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Multiscale Numerical Modeling and Characterization of Carbon/Carbon Composites

Borys Drach

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MULTISCALE NUMERICAL MODELING AND CHARACTERIZATION OF CARBON/CARBON COMPOSITES

BY

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DISSERTATION

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To my family
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ABSTRACT

MULTISCALE NUMERICAL MODELING AND CHARACTERIZATION OF CARBON/CARBON COMPOSITES

by

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May, 2013

Carbon/carbon composite is a high-performance material with a unique set of physical and thermo-mechanical properties making it irreplaceable for severe environment aerospace applications. Its complex hierarchical microstructure presents materials scientists and engineers with substantial modeling challenges when it comes to predicting material’s behavior under various loading conditions. My research has been focused on characterization, multiscale modeling and mechanical testing of this composite. A set of new elasticity solutions for the carbon fiber surrounded by multiple layers of pyrolytic carbon represented as a multicoated cylinder is presented on the microscale. On the mesoscale, deterministic and statistical approaches to evaluation of contributions of irregularly shaped pores in C/C composites to the effective mechanical properties are proposed. Finally, on the macroscale, tension/compression testing is utilized to estimate elastic moduli of C/C composite specimens. Methods that are presented in this dissertation provide valuable tools applicable not only to carbon/carbon composites, but a wide range of fiber-reinforced composites and materials with irregularly shaped pores and particles.
I. INTRODUCTION

Carbon/carbon (C/C) composites consist of pure carbon in two distinct forms: carbon fibers and pyrolytic carbon (PyC) matrix. The composites possess unique properties combining very high stiffness, strength and outstanding performance at temperatures up to 2000°C. For these reasons, C/C composites are of great importance in aerospace industry. Initially developed for the nose cones of intercontinental ballistic missiles (Schwartz (2005)), C/C composites are now used for brake discs and brake pads in racing cars and commercial airplanes, thermal shielding in rocket exhaust systems and high temperature heating elements (Savage (1993)). The material also possesses excellent compatibility with human tissue and is used in production of replacement joints and implants.

![Chemical vapor infiltration manufacturing process](image)

Fig. I.1. Chemical vapor infiltration manufacturing process

One of the manufacturing processes to produce C/C composites is called chemical vapor infiltration (CVI). The schematic of the process is shown in Fig. I.1. It starts with a carbon fiber preform, which can be either randomly oriented fibers (carbon fiber felt) or
layers of stitched unidirectional fibers. The preform is placed into the CVI reactor chamber, through which a carbon containing gas (e.g. methane or ethanol) is passed under high temperature and controlled pressure. Temperature causes the gas to decompose into carbon particles, hydrocarbons, carbon oxides, water and hydrogen (depending on the precursor, see Li et al. (2011)). The carbon particles deposit on the fibers in the form of layers of PyC slowly producing the C/C composite. The entire process may take days or even weeks making the composite very expensive to produce. The resulting material and its constituents are brittle and their behavior can be modeled as linearly elastic in loading and temperature service ranges.

CVI manufacturing process results in complex hierarchical microstructure shown in Fig. I.2 (Reznik and Hüttinger (2002), Reznik et al. (2003), Tsukrov et al. (2009)). The composite consists of carbon fibers, layers of PyC with nanotexture that depends on manufacturing conditions, and pores of irregular shapes (varying in size from 20 to 300 μm, shown in Fig. I.2a). Fig. I.2b shows the basic building block of a C/C composite – a carbon fiber (8-10μm in diameter) surrounded by several layers of pyrolytic carbon. Layers of pyrolytic carbon and carbon fibers are cylindrically orthotropic with different sets of thermoelastic parameters for each layer, making modeling of the composite a non-trivial problem.

Micromechanical modeling of C/C composites is performed on several length scales: submicron homogenization to predict the effective elastic properties of pyrolytic carbon
layers, microscale homogenization of bundles of carbon fibers surrounded by multiple layers of PyC, and mesoscale evaluation of contributions from pores. The nanoscale modeling (performed by collaborators from Karlsruhe Institute of Technology in Germany) is briefly described in the text to follow; microscale and mesoscale modeling and experimental measurements on the macroscale are presented in the subsequent Chapters.

One of the challenges in predicting the overall elastic behavior of pyrolytic carbon is that its degree of organization and mechanical properties are determined by the manufacturing parameters as well as by the topology and composition of the substrate. When the material is produced by CVI, the manufacturing parameters include temperature, pressure, residence time and the choice of precursor gas. It has been shown that variations in these parameters may result in isotropic, low-, medium- or high-textured PyC (Reznik and Hüttinger (2002), López-Honorato et al. (2010)). The level of texture can also change with the distance from the substrate (Reznik et al. (2003), Piat et al. (2008)).

PyC (Fig. I.2c) can be considered as an assemblage of submicron transversely isotropic domains consisting of aligned graphene planes. Predictions of its effective elastic properties by traditional homogenization techniques, e.g. first order bounds, are rather inaccurate due to high domain anisotropy. A possible approach is proposed in Böhlke et al. (2010). In that paper the authors assume spherically shaped PyC domains with the properties of highly oriented pyrolytic graphite measured by ultrasound phase spectroscopy with the following components of the stiffness tensor (Gebert et al. (2010)): \( C_{1111} = 40 \text{GPa}, \ C_{3333} = 18.2 \text{GPa}, \ C_{1122} = 20 \text{GPa}, \ C_{1133} = 13 \text{GPa}, \ C_{2222} = 1.8 \text{GPa} \). The orientation distribution of the domains is modelled as von-Mises-Fisher distribution characterized by the concentration parameter \( \kappa \). Three specific orientation distributions of domains are considered in Chapter III of this dissertation: \( \kappa = 0 \) (isotropic overall material behaviour), \( \kappa = 0.1 \) (typical low texture PyC), and \( \kappa = 100 \) (high texture PyC).
The components of the corresponding PyC stiffness tensors are given in sections III.4.1 and III.5.

Fig. I.3 illustrates different methods used to characterize the nanotexture of PyC. A schematic representation of one fiber (F), surrounded by layers of PyC is shown in Fig. I.3a. Polarized light microscopy (PLM) of the material on the scale of tens of microns allows characterization of the level of texture orientation by the extinction angle (Fig. I.3b), see Bortchagovsky et al. (2003), Gray and Cathcart (1966). Transmission electron microscopy (Fig. I.3c) with segmentation analysis or selected area electron diffraction (SAED) technique (an example of an anisotropic diffraction pattern is shown in Fig. I.3d) can be used to resolve the spatial distribution of nanotexture orientation on the scale of tens of nanometers, see Lin et al. (2010), Reznik and Hüttinger (2002) for detailed information on the methods.

![Fig. I.3. Characterization of the nanotexture by polarized light microscopy (PLM) and selected area electron diffraction (SAED)](image)

Due to the complexity of the material's structure, studies on several length scales (nanoscale, microscale, mesoscale and macroscale) are needed for full characterization of the material's behavior. Since test specimens are very expensive to manufacture, characterization by experiment alone is impractical and extensive analytical modeling is
needed. Methods that have been developed for characterization and modeling of the C/C composites within the framework of my research are applicable to a wide range of other composite materials.

This dissertation is organized as follows. Chapter II focuses on elasticity solutions for the carbon fiber surrounded by multiple layers of PyC (basic material unit of the C/C composites) represented as a multicoated cylinder. Chapter III presents a numerical approach to evaluation of contributions of irregularly shaped pores to the effective material properties of C/C composites. Characterization of porosity in a C/C composite sample and a statistical model that correlates pore geometric parameters to the pore compliance contribution parameters are given in Chapter IV. Chapter V describes the experimental measurements of elastic moduli of the C/C composite felt specimens via tension/compression testing. Final conclusions followed by the list of references are given in Chapter VI.
II. ELASTIC DEFORMATIONS AND EFFECTIVE THERMOMECHANICAL PROPERTIES OF COMPOSITE CYLINDERS WITH CYLINDRICALLY ORTHOTROPIC LAYERS

II.1. Introduction and literature review

Elasticity solutions for layered fibers and cylindrical inclusions are of interest in several important areas of mechanics of materials including composites reinforced by fibers with imperfect or modified interface modeled as a certain interphase zone (as in Hashin (2002)), nanowires in semiconductors (see references in Shokrolahi-Zadeh and Shodja (2008)), composites with coated fibers (see, for example, Honjo (2007)), and carbon/carbon composites produced by chemical vapor infiltration (Reznik et al. (2003), Tsukrov et al. (2005)). The ability to quantify deformations and stresses in interphase regions increases in importance with introduction and development of nanoreinforced materials characterized by several orders of magnitude increase in interface surface to volume ratio as compared to conventional composites.

