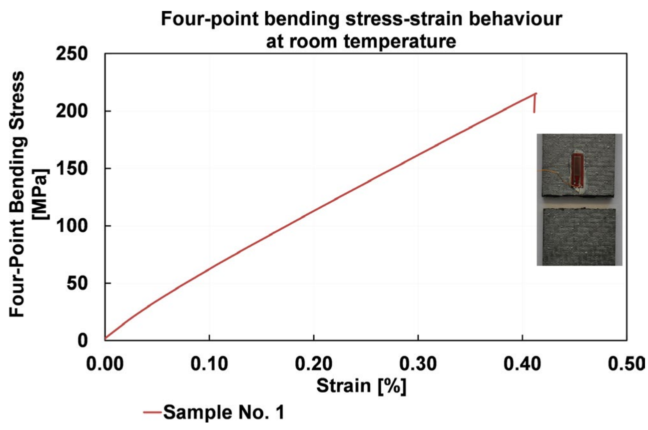


**FIGURE 5** Four-point bending test of C/C-SiC specimen at room temperature: typical stress-strain behavior and typical fracture surface

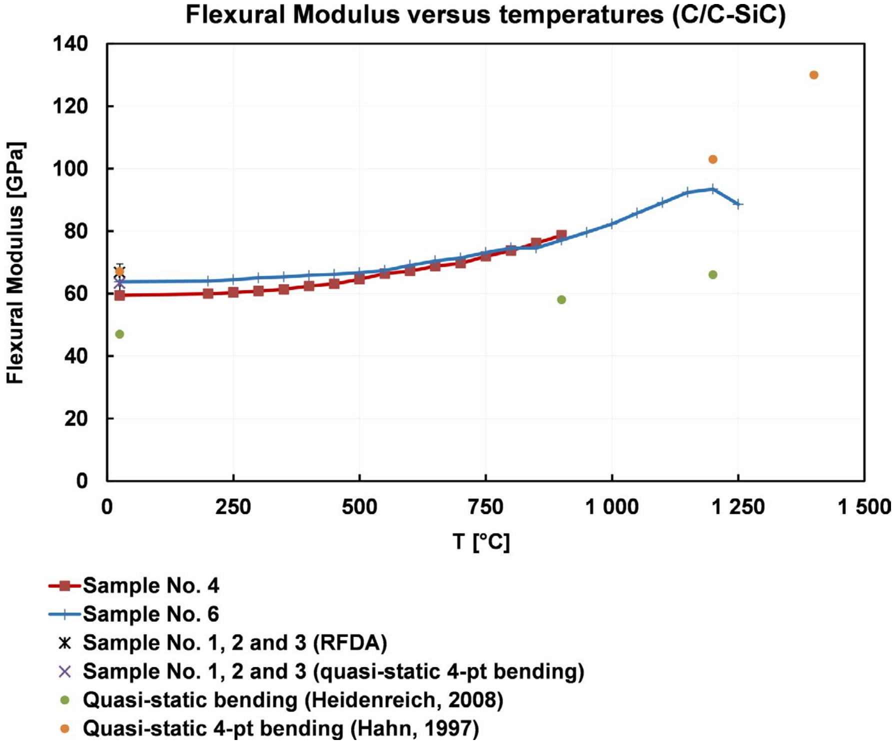


**FIGURE 6** Young's moduli of C/C-SiC samples measured via RFDA and quasi-static bending tests at different temperatures. RFDA, Resonant Frequency Damping Analysis

elevated temperature up to around 1200°C to achieve a value of 90 GPa. Since the measurement of sample 4 was only performed up to 900°C, the modulus only reaches 79 GPa. From 1200°C up to 1250°C, the modulus decreases. Indeed, above 1200°C the virtually inert atmosphere through argon purging (argon gas insertion without vacuum support) was not sufficient to prevent the nonoxide C/C-SiC material from oxidation. Figure 7 shows the cut edge microstructure of specimen 6 after the RFDA measurement up to 1250°C. Single fibre degradation as well as bundle degradation due to oxidation can be observed, leading to the modulus decrease.

