Requirements for technical support of measurements of local elastic characteristics of CCCM by indentation method

Y D Andreeva\(^1\) and I V Magnitskiy\(^1\)

\(^1\) JSC “Kompozit”, Pionerskaya Str., 4, Korolev, Moscow region, Russia, 141070

Abstract. The paper is devoted to the study of the applicability of the indentation method for determining local properties of the matrix of carbon-carbon composite materials. It is shown that from the point of view of preserving the integrity of the matrix, it is advisable to use an indenter with the maximum possible diameter of spherical blunting. The characteristic values of contact stiffness, which have a significant value, are determined, which imposes restrictions on the components of the power circuit of the indentation device. The laws of changes in the level of stresses in the sample under various conditions, including the friction coefficients in the contact, are obtained. Estimates of the influence of the structural heterogeneity of the material on the measurement results are given, and it has been established that with a rational organization of the experiment, they are negligible.

1. Introduction
Currently, aviation, high-speed transport, energy widely use spatially reinforced carbon-carbon composite materials (CCCM) [1].

In this context, the task of designing new CCCMs with the required properties is topical. In view of the high cost of such materials and the considerable duration of the cycle of their production [2], it is advisable to preliminarily model their properties, primarily physical and mechanical, which requires knowledge of the characteristics of their components. In spatially reinforced CCCM, such components are carbon rods and the carbon matrix.

Now to determine the properties of the components of composite materials scientists mainly use indirect methods, both purely theoretical [3] and computational-experimental, for example, identification [4]. The results obtained in this way usually agree well with the experimental values [5], but their reliability in each specific case depends on the adequacy of the physical and mathematical models used to the phenomena observed in practice.

Experimental measurement of the elastic characteristics of the CCCM components is a complex task, since the rods and the matrix in them constitute a single material, and it is difficult to isolate a separate component and test it. Therefore, the development of methods that would allow to obtain the characteristics of the components in the composite.

An analysis of the literature has shown that methods of dynamic and kinetic indentation are often used to solve this problem [6–12]. Most common is the kinetic indentation, a process controlled by a special test setup, in which there is a continuous penetration of an indenter into the test sample under the action of a smoothly increasing load with its subsequent removal and recording the dependence of the tip movement on the load [13]. A method for measuring the set of physicomechanical characteristics of materials during indentation was proposed in [14] and currently, it is very popular and is widely used to determine the characteristics of hardness, stiffness and crack resistance in isotropic materials [15-17].
However, in the considered sources there are no comprehensive studies of the applicability of indentation methods for determining the local properties of the CCCM, including elastic ones. As a rule, the authors of papers associated with the corresponding measurements [6, 8] do not provide a justification for the correctness of the transfer of methods originally developed for metals and alloys to a heterogeneous material with fragile components.

In this paper, we will consider the issues of influence on the observed results of determining the local elastic modulus during indentation of the matrix of the CCCM of structure heterogeneity of the latter, including deviations related to the accuracy of the indenter positioning and characteristics of material sampling.

A model CCCM with a 4DL reinforcement pattern (hexagonal transversely - isotropic according to [18]) and a characteristic rod cross-section size of 0.7 mm was selected as a research object.

2 Task setting
The task was carried out in two stages. Ansys software package was used for calculations.

At the first stage, an indenter suitable for measuring the matrix of the CCCM was chosen its stressed state. Selection criteria in order of priority were considered minimum equivalent voltages and maximum displacements with the same efforts. Due to the fact that the pointed indenters near the top create in brittle materials, which include the CCC matrix, endless stresses [19], only spherically blunt indenters were considered.

To calculate the equivalent stresses, there were considered strength criteria of Mohr-Coulomb, Pisarenko- Lebedev and Botkin-Mirolyubov [20]. For our task, it was enough to make only a comparative assessment of the resulting stress values in the carbon matrix, so we established only general trends. For a more complete picture and taking into account the most unfavorable cases during the experiment, the coefficient of friction between the indenter and the sample and the Poisson's ratio of the matrix were varied.

The problem was solved in a two-dimensional axisymmetric formulation. A model of an indenter and a matrix was built, the hemispherical volume radii of which were 1:10 (figure 1), which ensured the absence of the influence of boundary conditions on the results. Geometric boundary conditions were set for rigidly fixing the matrix on the lower surface of the hemisphere and moving the back side of the indenter. The finite element grid had a thickening at the contact boundary. Verification of the constructed model by analytical calculation according to [23] showed satisfactory agreement.