Most of the available results dealing with interphase zones in composite materials are devoted to spherical and ellipsoidal heterogeneities, see Lutz and Zimmerman (1996), Lutz and Zimmerman (2005), Garboczi and Bentz (1997), Wang and Jasiuk (1998), Sevostianov and Kachanov (2007a) and literature therein.

For cylindrical inclusions, i.e. fibers, the important solutions relevant to prediction of the effective elastic and thermoelastic properties can be found in Hashin and Rosen (1964), Walpole (1969), Christensen and Lo (1979) and later works of Avery and Herakovich (1986), Kanaun and Kudryavtseva (1989), Hashin (1990), Hashin (2002), Chen et al. (1990), Herve and Zaoui (1995). In particular, for anisotropic constituents, Avery and Herakovich (1986) derived an analytical solution for a cylindrically orthotropic fiber in an isotropic matrix subjected to thermal stresses (traction-free axisymmetric problem), and investigated the influence of fiber orthotropy on stress
concentrations. Chen et al. (1990) considered thermomechanical loading of a cylindrically orthotropic fiber surrounded by transversely isotropic coating and placed in an infinite transversely isotropic matrix. For this 3-phase material system, they produced solutions for the remotely applied axisymmetric, transverse shear and longitudinal shear loadings. The solutions were then used to predict the effective thermoelastic properties of the composites reinforced by coated fibers (Chen et al. (1990), Benveniste et al. (1991)). In approximately the same time, Hashin (1990) analyzed a composite cylinder consisting of a fiber surrounded by a finite-thickness layer with both materials being cylindrically orthotropic. He solved the corresponding elasticity and conductivity problems utilizing homogeneous boundary conditions on the external surfaces, and produced predictions of the effective transversely isotropic elastic and conductive properties of the composite.

Honjo (2007) calculated thermal stresses and effective material parameters in the ceramic matrix composites reinforced by fibers coated with a layer of pyrolytic carbon. By considering different levels of anisotropy, the author demonstrated the importance of taking into account the cylindrically-orthotropic nature of interfacial carbon coating. Shokrolahi-Zadeh and Shodja (2008) investigated elastic fields in the anisotropic layered cylinders embedded in an unbounded elastic isotropic medium. They utilized their modification of the equivalent inclusion method to produce methodology suitable for general far-field loading. Theotokoglou and Stampouloglou (2008) considered in-plane axisymmetric geometries. They produced mathematical formulation for a radially inhomogeneous cylindrically anisotropic material, and derived solutions for certain distributions of Young's modulus in the isotropic case.

Several publications have been devoted to hollow elastic tubes subjected to various boundary conditions. Gal and Dvorkin (1995) considered a plane strain problem for a cylindrically anisotropic tube subjected to the inside and outside pressure (a generalization of the classical Lamé problem). One of their interesting observations was that, differently from the isotropic case, the anisotropic solution does not converge to
any asymptotic value as the outer radius increases. Tarn and Wang (2001) provided an efficient approach to finding deformations and stresses in laminated anisotropic tubes subjected to extension, torsion, pressure, and bending. They proposed using stress components multiplied by radius as new state variables, and utilized the transfer matrix procedure to transmit the state vector to the outer surface where boundary conditions can be applied. Chatzigeorgiou et al. (2008) investigated effective elastic properties of an anisotropic hollow layered tube with discontinuous elastic coefficients and produced predictions of effective response under torsion and axisymmetric loading.

Only two of the abovementioned papers deal with the situation when both the inner cylinder (fiber) and the surrounding material (matrix) are non-isotropic. However, the solutions of Hashin (1990) are limited to only one layer of a cylindrically-orthotropic material around fiber, while in Shokrolahi-Zadeh and Shodja (2008) the boundary conditions have to be applied at infinity. These deficiencies are addressed in this Chapter which provides explicit expressions for stress and displacement fields in a multilayered composite cylinder with an arbitrary number of cylindrically orthotropic layers subjected to the boundary conditions assigned on the lateral surface of the outer cylinder of finite radius. The following basic homogeneous loading conditions are considered: axial tension/compression, transverse hydrostatic loading, unconstrained thermal expansion, transverse shear, and axial shear loadings (see Fig. II.1). The material in each layer is modeled as linearly elastic; the strains are small. The general form of the solutions is chosen in the form presented in Hashin (1990). The layers are assumed to be perfectly bonded so that the transfer matrices approach described, for example, in Herve and Zaoui (1995) can be utilized.

The mathematical formulation is given in section II.2. Sections II.3-II.5 are devoted to axisymmetric problems, axial shear and transverse shear, correspondingly. In section II.6 expressions to calculate the effective thermomechanical properties of layered cylinders are given. In section II.7 several test problems are solved to illustrate the proposed
solution procedure. The results are compared with the existing solutions, when available, to validate the approach.

Fig. II.1. Basic loading cases: a) axial tension; b) transverse hydrostatic load; c) unconstrained thermal expansion; d) axial shear; e) transverse shear

II.2. Formulation of the problem

Let us consider a material system consisting of a cylindrically-orthotropic or transversely isotropic cylinder of radius $R_1$ surrounded by $(n-1)$ concentric layers ($R_{k-1} \leq r \leq R_k$, $k=2,\ldots,n$) where $r, \theta, z$ are the coordinates in the cylindrical coordinate system as shown in Fig. II.2. Each layer is cylindrically orthotropic so that the stress-strain relations can be presented in the form:

$$\begin{bmatrix}
\sigma_{rr} \\
\sigma_{\theta\theta} \\
\sigma_z \\
\sigma_{r\theta} \\
\sigma_{rz} \\
\sigma_{\theta z}
\end{bmatrix} = \begin{bmatrix}
C_{rr} & C_{r\theta} & C_{rz} & 0 & 0 & 0 \\
C_{r\theta} & C_{\theta\theta} & C_{\theta z} & 0 & 0 & 0 \\
C_{rz} & C_{\theta z} & C_z & 0 & 0 & 0 \\
0 & 0 & 0 & 2G_{\theta z} & 0 & 0 \\
0 & 0 & 0 & 0 & 2G_{rz} & 0 \\
0 & 0 & 0 & 0 & 0 & 2G_{r\theta}
\end{bmatrix} \begin{bmatrix}
\epsilon_{rr} \\
\epsilon_{\theta\theta} \\
\epsilon_z \\
\epsilon_{r\theta} \\
\epsilon_{rz} \\
\epsilon_{\theta z}
\end{bmatrix}^{(k)} + \begin{bmatrix}
\Gamma_r \\
\Gamma_{\theta} \\
\Gamma_z \\
\Gamma_{r\theta} \\
\Gamma_{rz} \\
\Gamma_{\theta z}
\end{bmatrix} \cdot \Delta T, \quad (II.1)
where \( C_{mn}^{(k)} \) and \( G_{mn}^{(k)} \) are the components of the stiffness matrix of \( k \)-th layer; \( \Gamma_r^{(k)}, \Gamma_\theta^{(k)}, \Gamma_z^{(k)} \) are expressed in terms of stiffness matrix components and layer's coefficients of thermal expansion as follows:

\[
\begin{align*}
\Gamma_r^{(k)} &= -(C_{rr}^{(k)} \alpha_r^{(k)} + C_{r\theta}^{(k)} \alpha_\theta^{(k)} + C_{rz}^{(k)} \alpha_z^{(k)}), \\
\Gamma_\theta^{(k)} &= -(C_{r\theta}^{(k)} \alpha_r^{(k)} + C_{\theta\theta}^{(k)} \alpha_\theta^{(k)} + C_{\theta z}^{(k)} \alpha_z^{(k)}), \\
\Gamma_z^{(k)} &= -(C_{rz}^{(k)} \alpha_r^{(k)} + C_{\theta z}^{(k)} \alpha_\theta^{(k)} + C_{zz}^{(k)} \alpha_z^{(k)}).
\end{align*}
\]

The core cylinder \((r \leq R_s)\) is treated as the first layer. By choosing the external radius \(R_s\) to be much larger than \(R_{s-1}\), we produce a model of a layered cylinder of a finite diameter (for example, a fiber surrounded by a layered interphase region) in a cylindrically orthotropic or isotropic matrix.

