Estimation of the effective elastic characteristics of carbon composite materials by testing ring-shaped samples under high temperatures

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Abstract. A computational and experimental method is suggested to determine indirectly the elastic properties of carbon composite materials at room temperature up to 2200°C and more. The method involves loading of ring-shaped test specimens through diametrical compression using non-cooled indenters at three points, within the elastic strain range, with ductility measurements. Based on the measured ductility values, shear moduli, as well as the longitudinal and transversal elasticity moduli in the test specimen cutting plane, can be determined by solving the identification problem. Results of experimental assessment of the permissibility of repetitive mechanical and thermal loading of the carbon-carbon composite material are presented. Method tryout with high-density needle-punched composite material is described.

Keywords: carbon-carbon composite, elasticity modulus, shear modulus, high temperature, cyclic loading

1. Introduction

Analysis of the effective mechanical characteristics, particularly, the elastic properties of high-temperature composites, within the working temperature range, is critical in terms of simulation modeling of structural components made of these materials. For this purpose, it is necessary to perform strain measurements over the gauge length of the test specimen during mechanical tests. For strain measurements, contact [1–3] as well as contactless (generally, optical [4, 5]) strain gauges are used.

Direct measurements methods have a number of hardware limitations, given the high test temperatures; therefore, of equal interest are indirect methods that involve estimation of the elastic properties based on measurements of the change in the position of the test plant crosshead. In addition to the Young's modulus of elasticity, shear modulus measurements are also topical. For anisotropic materials, V-notched test specimens are usually used [6, 7], especially when it comes to high temperature tests. The test specimens of this sort, however, have a number of shortcomings, namely, the narrow area of pure shear strains and the difficulty of their unambiguous differentiation from bending strains. In case of high-temperature measurements, when it is impossible to use any strain-measuring means, the finite rigidity of the test fixture and, when applying local heating to the test specimen, is in a complex stress state under steep thermal gradient conditions, contributes to the measurement error.
Problem formulation
This paper describes a method for indirect estimation of the elasticity/shear moduli of carbon-based composite materials (particularly, CCCM), under uniaxial diametrical compression of ring-shaped test specimens. The method is applicable both at room temperature [8] and at high temperatures. With this method it is possible to determine elasticity moduli (longitudinal $E_1$ and transversal $E_2$) and shear modulus $G_{12}$ for a material orthotropic in the ring cutting plane, using the same test specimen, over a wide temperature range.

The method involved measurement of the ductility values by applying compressive loads to the ring-shaped test specimen, within the elastic strain range, using square-section indenters, consecutively, first along one of the orthotropy axes (see Figure 1, a), then at 45° to this axis (see Figure 1, b), and finally at 90° to this axis (see Figure 1, c).

![Figure 1. Load patterns (a, b, c) for consecutive loading of the ring-shaped test specimen.](image)

Under diametrical compression, ductility values of ring-shaped test specimens are greater than those of test specimens that have no hollow space. Moreover, there are unambiguous dependences of the deflection towards the load direction on the elasticity/shear moduli. For the elastic properties within the CCCM characteristic range, influence of Poisson’s ratio $\nu_{12}$ on the stress-strain state of the ring is very small.

Dependence of the ring-shaped test specimen’s ductility along the load axis $S_{\text{theor}}$ on its elastic properties, for a certain geometry, can be summarized in a table using one of the existing calculation methods, e.g. [9, 10]. In our case, we calculated the dependences using the finite-element simulation modeling. Relative ductility $S$ of the ring-shaped test specimen of particular dimensions can be specified as a function of two relative elastic characteristics $e_2$ and $g_{12}$:

$$s = S_{\text{theor}} E_i = f(e_2, g_{12}),$$

where

$$e_2 = \frac{E_2}{E_1}, \quad g_{12} = \frac{G_{12}}{E_1}.$$

Characteristic dependences of the relative ductility obtained for the ring-shaped test specimen, 50 mm outer diameter, 10 × 10 mm cross section, on $e_2$ and $g_{12}$ are shown in Figure 2.
Since the suggested method does not involve loading of the test specimen beyond the elastic strain range, it is possible, given the technological features of the material being investigated, to perform a full cycle of measurements with the same test specimen over the entire temperature range, thus reducing variations in the experimental values of the elasticity modulus.

For ductility measurements under temperatures close to the room temperature, an extensometer fixed on the ring’s inner surface along the load direction can be used. Under high temperatures, it is suggested to use local heating in the inert atmosphere on the UVTK-type high-temperature mechanical test plant [11].

The algorithm for determining the elastic characteristics for the ring-shaped test specimen involves the following steps:

1. Consecutive loading of the test specimen within the elastic strain range as per load patterns a, b and c (see Figure 1) at room temperature, measuring ductility $S_0$ of the entire powertrain, based on the crosshead displacement, and ductility $S_{0p}$ of the ring by extensometer readings.

2. Evaluation of ductility contribution $S'$ to the total ductility of the entire force loop as follows:

   \[ S' = S_0 - S_{0p} \]

For loading, cooled test fixture is used, its temperature and ductility depending only slightly on the test specimen local heating temperature. Design and material of the non-cooled indenters are chosen so that their ductility can be deemed negligibly small as compared to the ductilities of the cooled test fixture and the heated test specimen.

3. Consecutive loading of the same test specimen at temperature $T_i$ as per load patterns a, b and c (see Figure 1), measuring ductility $S_i$ of the entire force loop.

**Figure 2.** Characteristic dependences of ring-shaped test specimen’s relative ductility on its relative elastic characteristics.
4. Calculation of the test specimen ductility at temperature $T_i$ based on the measured ductility $S_i$ values as follows:

$$S_i^{op} = S_i - S'_i.$$ 

Repeat steps 3 and 4 for each necessary temperature point.