The averaged quasi-static and dynamic moduli of all the C/C-SiC samples measured at room temperature in this study, 63 ± 3 GPa and 65 ± 4 GPa respectively, are closed. This indicates that a good reproducibility of the modulus measurement at room temperature with both methods is guaranteed, even though the samples present different geometries (see Table 1). Geometry variations between the samples are considered within the calculation of the resulting Young's modulus values.

As the dynamic modulus is approximately identical to the quasi-static 4-point flexural modulus values at room temperature it can be assumed, that the quasi-static modulus will also increase with rising temperature under inert atmosphere.

The moduli from literature[8,9] are 12-20 GPa lower than those measured in this study (Figure 6), whereas the moduli measured in[21] approaches the moduli of this study. It should be noticed, that in references[8,9] no information about the testing procedure is given. In[21] Hahn characterised the modulus with 4-point bending method in vacuum, which is comparable

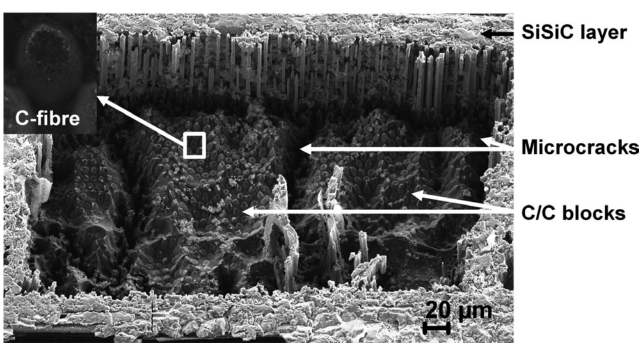


**FIGURE 7** Microstructure of cut edge of C/C-SiC specimen 6 after high temperature RFDA measurement up to 1250°C. RFDA, Resonant Frequency Damping Analysis

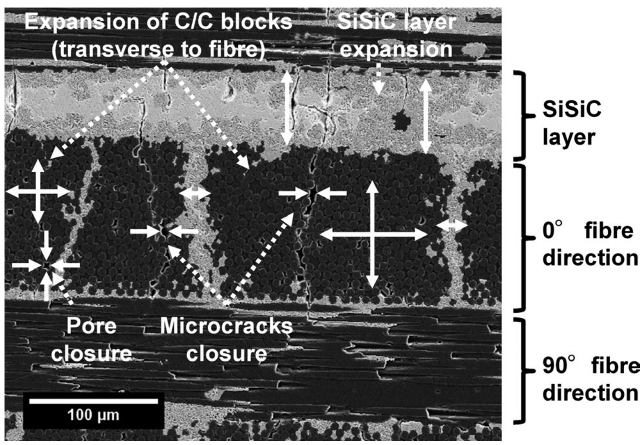


**FIGURE 8** Microstructure modifications of C/C-SiC during thermal expansion

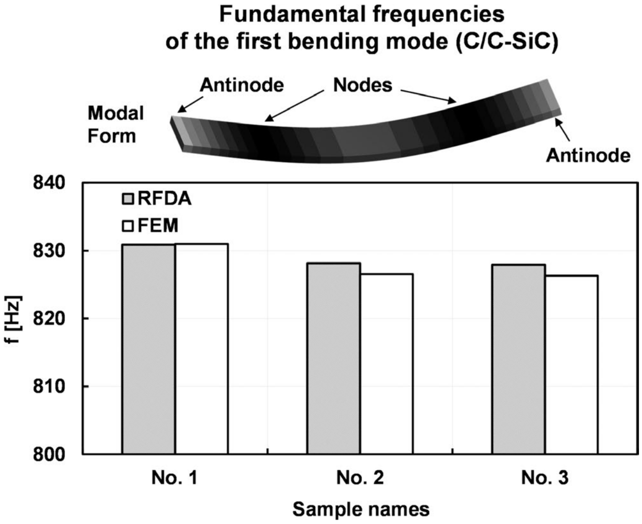


**FIGURE 9** C/C-SiC: fundamental frequencies estimated via RFDA technique and numerical modal analyses at room temperature. RFDA, Resonant Frequency Damping Analysis

to the present experiment. Nevertheless, as measured here a comparable increase of the flexural modulus along the temperature is observed for C/C-SiC material in.[8,9]