The height of the cylinder is \(h\) \((-h/2 \leq z \leq h/2\)). Some of the elastic solutions presented in this Chapter are derived for the infinitely long cylinder \((h \to \infty)\).

In the text to follow, parameters associated with a certain layer will be denoted by the layer number shown as a superscript or a subscript depending on the convenience of presentation. A special comment will be made when there is a possibility of confusion with other indices.

The layers are assumed to be perfectly bonded, so the displacements and radial components of traction are continuous through the interface between any two adjacent layers \(k\) and \((k+1)\):

\[
\begin{align*}
\nu_r^{(k)}(R_k) &= \nu_r^{(k+1)}(R_k), & \nu_\theta^{(k)}(R_k) &= \nu_\theta^{(k+1)}(R_k), & \nu_z^{(k)}(R_k) &= \nu_z^{(k+1)}(R_k), \\
\sigma_r^{(k)}(R_k) &= \sigma_r^{(k+1)}(R_k), & \sigma_\theta^{(k)}(R_k) &= \sigma_\theta^{(k+1)}(R_k), & \sigma_z^{(k)}(R_k) &= \sigma_z^{(k+1)}(R_k).
\end{align*}
\]
The equilibrium equations in the absence of body forces are as follows:

\[
\frac{\partial \sigma_{rr}}{\partial r} + \frac{1}{r} \frac{\partial \sigma_{r\theta}}{\partial \theta} + \frac{\partial \sigma_{zr}}{\partial z} + \frac{\sigma_{rr} - \sigma_{\theta\theta}}{r} = 0, \\
\frac{\partial \sigma_{r\theta}}{\partial r} + \frac{1}{r} \frac{\partial \sigma_{\theta\theta}}{\partial \theta} + \frac{\partial \sigma_{z\theta}}{\partial z} + 2\sigma_{r\theta} = 0, \\
\frac{\partial \sigma_{zz}}{\partial r} + \frac{1}{r} \frac{\partial \sigma_{z\theta}}{\partial \theta} + \frac{\partial \sigma_{\theta\theta}}{\partial z} + \sigma_{zz} = 0. \tag{II.3}
\]

The relations between displacements and (small) strains are

\[
\varepsilon_{rr} = \frac{\partial u_r}{\partial r}, \quad \varepsilon_{\theta\theta} = \frac{1}{2} \left( \frac{\partial u_\theta}{\partial r} + \frac{1}{r} \frac{\partial u_r}{\partial \theta} - \frac{u_\theta}{r} \right), \quad \varepsilon_{\theta z} = \frac{1}{2} \left( \frac{\partial u_\theta}{\partial z} + \frac{u_r}{r} \right), \\
\varepsilon_{zz} = \frac{\partial u_z}{\partial z}, \quad \varepsilon_{\phi\phi} = \frac{1}{2} \left( \frac{\partial u_\phi}{\partial r} + \frac{\partial u_\phi}{\partial z} \right) \tag{II.4}
\]

In the next sections, we solve the system of differential equations (II.3) and produce explicit expressions for stress and displacement fields in a $n$-layered composite cylinder with cylindrically orthotropic layers subjected to five basic loading cases: axial tension/compression, transverse hydrostatic loading, unconstrained thermal expansion, axial shear and transverse shear loadings.
II.3. Axial tension/compression and transverse hydrostatic loading

Consider a concentric composite cylinder subjected to the prescribed axial elongation or contraction \( \varepsilon_A \) (Fig. II.1a), external transverse hydrostatic loading \( \sigma_r \) (tension or compression, Fig. II.1b), and prescribed temperature change \( \Delta T \) (Fig. II.1c). The resulting deformation will be axisymmetric: there will be no angular displacement \( u_\theta = 0 \), and the radial displacement will depend upon the radial coordinate only, \( i.e. u_r = u(r) \). Substitution of equations (II.4) and (II.1) into the equilibrium equation (II.3) yields the following differential equation for radial displacement \( u(r) \):

\[
r^2 u'' + ru' - \lambda^2 u + \frac{(C_r - C_{0z}) \varepsilon_A + (\Gamma_r - \Gamma_\theta) \Delta T}{C_r} r = 0, \tag{II.5}
\]

where \( \lambda = \sqrt{C_{99}/C_{rr}} \), and material constants \( C_r, C_{0z}, C_{rr}, C_{99}, \Gamma_r, \Gamma_\theta \) assume their corresponding values for each layer. The general solution of this ordinary differential equation is given, for example, in Hashin (1990):

\[
u_r = A \frac{r^4}{R^{4-1}} + B \frac{R^{4-1}}{r^4} + (\beta \varepsilon_A + \gamma \Delta T) r, \tag{II.6}
\]

where \( \beta = \frac{C_r - C_{0z}}{C_{99} - C_{rr}} \) and \( \gamma = \frac{\Gamma_r - \Gamma_\theta}{C_{99} - C_{rr}} \), \( R \) is the external radius of the considered layer, \( A \) and \( B \) are the integration constants.

From the general solution (II.6), strain-displacement relations (II.4) and stress-strain dependence (II.1) we obtain the expressions for normal stress components:

\[
\sigma_r = A \left( \lambda C_r + C_{\rho r} \right) \left( \frac{r}{R} \right)^{4-1} + B \left( -\lambda C_r + C_{\rho r} \right) \left( \frac{r}{R} \right)^{-4+1} + \left[ \beta \left( C_r + C_{\rho r} \right) + C_{rr} \right] \varepsilon_A + \left[ \gamma \left( C_r + C_{\rho r} \right) + \Gamma_r \right] \Delta T, \tag{II.7}
\]

\[
\sigma_{\rho r} = A \left( \lambda C_r + C_{\rho r} \right) \left( \frac{r}{R} \right)^{4-1} + B \left( -\lambda C_r + C_{\rho r} \right) \left( \frac{r}{R} \right)^{-4+1} + \left[ \beta \left( C_r + C_{\rho r} \right) + C_{99} \right] \varepsilon_A + \left[ \gamma \left( C_r + C_{\rho r} \right) + \Gamma_\theta \right] \Delta T,
\]
The integration constants $A$ and $B$ assume different values for each layer: $A_1, B_1, A_2, B_2, ..., A_n, B_n$. These values can be found from the continuity conditions (II.2).

For axisymmetric deformation, we prescribe radial stresses and radial displacements to be continuous through the interface between $k$ and $(k+1)$ layers:

$$\sigma_\theta^{(k)}(R_k) = \sigma_\theta^{(k+1)}(R_k),$$
$$u_\theta^{(k)}(R_k) = u_\theta^{(k+1)}(R_k). \quad (\text{II.8})$$

Following the influence matrix approach similar to that used in Herve and Zaoui (1995) for isotropic constituents, we represent equations (II.8) in the matrix form:

$$J_i(R_k) V_i + \xi_i L_i(R_k) + \Delta T S_i(R_k) = J_{i+1}(R_k) V_{i+1} + \xi_{i+1} L_{i+1}(R_k) + \Delta T S_{i+1}(R_k),$$

$$k = 1, ..., (n-1), \quad (\text{II.9})$$

where $V_i = [A_i, B_i]^T$ is the vector of integration constants of an $i$-th layer ($i = 1, ..., n$), vectors $L_i(r) = [\beta_i r, \beta_i (C_\eta^{(0)} + C_\varphi^{(0)}) + C_\theta^{(0)}]^T$ and $S_i(r) = [\gamma_i r, \gamma_i (C_\eta^{(0)} + C_\varphi^{(0)}) + \Gamma^{(0)}]^T$ depend on the material properties. Matrix $J_i(r)$ is given by:

$$J_i(r) = \begin{pmatrix}
\frac{r^{(0)} - R_i}{R_i^{(0)} - R_i} & \frac{R_i^{(0)} - R_i}{r^{(0)} - R_i} \\
\left(\lambda^{(0)} C_\eta^{(0)} + C_\varphi^{(0)}\right)\left(\frac{r}{R_i}\right)^{\lambda^{(0)} - 1} & \left(-\lambda^{(0)} C_\eta^{(0)} + C_\varphi^{(0)}\right)\left(\frac{R_i}{r}\right)^{\lambda^{(0)} - 1}
\end{pmatrix}.$$}

Representation (II.9) allows us to construct a recurrent procedure to find all integration constants, and thus obtain the complete displacement, strain and stress fields as given by equations (II.6), (II.4) and (II.7). The procedure is described in the text to follow.