5. Evaluation of moduli $E_{1i}, E_{2i}$ and $G_{12i}$ based on value sets $S_{a_{i}}^{op}, S_{b_{i}}^{op}$ and $S_{c_{i}}^{op}$.

Evaluation of the target moduli represents an identification problem and can be reduced to minimization of residual function $\Phi_i$ that looks as follows:

$$\Phi_i = \left( \frac{S_{a_{i}}^{\text{theor}} (E_{1i}, E_{2i}, G_{12i}) - S_{a_{i}}^{op}}{S_{a_{i}}^{op}} \right)^2 + \left( \frac{S_{b_{i}}^{\text{theor}} (E_{1i}, E_{2i}, G_{12i}) - S_{b_{i}}^{op}}{S_{b_{i}}^{op}} \right)^2 + \left( \frac{S_{c_{i}}^{\text{theor}} (E_{1i}, E_{2i}, G_{12i}) - S_{c_{i}}^{op}}{S_{c_{i}}^{op}} \right)^2,$$

where $S_{a_{i}}^{\text{theor}}, S_{b_{i}}^{\text{theor}}$ and $S_{c_{i}}^{\text{theor}}$ – calculated ring-shaped test specimen ductility values for load patterns a, b and c, correspondingly. The target moduli values are those corresponding to the relevant calculated ductility values.

**Assessment of the permissibility of repetitive mechanical and thermal loading of the test specimen**

An important feature of the suggested method is a series of consecutive measurements during repetitive cycles of test specimen loading/unloading. This may result in degradation of the material properties during the experiment, which is unacceptable.

Tests of materials under investigation have shown no changes in the experimental rigidity values under uniaxial cyclic tests at room temperature, when the loads stay within the proportional elastic limit along the load direction [8].

Assessment of the permissibility of measurements under repetitive mechanical and thermal loading required additional investigations. For the assessment, we used high-density 4D-inplane rod-reinforced CCCM with a coal-tar pitch coke matrix after multiple high temperature treatment (HTT) at about 2250 °C. The tests were performed using the UVTK-type thermomechanical test plant [11], with tensile test specimens cut at 30° to the reinforcement direction, in the transversal anisotropy plane. The loading was performed according to the sinusoidal law, with the frequency of 1 Hz, from zero load up to 20% of proportional elastic limit of the material, over the target temperature range up to 2500 °C. The results obtained over the entire temperature range (up to the HTT temperature) showed that cyclic loading, after hundreds of load cycles, does not have a significant impact on the ductility values both at the target temperature and after the test specimen has cooled down, as related to the initial ductility values. Dependence of the ductility of the powertrain on the test time including the time of test specimen heating, soaking under 2200 °C and cooling is shown in Figure 3.

Repetitive (about ten) heating cycles up to temperatures not exceeding the HTT temperature did not show any impact on the rigidity level either.

At test temperatures higher than the HTT temperature, there was a gradual change in the ductility level, both when soaked under high temperature and after the test specimen has cooled down. The change increased significantly as the temperature approached 2500°C.
Figure 3. Time dependence of the ductility under cyclic loading during heating, soaking under high temperature and cooling (for 4D-inplane-reinforced CCCM).

Results of method tryout

For room temperature measurements, the method was tried out with several CCCM types, primarily, rod-reinforced CCCM. Results obtained for ring-shaped test specimens showed good agreement with those obtained using other direct measurement methods [8].

To try out the method over a wide temperature range, we developed a special test fixture and a special heater to provide uniform temperature distribution in the test specimen.

The method was tried out with high-density 2.5D-type needle-punched CCCM [12] based on Torayca T700S-type fiber-based carbon tape and nonwoven web with a coal-tar pitch coke matrix, after HTT at about 2250°C. During trial testing, we decided not to exceed the HTT temperature, as it would require an additional study of the influence of these temperature conditions on the elastic properties of the test specimen material.

The test specimens were cut in the XZ plane of the test material (here X – one of the carbon tape laying directions, Z – needle punching direction). Results obtained at room temperature were compared with those obtained using strain-gage sensors during tensile tests (X axis), compressive tests (X and Z axes), as well as shear tests, using V-notched shear specimens [7]. In all cases, there was a good agreement between the results obtained with the ring-shaped test specimens and those obtained using alternative methods.

High temperature tests were performed at 1000, 1500, 2000 and 2200°C. Results obtained at room and high temperatures, as well as comparison values for room temperature, are shown in Figure 4.

The temperature dependences obtained for the elastic properties during trial tests with needle-punched CCCM are in a good agreement with the dependences stated in the available publications [13], which attests to applicability of the method.
Figure 4. Results of method tryout with 2.5D-type needle-punched layered CCCM (X – one of carbon tape laying directions, Z – needle punching direction).

Conclusions
A computational and experimental method is suggested to determine indirectly the elastic properties of carbon composite materials under local heating. The method is based on diametrically-compressed test specimen ductility measurement along different directions, with concurrent measurement data processing.

Prior to high-temperature testing of CCCM, assessment of the permissibility of repetitive mechanical and thermal loading of CCCM was performed. Cyclic loading up to 20% of the proportional elastic limit, after hundreds of load cycles, was shown not to have an impact on the ductility values both at high temperature and after the test specimen has cooled down, as related to the initial ductility values. Trial testing of the method was performed with pitch-based needle-punched CCCM at room temperature to 2200°C. Results obtained at room temperature show good agreement with the data obtained using direct measurements with strain-measuring means. The temperature dependences of the properties being investigated correspond to those described in the available publications.

The main advantages of the method are small size of the test specimen, the possibility of testing the same test specimen over a wide temperature range, as well as satisfactory evaluation of the shear modulus at high temperatures.

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