Maier[16] measured the tensile and compressive moduli of C/C-SiC called P2 which is comparable to the material tested in this work at temperatures up to 1600°C in vacuum. The results proved that the tensile modulus is constant for temperatures up to about 1350°C. Maier measured also an increase in the compressive modulus up to temperatures of about 1400°C.

The evolution of the bending, tensile, and compressive stiffness behavior along the temperature is driven by the microstructure. The transverse CTE of HTA carbon fibres increases from $0.10^{-6}$/C at room temperature to $7.10^{-6}$/C at 1000°C whereas the longitudinal CTE increases more slowly from $0.10^{-6}$/C at room temperature to $1.10^{-6}$/C at 1000°C.[25] The CTE of SiC ($4.10^{-6}$/C) is also higher than the CTE longitudinal to the fibres.[26] Therefore during thermal expansion under inert atmosphere, the SiSiC layers as well as the C/C blocks in transverse direction expand faster and more than the C/C blocks in longitudinal direction. The microcracks inside the SiSiC layers and C/C-blocks and transversal to fibre direction closed then during temperature increase, the thermal expansion is illustrated on Figure 8. This is confirmed by Wanner et al. who observed increase in flexural modulus and closure of microcracks in bidirectional carbon/carbon composites inside C/C-blocks along the temperature.[22] The compressive and flexural properties are dependent on the fibre Young's modulus and on the matrix stiffness. Due to pore closing during thermal expansion, the matrix stiffness increases along the temperature up to about 1350°C, whereas the fiber modulus remains constant. It results in an increase of the compressive and flexural moduli of the C/C-SiC materials. Up to a temperature of approximately 1413°C the residual silicon is melting and further microstructural mechanisms occur inducing different mechanical behaviour.[16]

## 3.2 | Numerical modal analysis

### 3.2.1 | Numerical modal analysis performed at room temperature

For each sample analyzed via RFDA, the fundamental frequency of the first bending mode was numerically analyzed.