We first solve matrix equation (II.9) for the set of integration constants of the $(k+1)$ th layer:
\[ V_{k+1} = N^{(k+1)} V_i + \varepsilon A M^{(k+1)} + \Delta T T^{(k+1)}, \]

where \( N^{(k+1)} = J_{1+1}^1 (R_k) \cdot J_1^1 (R_k) \),
\[ M^{(k+1)} = J_{1+1}^1 (R_k) \cdot \left[ L_1^1 (R_k) - L_{k+1}^1 (R_k) \right], \]
\[ T^{(k+1)} = J_{1+1}^1 (R_k) \cdot \left[ S_1^1 (R_k) - S_{k+1}^1 (R_k) \right]. \]

Formula (II.10) can be utilized to express the integration constants in any \((k+1)\)th layer in terms of integration constants of the 1st layer (inner core cylinder):

\[ V_{k+1} = Q^{(k+1)} V_i + \varepsilon A P^{(k+1)} + \Delta T U^{(k+1)}, \]

where

\[ Q^{(k+1)} = \prod_{j=k+1}^{2} N^{(j)}; \quad P^{(k+1)} = M^{(k+1)} + \sum_{i=2}^{n} \left[ \prod_{j=k+1}^{i} N^{(j)} \right] M^{(i)}; \quad U^{(k+1)} = T^{(k+1)} + \sum_{i=2}^{n} \left[ \prod_{j=k+1}^{i} N^{(j)} \right] T^{(i)}. \]

In the formulae above, multiplication of the matrices is performed in the prescribed order so that, for example:

\[ \prod_{j=4}^{2} N^{(j)} = N_4 N_3 N_2. \]

Expanding formula (II.11) and assigning \( B_i = 0 \) for the core cylinder to avoid singularity of the radial displacement at the center \((r = 0)\), we produce the following representation for the vector of integration constants:

\[ \begin{bmatrix} A_{k+1} \\ B_{k+1} \end{bmatrix} = \begin{bmatrix} Q_1^{(k+1)} & Q_2^{(k+1)} \\ Q_2^{(k+1)} & Q_2^{(k+1)} \end{bmatrix} \begin{bmatrix} A_i \\ 0 \end{bmatrix} + \varepsilon A \begin{bmatrix} P_1^{(k+1)} \\ P_2^{(k+1)} \end{bmatrix} + \Delta T \begin{bmatrix} U_1^{(k+1)} \\ U_2^{(k+1)} \end{bmatrix} \]

\[ k = 1, \ldots, (n-1). \quad (II.12) \]

These formulae can be resolved to express all integration constants in terms of \( A_i \), so that for each \( i \)-th layer:

\[ A_i = \frac{Q_1^{(i)}}{Q_1^{(n)}} A_i + \varepsilon A \left( \frac{P_1^{(i)}}{Q_1^{(n)}} - \frac{Q_1^{(i)}}{Q_1^{(n)}} P_1^{(n)} \right) + \Delta T \left( \frac{U_1^{(i)}}{Q_1^{(n)}} - \frac{Q_1^{(i)}}{Q_1^{(n)}} U_1^{(n)} \right) \]

\[ B_i = \frac{Q_2^{(i)}}{Q_1^{(n)}} A_i + \varepsilon A \left( \frac{P_2^{(i)}}{Q_1^{(n)}} - \frac{Q_1^{(i)}}{Q_1^{(n)}} P_2^{(n)} \right) + \Delta T \left( \frac{U_2^{(i)}}{Q_1^{(n)}} - \frac{Q_1^{(i)}}{Q_1^{(n)}} U_2^{(n)} \right), \]

\[ i = 1, 2, \ldots, n. \quad (II.13) \]
Using these expressions for the integration constants, displacements and stresses in each layer can be found from formulae (II.6) and (II.7) in terms of parameters $\varepsilon_A$, $A_n$ and $\Delta T$ which are determined from the boundary conditions of the corresponding basic problem as described below.

**II.3.1. Longitudinal deformation of the cylinder**

In the case of longitudinal elongation or contraction of the cylinder ($\varepsilon_z = \varepsilon_A$) with no lateral constraints, the boundary conditions are

$$u_z = \varepsilon_A z, \quad \sigma_r (R_n) = 0, \quad \Delta T = 0. \quad \text{(II.14)}$$

Substituting $r = R_n$ in the expression for the radial component of stress (II.7), the following equation is obtained:

$$A_n \left( \lambda^{(n)} C^{(n)}_r + C^{(n)}_{\theta r} \right) + B_n \left( -\lambda^{(n)} C^{(n)}_r + C^{(n)}_{\theta r} \right) + \left[ \beta^{(n)} \left( C^{(n)}_r + C^{(n)}_{\theta r} \right) + C^{(n)}_{\theta \theta} \right] \varepsilon_A = 0.$$

This equation contains two integration constants of the $n$-th layer $A_n$ and $B_n$. Expressing $B_n$ in terms of $A_n$ and $\varepsilon_A$ by means of the second equation in (II.13), we derive the expression for the integration constant $A_n$:

$$A_n = - \frac{\varepsilon_A \left( \frac{p^{(n)} - Q^{(n)}_z}{Q^{(n)}_z} \right) \left( -\lambda^{(n)} C^{(n)}_r + C^{(n)}_{\theta r} \right) + \left[ \beta^{(n)} \left( C^{(n)}_r + C^{(n)}_{\theta r} \right) + C^{(n)}_{\theta \theta} \right]}{\left( \lambda^{(n)} C^{(n)}_r + C^{(n)}_{\theta r} \right) + \frac{Q^{(n)}_z}{Q^{(n)}_z} \left( -\lambda^{(n)} C^{(n)}_r + C^{(n)}_{\theta r} \right)}}, \quad \text{(II.15)}$$

Representation (II.15) and formulae (II.13) can then be utilized to calculate the integration constants for all layers.

**II.3.2. Transverse hydrostatic loading**

For uniform external lateral compression or tension of the cylinder, the boundary conditions are
\[ u_z(z = \pm h/2) = 0, \quad \sigma_n(R_n) = \sigma_t, \quad \Delta T = 0. \] (II.16)

The first condition of (II.16) defines the plane strain mode of deformation so that \( \varepsilon_a = 0 \). The second condition can be utilized to produce the equation for the integration constants \( A_n \) and \( B_n \). Substituting the expanded expression from (II.7) for \( r = R_n \), we obtain:

\[ A_n \left( \lambda^{(s)} C_{n,\sigma}^{(s)} + C_{r,\sigma}^{(s)} \right) + B_n \left( -\lambda^{(s)} C_{n,\sigma}^{(s)} + C_{r,\sigma}^{(s)} \right) = \sigma_t. \]

Expressing \( B_n \) in terms of \( A_n \), see (II.13), we produce the desired formula for the integration constant \( A_n \):

\[ A_n = \frac{\sigma_t}{\left( \lambda^{(s)} C_{n,\sigma}^{(s)} + C_{r,\sigma}^{(s)} \right) + \frac{Q_{11}^{(s)}}{Q_{11}^{(s)}} \left( -\lambda^{(s)} C_{n,\sigma}^{(s)} + C_{r,\sigma}^{(s)} \right)} \] (II.17)

which, in combination with (II.13), can be used to calculate \( A_i \) and \( B_i \) \((i = 1, 2, \ldots, n)\), and thus the complete expressions for displacement, strain and stress fields in the cylinder (see formulae (II.6), (II.4) and (II.7)).