Figure 1. Calculated model of indenter and matrix in axisymmetric formulation.
The indenter was considered to consist of tungsten carbide with an elastic modulus of 656 GPa, and a Poisson ratio of 0.31 [21]. The properties of the CCCM components here and hereafter correspond to [22].
For a reasonable choice of the size of the indenter, we considered a number of spherical indenters, the sizes of which ranged from 0.10 mm to 0.30 mm in diameter with a step of 0.02 mm. The choice of values was determined as the dimensions of the structural cell of the material under study, and the prevalence of the frame size indentors. At the second stage, a number of tasks related to the presence of the structure of the CCCM were solved. The first of these was to determine the influence of the direction of indentation on the results of changes. For this purpose, the penetration of the indenter into the matrix on the structural cell of the CCCM in the material orthotropy planes of the material was simulated (Figure 2). Indentation was performed in the region as far as possible from the rods. A finite element grid was formed with condensation in the contact area. This choice of grid parameters ensured solution convergence.

**Figure 2.** The considered structural cell of the material with the 4DL reinforcement pattern and the indentation plane of the 4DL structure matrix material.

Geometric boundary conditions were set on the cell. The face opposite to the loaded one was completely fixed. On the rear face of the indenter, displacements normal to this face were set, up to 10 μm. On the surface of contact of the indenter with the material, the contact problem was solved without friction.

The second task was to analyze the influence of the characteristics of rods on the results of measuring the parameters of the matrix. For its solution, the technical elastic constants of the rods were varied, and the Poisson ratios were considered constant, while the elastic and shear moduli were reduced by 2 and 5 times, and also increased by 2 times relative to the initial values. The finite element cell model was as described above.

Hereinafter, when assessing the influence of certain factors on the results of indentation, we used the value of the relative change in force equal to

\[ b = \left| 1 - \frac{P}{P_{\text{ref}}} \right| \]  

(1)

where \( P \) is the indentation power obtained for a given calculation point, and \( P_{\text{ref}} \) is the reference value of the force calculated for indentation in the corresponding plane of the material with the initial values of the characteristics of the components for the current depth of the indenter.

The influence of a factor was considered significant if for it the value of the value \( b \) exceeded the criterion value \( b_0 \). The latter was calculated as the maximum value of \( b \) obtained by varying the Poisson's ratio of the matrix in the range \([0; 0.45]\), corresponding to the expected values of this quantity for the carbon material. This choice of the value \( b_0 \) is due to the fact that the unavoidable error of measurement of the modulus of elasticity of the matrix by the indentation method is the unaccounted influence of its second elastic characteristic, the measurement of which is impossible in the same experiment. If the influence of the factor on the measurement results is less than this value, it is unobservable and, therefore, may not be taken into account. Such a choice of the value of \( b_0 \) despite...
the obvious physical sense, is nevertheless conditional and intended only for a rough assessment of the materiality of the influence. The results obtained in this way are qualitative.

The last task was to assess the influence of the position of the CCCM volume boundary on the experimental results. Since the boundary of the real sample will differ from the boundary of the simulated cell, calculations were made for volumes of material in which the plane of indentation was shifted relative to the initial one in the direction of motion of the indenter by the value \( \frac{1}{2} \) and \( \frac{3}{4} \) rod diameter. The size of the cell, the orientation of the planes, the boundary conditions and settings of the finite element grid remained the same.

3 Results

The results of calculations of the first stage showed that the values of equivalent stresses obtained by the three criteria considered coincide with high accuracy. Further general data are given.

Figure 3 shows the graphs of the dependences of the maximum values of equivalent stresses on the depth of penetration (loading diagrams) by indenters with a diameter of 0.1, 0.2 and 0.3 mm. It can be seen from them that with increasing diameter, the stress level decreases, which makes it possible to consider large indenters to be preferable. The equivalent stresses reach maximum values depending on the penetration depth either at a shallow depth under the tip of the indenter or on the surface near the area where the contact status of the indenter changes with the sample. These two dangerous areas can be considered equiprobable.

![Graphs of dependencies of equivalent stresses on the penetration depth.](image)

The results of calculating the maximum values of equivalent stresses for different values of the friction coefficient in the contact zone show that with increasing friction coefficient from 0 to 0.2, the stresses decrease, after which it will increase further to remain almost unchanged. The general view of the dependencies does not change. Based on this, a model without friction was used for further calculations due to its better convergence.