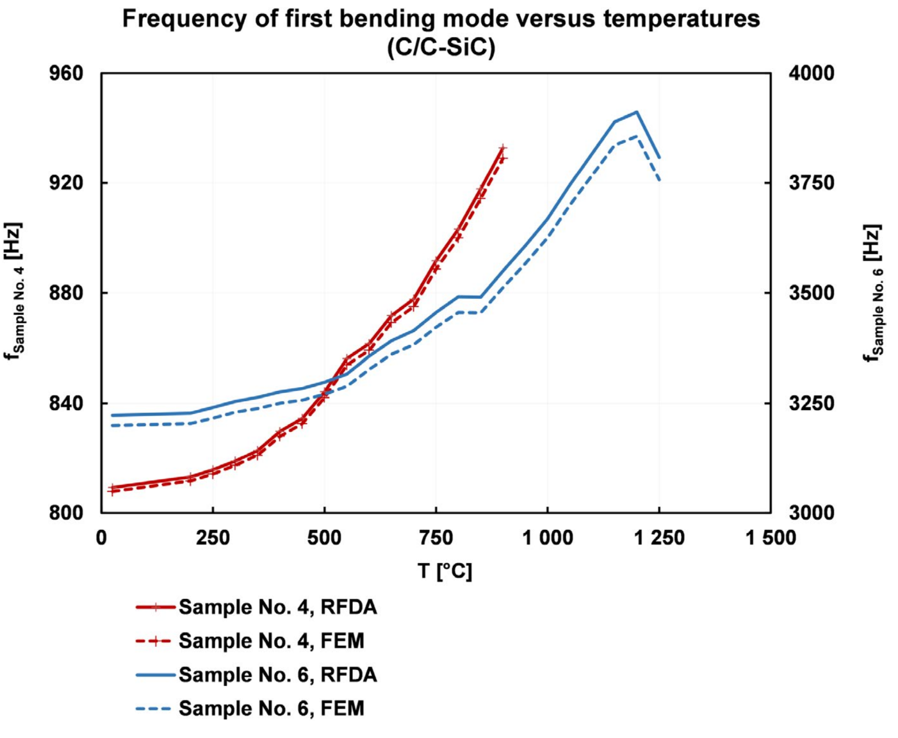


**FIGURE 10** C/C-SiC: fundamental frequencies characterised via RFDA technique and resulting from numerical modal analyses along the temperature. RFDA, Resonant Frequency Damping Analysis

As the C/C-SiC samples are relatively thin (factor length/thickness of 50), the nodes and antinodes of the first bending modes are well defined. The representative modal form of the C/C-SiC sample and the fundamental frequencies determined experimentally and numerically at room temperature are shown on Figure 9.

For the numerical model of C/C-SiC, experimental orthotropic material properties (Table 2) were used. The difference between the experimental and numerical fundamental frequencies reaches a maximal value for the sample 2 and corresponds to 0.2% of the averaged experimental dynamic frequencies for this sample. Therefore the Poisson's ratio of 0.2 used in Equation (1) for the RFDA method has a negligible influence on the results and the presented FEM model at room temperature is realistic for orthotropic C/C-SiC materials.

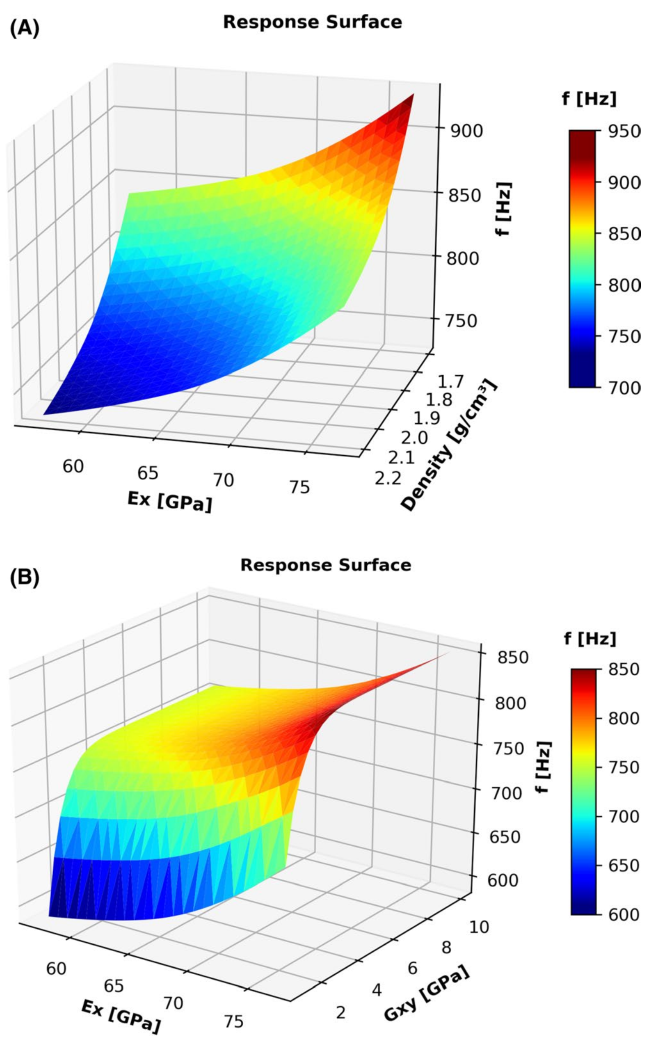


**FIGURE 11** (A) and (B): Response surfaces: evolution of the first fundamental bending frequency with variation of the most influencing material properties ($E_x$: bending longitudinal modulus, $G_{xy}$: intralaminar shear modulus, $f$: eigenfrequency)

### 3.2.2 | Numerical modal analysis performed at high temperatures

The C/C-SiC samples 4 and 6 are numerically analyzed up to a temperature of 900°C and 1250°C respectively. For each sample, the fundamental frequency of the first bending mode at room temperature and in 50 K steps between 200 and 900/1250°C is studied. The numerical and experimental frequencies are compared to each other in Figure 10.