II.3.3. Unconstrained thermal expansion

This loadcase assumes that the same (homogeneous) temperature change \( \Delta T \) is experienced by the entire material system:

\[ \bar{\sigma}_x = 0, \quad \sigma_n(R_n) = 0, \quad \Delta T \neq 0, \] (II.18)

where \( \bar{\sigma}_x = 0 \) is the average axial stress on the terminal surfaces \((z = \pm h/2)\). From the equations (II.18) and (II.7) we obtain the following relationship for the stresses in external layer

\[ A_n \left( \lambda^{(s)} C_{n,\sigma}^{(s)} + C_{r,\sigma}^{(s)} \right) + B_n \left( -\lambda^{(s)} C_{n,\sigma}^{(s)} + C_{r,\sigma}^{(s)} \right) + \left[ \beta^{(s)} \left( C_{n,\sigma}^{(s)} + C_{r,\sigma}^{(s)} \right) + C_{n,\sigma}^{(s)} \right] \varepsilon_A + \left[ \gamma^{(s)} \left( C_{n,\sigma}^{(s)} + C_{r,\sigma}^{(s)} \right) + \Gamma_{r,\sigma}^{(s)} \right] \Delta T = 0. \]

Coefficient \( B_n \) in this relationship can be expressed in terms of \( A_n \) utilizing (II.13):
Thus, the expression for $A_n$ is

\[
A_n = -\left[ \left( \beta^{(n)} \left( C_{n}^{(n)} + C_{r}^{(n)} \right) + C_{r}^{(n)} \right) \left( p_2^{(n)} - \frac{Q_{11}^{(n)}}{Q_{11}^{(n)} + C_{r}^{(n)}} p_1^{(n)} \right) \right] \epsilon_A + \\
+ \left[ \gamma^{(n)} \left( C_{n}^{(n)} + C_{r}^{(n)} \right) + \Gamma_r^{(n)} \right) + \left( C_{r}^{(n)} - \lambda^{(n)} C_{r}^{(n)} \right) \left( U_2^{(n)} - \frac{Q_{21}^{(n)}}{Q_{11}^{(n)} + C_{r}^{(n)}} U_1^{(n)} \right) \right] \Delta T = 0.
\]

(II.19)

where $\epsilon_A$ has to be chosen to provide zero average longitudinal stresses ($\bar{\sigma}_n = 0$) when the temperature change $\Delta T$ is applied. This parameter is found from the equation

\[
\int_0^{2\pi} \int_0^R \sigma_n (\epsilon_A, \Delta T, r, \theta) r \, dr \, d\theta = 0,
\]

(II.20)

where expression for $\sigma_n$ is given by (II.7) with parameters $A_n, B$, defined by (II.13) and (II.19).

II.4. Axial Shear Deformation

We consider longitudinal shear of the cylinder (Fig. II.1d) such that the in-plane displacements are equal to zero:

\[
\epsilon_{xy} = 0, \quad u_r = 0, \quad u_\theta = 0.
\]

(II.21)

Note that this deformation is different from the one considered in Hashin (1990) where non-zero in-plane displacements $u_r$ and $u_\theta$ were also assumed. Conditions (II.21) correspond to the following axial displacements of the lateral surface:
\[ u_z = sR \cos \theta. \]  

The ends \( z = \pm h/2 \) of the cylinder are traction-free. To determine the distribution of vertical displacements through the cross-sectional area of the multilayered cylinder, we utilize the form of the solution proposed in Hashin (1990)

\[ u_z = s\psi (r, \theta), \]

where \( \psi \) is an unknown function. Calculating the stresses by substituting the expressions for displacements into formulae (II.4) and (II.1) we observe that there are two non-zero components

\[ \sigma_\rho = sG \frac{\partial \psi}{\partial r}, \quad \sigma_\theta = sG \frac{\partial \psi}{r \partial \theta}. \]

Substitution of these stresses into the third equilibrium equation of (II.3) yields the following second order differential equation for unknown function \( \psi (r, \theta) \):

\[ G \left[ \frac{\partial^2 \psi}{\partial r^2} + \frac{\partial \psi}{r \partial r} \right] + G \frac{\partial^2 \psi}{r^2 \partial \theta^2} = 0. \]  

It can be observed that representation

\[ \psi (r, \theta) = (Ar^4 + Br^{-4}) \cos \theta, \]

satisfies equation (II.25), where \( \lambda = G / G_\rho \) and \( A, B \) are the integration constants that are different for each layer. To find these constants we make use of continuity conditions (II.2).

The requirements of continuous displacements \( u_z^{(k)} (R_k) = u_z^{(k+1)} (R_k) \) and tractions \( \sigma_\rho^{(k)} (R_k) = \sigma_\rho^{(k+1)} (R_k) \) on the interface between \( k \)-th and \( (k+1) \)-th layers, produce the following equations relating their constants:

\[
\begin{align*}
A_k R_k^4 + B_k R_k^{-4} &= A_{k+1} R_{k+1}^4 + B_{k+1} R_{k+1}^{-4}, \\
G (A_k R_k^{4-k} - \lambda_k B_k R_k^{-4-k}) &= G (A_{k+1} R_{k+1}^{4-k} - \lambda_{k+1} B_{k+1} R_{k+1}^{-4-k}).
\end{align*}
\]

In the matrix form these equations can be written as
\[ J_i (R_i) V_i = J_{i+1} (R_i) V_{i+1}, \]  

(II.27)

where \( V_i = [A_i, B_i]^T \) is the vector of integration constants of the \( i \)-th layer, and \( J_i (r) \) is the coefficient matrix:

\[
J_i (r) = \begin{pmatrix}
    r^{\lambda_i} & r^{-\lambda_{i-1}} \\
    \lambda_i G_{\alpha}^{(i)} r^{\lambda_{i-1}} & -\lambda_i G_{\alpha}^{(i)} r^{-\lambda_{i-1}}
\end{pmatrix}, 
\]

\( i = 1, 2, ..., n. \)

Equation (II.27) can be used to express the integration constants for any \( k \)-th layer in terms of the inner core integration constants \( A_i, B_i \):

\[
\begin{pmatrix}
    A_k \\
    B_k
\end{pmatrix} = Q^{(k)} \begin{pmatrix}
    A_i \\
    B_i
\end{pmatrix},
\]

(II.28)

where \( Q^{(k)} = \prod_{j=1}^{k} N^{(j)} \) and \( N^{(i)} = -\frac{R_{i-1}}{2\lambda^{(i)} G^{(i)}_{\alpha}} \begin{pmatrix}
    N_{11}^{(i)} & N_{12}^{(i)} \\
    N_{21}^{(i)} & N_{22}^{(i)}
\end{pmatrix}, \)

with the components

\[
N_{11}^{(i)} = -R_{i-1}^{(i-1)-\lambda_{i-1}} \left( \lambda^{(i-1)} G^{(i-1)}_{\alpha} + \lambda^{(i)} G^{(i)}_{\alpha} \right),
\]

\[
N_{12}^{(i)} = R_{i-1}^{(i-1)-\lambda_{i-1}} \left( \lambda^{(i-1)} G^{(i-1)}_{\alpha} - \lambda^{(i)} G^{(i)}_{\alpha} \right),
\]

\[
N_{21}^{(i)} = R_{i-1}^{(i-1)+\lambda_{i-1}} \left( \lambda^{(i-1)} G^{(i-1)}_{\alpha} - \lambda^{(i)} G^{(i)}_{\alpha} \right),
\]

\[
N_{22}^{(i)} = -R_{i-1}^{(i-1)+\lambda_{i-1}} \left( \lambda^{(i-1)} G^{(i-1)}_{\alpha} + \lambda^{(i)} G^{(i)}_{\alpha} \right).
\]

However, the integration constant \( B_i \) should be chosen to be zero to avoid singularity of the stresses at the axis of the cylinder \( (r = 0) \), see equation (II.26). Thus, the integration constants of the \( i \)-th layer are given by

\[
A_i = Q_{i1}^{(i)} A_i, \quad B_i = Q_{i2}^{(i)} A_i.
\]

(II.29)

Note, that formulae (II.28) and (II.29) can be used to relate \( A_i \) and \( B_i \) as follows:

\[
B_i = \frac{Q_{i2}^{(i)}}{Q_{i1}^{(i)}} A_i,
\]

(II.30)
To determine the remaining independent integration constant, we utilize boundary equation (II.22). Substituting expressions (II.23) and (II.26) we obtain:

\[ A_n R_n^{(n)} + B_n R_n^{(-n)} = R_n, \]

which can be solved in combination with (II.30) to produce:

\[ A_n = \left( R_n^{(n)-1} + \frac{Q_n^{(n)}}{Q_1^{(n)}} R_n^{(-n)-1} \right)^{-1}. \]

Now, from (II.29),

\[ \frac{A_n}{Q_1^{(n)}}, \]

and all integration constants can be readily obtained.