From similar results for different values of the Poisson's ratio of the matrix, it is clear that the nature of the dependences remains unchanged throughout the entire range of this magnitude considered, and the absolute values of equivalent voltages decrease monotonically. Since our task is to establish general laws, the Poisson coefficient value of 0.25 is considered further.

An important characteristic of the indenter, which determines the requirements for the means of measuring deformations and the power circuit of the device, is the stiffness of the contact area. According to the calculated load diagrams, this parameter can be estimated as a derivative of the current effort by the penetration depth. Table 1 shows values of this parameter for indenters of various diameters with a penetration depth of 10 \( \mu \)m.

The increase in diameter leads to an increase in stiffness, and, consequently, a decrease in the values of displacements under the same load, which complicates the process of measuring them. However, indenters of small diameters create large equivalent stresses in the material, which is dangerous from the point of view of its failure. In our case, it is more important to exclude possible damage to the
material, since this leads to the incorrectness of the physical model of the experiment and the inability to correctly interpret its results, therefore the indenter of the maximum allowable diameter is preferable. This leads to the need to use devices in power circuits for indentation of the most rigid elements.

The results of calculating the stiffness for different values of the Poisson’s ratios provide the opportunity to estimate the value of \( b_0 \) as of 0.16, with more significant deviations observed at large values of Poisson’s ratio.

| Diameter of the indenter, mm | 0.10 | 0.12 | 0.14 | 0.16 | 0.18 | 0.20 | 0.22 | 0.24 | 0.26 | 0.28 | 0.30 |
|-----------------------------|------|------|------|------|------|------|------|------|------|------|------|
| Rigidity, N/mm              | 89.3 | 97.4 | 104.9| 111.8| 118.3| 124.4| 130.3| 135.8| 141.2| 146.3| 151.3|

Figure 4 shows the calculated loading diagrams of a CCCM cell with a 4-DL reinforcement pattern obtained for different planes by varying the stiffnesses of the rods included in its composition. Some non-monotonicity of the obtained curves is related to the error of calculations at small depths of penetration. The influence of the plane in which the indentation is carried out does not exceed 2%, which is significantly lower than the value of \( b_0 \).

As can be seen from the figure, the effect of the elastic characteristics of the rods is also small, reaching 9% with a decrease in their moduli of elasticity and shear by 5 times. From these data, we can conclude that it is possible in principle to measure the local characteristics of a material using the indentation method, since the dependence of the obtained force values, and, therefore, the elastic moduli, on the material structure is negligible.

**Figure 4.** Load diagrams for the XZ, XY, YZ planes with variation of the stiffness of the rods.

The obtained estimates of the influence of the position of indentation surfaces relative to the structure of the CCCM on the force value with a penetration depth of 10 μm show that even when the sample surface is in the immediate vicinity of the rod, the value of the force deviates from the base by no more than 3%, which does not exceed the value \( b_0 \). Thus, it is possible not to impose any requirements on the samples in this respect.

Therefore, with a rational choice of the size of the spherical indenter, the influence of factors related to the heterogeneity of the CCCM on the observed values of the elastic properties of the matrix does not
exceed the error associated with the uncertainty of the Poisson ratio and, therefore, it is permissible not to take it into account when conducting measurements.

4 Conclusion
This paper enables us to draw the following conclusions.
From the point of view of the strength of the matrix of CCCM, it is preferable to use the indenter of the largest possible diameter, however, the stiffness of the contact area when increasing the size of the indenter grows. Since the no-failure requirement is a priority for the material, the diameter of the indenter is determined by the characteristic dimensions of the structural heterogeneity of the material based on the possibility of falling into a specific component of the composite. The stiffness value of the contact area is very large, which imposes restrictions on the corresponding characteristics of the components of the instrument’s power circuit for indentation, including the force sensor.
The dependence of the data obtained during the indentation of the data matrix on the heterogeneity of the local elastic properties of the CCCM is small. There is practically no influence of both the values of elastic constant rods and the position of the sample boundary relative to the structure of the material. This makes it possible not to take into account the presence of several components in the material with a rational choice of the indentation region and guaranteed hitting of the tip of the indenter to the region of exit to the matrix surface.
The results of this paper will be used to create a device for determining the local elastic properties of the CCCM matrix.

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