Due to the different geometries of specimens 4 and 6 (Table 1), the measured frequencies of sample 6 are approximately 4 times higher than those of sample 4. It is noticeable that the frequencies measured via RFDA and calculated numerically correspond to the evolution of the dynamic flexural modulus increase versus the temperature shown in Figure 6, as expected from RFDA Equation (1). Between the experimental RFDA frequencies and numerical frequencies a slight difference can also be observed, which remains almost constant along the temperature. The difference between the experimental and numerical frequency reaches a maximal absolute value of 0.4% and 1.4% of the experimental frequency for samples 4 and 6, respectively. Therefore it can be concluded that the FEM model predicts sufficiently the longitudinal dynamic flexural behavior of current developed C/C-SiC materials at high temperatures.

The influence of the material properties was estimated with a parameter correlation study and 3D response surfaces. Correlation coefficients between the material properties of the FEM model and the first fundamental bending frequency were calculated. The closer the absolute correlation value is to 1, the stronger the relationship. The sign of the coefficient indicates whether it is a positive or negative correlation between the input and output parameter.

With correlation coefficients of about 0.6 and −0.6, a strong dependence of the eigenfrequency from respectively the longitudinal dynamic modulus $E_x$ and the density was shown. The intralaminar shear modulus $G_{xy}$ shows a correlation coefficient of 0.2 indicated a lower dependence of the eigenfrequency from $G_{xy}$. The signs of the coefficients illustrates a frequency increase with $E_x$ and $G_{xy}$ and a decrease with the density, which corresponds to the behavior in RFDA Equation (1). The correlation coefficients between the eigenfrequency and the other material properties are negligible (between −0.07 to 0.04).

In a final step, the sensitivity of the eigenfrequency to the most influencing material parameter $E_x$, $G_{xy}$ and the density ρ was analyzed. In Figure 11 the resulting response surfaces illustrate an exponential dependency from the longitudinal modulus and the density. The dependency of the frequency from $G_{xy}$ exists up to a threshold value. The frequency increases exponentially with $G_{xy}$ until $G_{xy}$ reaches a value of about 4.5 GPa. If $G_{xy}$ is higher than this threshold value then the frequency remain constant. It can be stated that the longitudinal modulus as well as the density mainly influenced the first fundamental bending frequency if $G_{xy}$ is above the threshold value of 4.5 GPa.

## 4 | CONCLUSIONS

The RFDA was evaluated for the first time for measuring Young's moduli of C/C-SiC materials 0/90° at room and high temperature in fibre direction. The Young's moduli of C/C-SiC materials were measured via nondestructive RFDA and compared with destructive quasi-static 4-point bending method at room temperature. The dynamic modulus of C/C-SiC material was measured up to 1250°C via RFDA technique. The fundamental frequencies of the first bending modes of all the samples analyzed via RFDA were numerically evaluated via FEM modal analyses.

The room temperature modulus measured via RFDA (67 ± 3 GPa) is comparable to the quasi-static 4-point flexural modulus (63 ± 3 GPa). Therefore determination of Young's modulus via RFDA method is even valid for orthotropic materials as C/C-SiC.

While longitudinal tensile modulus of C/C-SiC material remains constant from room temperature up to approximately 1350°C under inert atmosphere, it was shown experimentally that longitudinal flexural modulus increases with temperature. Thus, for C/C-SiC materials subjected to bending loads at elevated temperature it is recommended to use the numerical approach by means of flexural modulus values.

Finally, it could be proved that the presented RFDA technique shows a high potential for cost-efficient characterisation of CMCs Young's modulus under bending load at operation temperature and allows to surrender laborious destructive bending tests with applied strain gauges.

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## ORCID

*Sandrine Hönig* https://orcid.org/0000-0003-1217-5396

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