II.5. Transverse Shear

In this section we consider a case of transverse shear (Fig. II.1e). In this plane strain problem we are looking for displacements in the form

\[ u_n = u(r) \sin 2\theta, \]
\[ u_\theta = v(r) \cos 2\theta, \]

where \( u(r) \) and \( v(r) \) are two unknown functions of radius \( r \). Substituting these displacements into (II.4) we obtain strains as

\[ \epsilon_n = u' \sin 2\theta, \]
\[ \epsilon_\theta = \frac{(u - 2v)}{r} \sin 2\theta, \]
\[ \gamma_\phi = \frac{(2u - v)}{r} \cos 2\theta, \]

which results in the following representation for stresses:
\[ \sigma_{rr} = \left( C_{rr} u' + C_{x_0} \frac{(u-2v)}{r} \right) \sin 2\theta, \]
\[ \sigma_{x_0} = \left( C_{x_0} u' + C_{x_0} \frac{(u-2v)}{r} \right) \sin 2\theta, \tag{II.34} \]
\[ \tau_{r\theta} = G_{r\theta} \left( \frac{2u-v}{r} + v' \right) \cos 2\theta. \]

The equilibrium equations (II.3) can then be rewritten as a system of two homogeneous differential equations of the second order:

\[ \begin{align*}
C_{rr} \left( r^2 u'' + ru' \right) - (C_{x_0} + 4G_{r\theta}) u - 2(C_{x_0} + G_{r\theta}) rv' + 2(C_{x_0} + G_{r\theta}) v &= 0, \\
2(C_{x_0} + G_{r\theta}) ru' + 2(C_{x_0} + G_{r\theta}) u + G_{r\theta} \left( r^2 v'' + rv' \right) - (4C_{x_0} + G_{r\theta}) v &= 0. \tag{II.35}
\end{align*} \]

Solution of this system is sought in the following form:

\[ u(r) = Ar^a, \quad v(r) = Br^a, \]

where \( A \) and \( B \) are some integration constants. The requirement of existence of non-zero solution yields the following characteristic equation for values of \( \alpha \):

\[ D\alpha^4 + E\alpha^2 + F = 0, \tag{II.36} \]

where

\[ D = C_{rr} G_{x_0}, \]
\[ E = 4(C_{x_0} + G_{r\theta})^2 - C_{rr} \left( 4C_{x_0} + G_{r\theta} \right) - G_{r\theta} \left( C_{x_0} + 4G_{r\theta} \right), \]
\[ F = 9G_{r\theta} C_{x_0}. \]

The roots of the equation (II.36) depend on the material properties of the considered layer. For all materials encountered so far, these roots have been real numbers. Similar observation is reported in Hashin (1990). In particular, in the case of the isotropic material, the discriminant of the bi-quadratic equation (II.36) is positive and equal to

\[ 16E^4 (1-\nu)^2 \left[ (1+\nu)^4 (1-2\nu^2) \right]. \]

The roots are then \( \alpha_{1,2} = \pm 1, \quad \alpha_{3,4} = \pm 3 \) which is consistent with the form of the solution presented in Savin et al. (1967) and Christensen and Lo (1979). In a more general case, for any real roots \( \alpha_1, \alpha_2, \alpha_3, \alpha_4 \) the representations for \( u(r) \) and \( v(r) \) are
\[ u(r) = A_1 r^{\alpha_1} + A_2 r^{\alpha_2} + A_3 r^{\alpha_3} + A_4 r^{\alpha_4}, \quad (\text{II.37}) \]

\[ v(r) = B_1 r^{\alpha_1} + B_2 r^{\alpha_2} + B_3 r^{\alpha_3} + B_4 r^{\alpha_4}. \]

Integration constants \( A_i \) and \( B_i \) \((i = 1, \ldots, 4)\) are linearly dependent. Utilizing, for example, the first equation of (II.35), we obtain:

\[ B_i = g_i A_i \quad (i = 1, \ldots, 4), \]

where

\[ g_i = \frac{\alpha_i^2 C_{\alpha i} - (C_{\alpha \alpha} + 4G_{\alpha \alpha})}{2(C_{\alpha \alpha} + G_{\alpha \alpha}) \alpha_i - 2(C_{\alpha \alpha} + G_{\alpha \alpha})}. \]

Thus, all components of displacement and stress for each \( k \)-th layer can be expressed in terms of parameters \( \alpha_i, g_i, A_i \) \((i = 1, \ldots, 4)\) which are different for different layers:

\[ u_\theta = \sum_{i=1}^{4} A_i r^{\alpha_i} \sin 2\theta, \quad u_\rho = \sum_{i=1}^{4} g_i A_i r^{\alpha_i} \cos 2\theta, \]

\[ \sigma_{\rho \rho} = \sum_{i=1}^{4} \left[ \left( A_i r^{\alpha_i-1} \right) \left( C_{\rho \alpha} \alpha_i + C_{\rho \rho} \left( 1 - 2g_i \right) \right) \right] \sin 2\theta, \quad (\text{II.38}) \]

\[ \sigma_{\theta \theta} = \sum_{i=1}^{4} \left[ \left( A_i r^{\alpha_i-1} \right) \left( C_{\theta \phi} \alpha_i + C_{\theta \theta} \left( 1 - 2g_i \right) \right) \right] \sin 2\theta, \]

\[ \sigma_{\rho \phi} = G_{\rho \phi} \sum_{i=1}^{4} \left[ \left( A_i r^{\alpha_i-1} \right) \left( 2 + \left( \alpha_i - 1 \right) g_i \right) \right] \cos 2\theta. \]

Parameters \( \alpha_1 - \alpha_4, g_1 - g_4 \) are expressed in terms of material moduli, while integration constants \( A_4 - A_4 \) for each layer are found from the continuity and boundary conditions as follows.

We start by utilizing the continuity conditions between two adjacent layers, \( k \) and \( (k+1) \). Substituting the expressions for stresses (II.38) into (II.2) we obtain the following matrix equation relating vectors of the integration constants:

\[ J_k \left( R_k \right) V_k = J_{k+1} \left( R_k \right) V_{k+1}, \quad (\text{II.39}) \]
where \( \mathbf{V}_i = \begin{bmatrix} A_1^{(i)} & A_2^{(i)} & A_3^{(i)} & A_4^{(i)} \end{bmatrix}^T \) is the vector of integration constants of the \( i \)-th layer.

Matrix \( \mathbf{J}_i(r) \) is given by

\[
\mathbf{J}_i(r) = \begin{pmatrix}
    r^{(i)} \sin 2\theta & r^{(i)} \sin 2\theta & r^{(i)} \sin 2\theta & r^{(i)} \sin 2\theta \\
    g_1^{(i)} r^{(i)} \cos 2\theta & g_2^{(i)} r^{(i)} \cos 2\theta & g_3^{(i)} r^{(i)} \cos 2\theta & g_4^{(i)} r^{(i)} \cos 2\theta \\
    p_1^{(i)} r^{(i)-1} \sin 2\theta & p_2^{(i)} r^{(i)-1} \sin 2\theta & p_3^{(i)} r^{(i)-1} \sin 2\theta & p_4^{(i)} r^{(i)-1} \sin 2\theta \\
    q_1^{(i)} r^{(i)-1} \cos 2\theta & q_2^{(i)} r^{(i)-1} \cos 2\theta & q_3^{(i)} r^{(i)-1} \cos 2\theta & q_4^{(i)} r^{(i)-1} \cos 2\theta
\end{pmatrix},
\]

where \( p_j^{(i)} = C_{\alpha}^{(i)} \alpha_j^{(i)} + C_{\gamma}^{(i)} (1 - 2g_j^{(i)}) \), \( q_j^{(i)} = G_{\rho}^{(i)} \left[ 2 + (\alpha_j^{(i)} - 1)g_j^{(i)} \right] \).

From the equation (II.39) all integration constants can be expressed in terms of the set of constants for one layer, for example, the inner core \( A_1^{(i)} - A_4^{(i)} \).

\[
\mathbf{V}_i = Q^{(t+i)} \mathbf{V}_i \tag{II.40}
\]

where, similarly to (II.11), \( Q^{(t+i)} = \prod_{j=1}^{2} [\mathbf{J}_j^{-1}(R_{j-1}) \mathbf{J}_{j-1}(R_{j-1})] \).

Let us find the integration constants for a composite cylinder subjected to the transverse shear strain \( \gamma_{\rho\theta} = s \) at infinity \( (r \to \infty) \):

\[
u_\rho = sr \sin 2\theta, \quad \nu_\theta = sr \cos 2\theta, \tag{II.41}
\]

which yield the following expressions for stresses in the exterior layer:

\[
\sigma_\rho = s \left(C_{\rho}^{(n)} - C_{\rho}^{(n)} \right) \sin 2\theta, \quad \sigma_\theta = s \left(C_{\rho}^{(n)} - C_{\rho}^{(n)} \right) \sin 2\theta, \quad \sigma_{\rho\theta} = 2s G_{\rho}^{(n)} \cos 2\theta. \tag{II.42}
\]

We assume that the exterior layer is transversely isotropic. It can be shown that similar to isotropic case, the roots of characteristic equations are

\[
\alpha_{1,2}^{(n)} = \pm 1, \quad \alpha_{3,4}^{(n)} = \pm 3,
\]

and the displacements are given by the following formulae:
\[ u^{(n)} = A_1^{(n)} r + \frac{A_2^{(n)}}{r} + \frac{A_3^{(n)}}{r^3} + \frac{A_4^{(n)}}{r^5}, \]
\[ v^{(n)} = g_1^{(n)} A_1^{(n)} r + \frac{g_2^{(n)} A_2^{(n)}}{r} + g_3^{(n)} A_3^{(n)} r^3 + \frac{g_4^{(n)} A_4^{(n)}}{r^5}. \]  

Substituting these expressions into the boundary conditions (II.41) we obtain \( A_1^{(n)} = s, A_2^{(n)} = 0 \). The remaining \( A_3^{(n)} \) and \( A_4^{(n)} \) can be found from (II.40):

\[ A_2^{(n)} = Q_2^{(n)} A_2^{(n)} + Q_2^{(n)} A_3^{(n)} + Q_2^{(n)} A_4^{(n)}, \]
\[ A_4^{(n)} = Q_4^{(n)} A_2^{(n)} + Q_4^{(n)} A_3^{(n)} + Q_4^{(n)} A_4^{(n)}, \]  

where \( A_2^{(n)} \) and \( A_4^{(n)} \) are set to be zero to avoid singularity at the center of the composite cylinder. We consider the first and the third equations of the system (II.40) to find \( A_3^{(n)} \) and \( A_4^{(n)} \):

\[
\begin{cases}
  s = Q_2^{(n)} A_2^{(n)} + Q_2^{(n)} A_3^{(n)}, \\
  0 = Q_4^{(n)} A_2^{(n)} + Q_4^{(n)} A_3^{(n)},
\end{cases}
\]

which yields \( A_3^{(n)} = \frac{s Q_3^{(n)}}{Q_2^{(n)} Q_3^{(n)} - Q_2^{(n)} Q_4^{(n)}} \) and \( A_4^{(n)} = -\frac{Q_3^{(n)} A_4^{(n)}}{Q_2^{(n)} Q_3^{(n)}} \). All integration constants in the solution (II.38) are thus found.

II.6. Effective thermoelastic properties of C/C composites

There are several micromechanical models that are traditionally used to predict the effective mechanical properties of composites reinforced by unidirectional randomly located fibers or other types of inhomogeneities. Most of them are based on an elastic solution for a single inhomogeneity placed in a matrix material, where presence of other inhomogeneities is accounted for by modifications either in the matrix material or in the applied stress/strain fields. The examples of such methods are the Mori-Tanaka approach (Mori and Tanaka (1973), Benveniste (1987)), the self-consistent effective medium method (Hershey (1954), Kröner (1958), Hill (1965), Budiansky (1965)), the generalized self-consistent scheme (Christensen and Lo (1979), Benveniste (2008)), the
self-consistent effective field method (Kanaun (1975), Levin (1976), Kanaun and Levin (2008)), the differential scheme (Salganik (1973), McLaughlin (1977), Zimmerman (1985), Zimmerman (1991b)) etc. Similar methodology can be applied to determine the effective thermal expansion coefficients as described in Sevostianov (2012).

The above techniques have been successfully implemented to predict a complete set of elastic parameters of unidirectional fiber composites with transversely isotropic or layered matrices, see Kanaun and Kudryavtseva (1989), Hashin (1990), Benveniste et al. (1991), Herve and Zaoui (1995), Sevostianov et al. (2005). However, approaches presented in the papers above cannot be immediately applied to fibers surrounded by multiple layers of cylindrically orthotropic material.

![Fig. II.3. Axes of material symmetry in carbon/carbon composite](image)

Experimental and analytical studies (Papadakis and Bernstein (1963), Gebert et al. (2010), Böhlke et al. (2010)) show that elastic symmetry of PyC in the CVI infiltrated carbon/carbon composites can be approximately characterized as transverse isotropy with the symmetry axis located in the transverse plane passing through the fiber axis.
perpendicular to the fiber surface, see Fig. II.3. Following Honjo (2007) we denote transversely isotropic materials with symmetry axes \( r, \theta \) and \( z \) as \( TI-r \), \( TI-\theta \) and \( TI-z \), correspondingly. All of these symmetries are special cases of cylindrical orthotropy. Note that carbon fibers can be either transversely isotropic (\( TI-z \)) or cylindrically orthotropic (for example, \( TI-r \) or \( TI-\theta \)), see Guigon et al. (1981). Because PyC matrix is \( TI-r \), the orientation of material symmetry axis at a given point depends on the point's location with respect to the fiber axis. Thus, the micromechanical models utilized for homogenization of carbon/carbon composites have to be able to deal with inhomogeneous (in terms of the orientation of elastic symmetry axes) matrix material.

We consider a unidirectional bundle of carbon fibers surrounded by multiple layers of PyC in the absence of pores (Note that contribution of pores to the effective response of various materials can be found in Zimmerman (1991a), Kachanov et al. (1994), Levin et al. (2000), Drach et al. (2011)). All constituents, fiber and various types of PyC, can be either isotropic or transversely isotropic with respect to fiber axis (\( TI-z \)), or cylindrically orthotropic. Assuming that fibers are randomly located within the bundle, the overall effective properties will be transversely isotropic (\( TI-z \)). Thus, the overall material is characterized by five independent elastic parameters and two independent thermal expansion coefficients (CTEs). As such a set of independent material parameters we choose Young's moduli in longitudinal and transverse directions, \( E^*_l \) and \( E^*_t \), shear moduli in longitudinal and transverse directions, \( G^*_l \) and \( G^*_t \), Poisson's ratio in longitudinal direction \( \nu^*_l \), and CTEs in longitudinal and transverse directions, \( \alpha^*_l \) and \( \alpha^*_t \). The goal of micromechanical modeling is to express these effective material parameters in terms of the properties of constituents and information on their distribution in the composite.

The most appropriate micromechanical approach in this case appears to be the Hashin's replacement scheme (Hashin and Rosen (1964), Hashin (1990)) which is also known as a composite cylinder assemblage (CCA) model. According to this model, we consider a material unit subjected to homogeneous stresses \( \sigma^{(0)} \) (or strains \( \varepsilon^{(0)} \)) which
correspond to tractions \( t = \sigma^{(0)} \cdot n \) (or displacements \( u = \varepsilon^{(0)} \cdot x \)) applied on the external surface \( \Gamma \) of the material unit, where \( x \in \Gamma \) and \( n \) is the outward unit vector normal to \( \Gamma \). If the resulting displacements (or tractions) on the surface \( \Gamma \) are such that they correspond to some homogeneous strain (or stress) field, i.e. \( u = \varepsilon^{(k)} \cdot x \) (or \( t = \sigma^{(k)} \cdot n \)), the material unit is considered to be CCA admissible under loading \( \sigma^{(0)} \) or \( \varepsilon^{(0)} \). The corresponding equivalent elastic parameters of the composite material can then be determined as the proportionality coefficients in equations

\[
\sigma^{(k)} = C_{\mu \nu}^{*} \varepsilon^{(k)} \quad \text{or} \quad \varepsilon^{(k)} = S_{\mu \nu}^{*} \sigma^{(k)}
\]

(II.45)

(no summation over \( k \) and \( l \))

where \( \varepsilon^{(0)} \) is the only non-zero applied component of the homogeneous strain field and \( C_{\mu \nu}^{*} \) is the equivalent stiffness (or \( \sigma^{(0)} \) is the applied component of the homogeneous strain field and \( S_{\mu \nu}^{*} \) is the equivalent compliance). Substitution of the homogeneous material with these elastic properties instead of inhomogeneous material inside of the chosen material unit does not change the elastic fields in the remaining part of the solid under the considered applied loading (Fig. II.4).

Analysis of the elastic solutions presented in this Chapter shows that a composite cylinder with cylindrically orthotropic layers is CCA admissible under uniaxial elongation, transverse hydrostatic loading and axial shear. Thus, four elastic parameters, namely \( E_{A}^{*}, K_{A}^{*}, \nu_{A}^{*} \) and \( G_{A}^{*} \), can be found by solving the corresponding elasticity problems.

Also, the unconstrained thermal expansion of the composite cylinder results in displacements of the external surface \( \Gamma \) that can be put into correspondence to a certain homogeneous strain field, so that the CCA approach can also be used to find CTEs \( \alpha_{x}^{*} \) and \( \alpha_{y}^{*} \).

However, a composite cylinder under transverse shear loading is not CCA admissible. Thus, the replacement model cannot be utilized to find the effective
transverse shear modulus $G_T$ of the composite. To determine this material parameter, one of the formulations of the self-consistent effective medium method can be employed. We consider a composite cylinder inserted in a transversely isotropic ($TI - z$) infinite matrix subjected to remotely applied transverse shear strain $\gamma_T$, see Fig. II.5. The transverse shear modulus $G_T$ of the matrix is assumed to be that of the overall composite. It is found from the requirement that the average transverse shear strain in the composite cylinder is equal to the average transverse shear strain in the matrix, and thus is the same as the applied strain $\gamma_T$:

$$\frac{1}{A} \iint_A \gamma_{xy}(G_T) dA = \gamma_T,$$

where $A$ is the cross-sectional area of the cylinder. Solving equation (II.46) for $G_T$ we obtain the effective transverse shear modulus of the overall composite. The elastic solution for a composite cylinder in the infinite transversely isotropic matrix under remotely applied transverse shear is presented in section II.5. Note that the utilized interpretation of the self-consistent method in terms of the average strains produces the same predictions as other commonly used interpretations, e.g. in terms of the average stresses or elastic strain energy.

Fig. II.4. Composite cylinder assemblage model: a) unidirectional composite material; b) material unit; c) equivalent material unit; d) equivalent material
Micromechanical models presented above can be combined with elastic solutions given in this Chapter to express the effective thermoelastic properties of bundle of unidirectional CVI-densified carbon fibers in terms of the material properties of fiber and PyC layers. These properties can then be used as a basic building block in micromechanical modeling of C/C composites having various microstructures and fiber orientation patterns.

The axisymmetric analysis procedure presented in section II.3 enables us to determine three material parameters of the equivalent transversely isotropic material: longitudinal Young's modulus and Poisson's ratio and transverse bulk modulus. The longitudinal Young's modulus $E_A^*$ and Poisson's ratio $\nu_A^*$ are found by subjecting a composite cylinder to loading conditions (II.14) and calculating displacements from (II.6) and stresses from (II.7). According to Hashin's CCA model, the effective properties are given by

$$E_A^* = \frac{\langle \sigma_m \rangle}{\varepsilon_A}, \quad \nu_A^* = -\frac{u(R)}{\varepsilon_A R},$$

(II.47)

where $\langle \sigma_m \rangle$ is the axial stress averaged over the cylinder cross section. Note that averaging procedure for $\sigma_m$ is needed because the CCA-compatibility condition for longitudinal stresses on the terminal planes $z = \pm h/2$ cannot be satisfied pointwise but
is applicable in an integral sense. However, according to Saint Venant’s principle, variations in values of $\sigma_n$ are only important at the distances comparable with the diameter of the cylinder. Since these distances (tens of microns for a typical 10 $\mu$m diameter fiber) are much smaller than typical fiber length (tens of millimeters), the averaging procedure seems to be appropriate, see discussions in Hashin (1990) and Chen et al. (1990).

Transverse (plane strain) bulk modulus $K_T^*$ relates change in the cross-sectional area of the composite with the applied in-plane hydrostatic loading described by the boundary conditions (II.16). This loading is CCA-admissible; the expression for $K_T^*$ is

$$K_T^* = \frac{\sigma_T}{2u(R_n) / R_n + 2\nu_A^*(\sigma_n) / E_A^*}$$  \hspace{1cm} (II.48)

Axial and transverse CTEs are found from the solution of the unconstrained thermal expansion problem described by (II.18), see section II.3. Axial CTE $\alpha_A^*$ is equal to the value of axial strain $\varepsilon_A$ obtained by solving equation (II.20) when $\Delta T = 1^\circ C$. The transverse CTE is given by

$$\alpha_T^* = \frac{u(R_n)}{R_n},$$  \hspace{1cm} (II.49)

when the homogeneous temperature change $\Delta T = 1^\circ C$ and longitudinal strain $\varepsilon_A = \alpha_A^*$ are applied.

The axial shear modulus is determined from the solution of the elasticity problem described in section II.4. The state of stress resulting from boundary conditions (II.21) and (II.22) is CCA admissible, and the expression for the effective modulus $G_A^*$ can be written as

$$G_A^* = \frac{\sigma_n(r = R_n, \theta = 0)}{s},$$  \hspace{1cm} (II.50)

where stress $\sigma_n$ is found from (II.24) utilizing (II.26) and (II.31).
The problem of transverse shear loading is not CCA admissible. The transverse shear modulus can be calculated using the self-consistent method. According to one of the method's formulations, the material unit (composite cylinder) is placed in the effective medium with properties found from the condition that average strains within the composite cylinder are equal to the average strains in the surrounding matrix. Thus, to find the transverse shear modulus, we solve equation (II.46) for $G_T^*$ utilizing representation (II.38). The transverse Young's modulus and Poisson's ratio are then expressed in terms of $K_T^*$ and $G_T^*$ as

$$E_T^* = \frac{4G_T^*K_T^*}{G_T^* + K_T^*}, \quad v_T^* = \frac{K_T^* - G_T^*}{2(K_T^* + G_T^*)}.$$  

(II.51)

II.7. Examples and Validation

Several test problems are solved to illustrate the developed analytical procedure and validate it against the existing solutions. In particular, we consider the canonical 2D case of a circular isotropic inhomogeneity in an infinite plane under remotely applied homogeneous loading. Then, we derive the solution for a fiber surrounded by anisotropic interphase zone and loaded as described in Shokrolahi-Zadeh and Shodja (2008) and compare our predictions with their results. We also provide solutions for hydrostatic loading of a carbon fiber coated by several layers of cylindrically orthotropic pyrolytic carbon. To validate the solutions for unconstrained thermal expansion loading we compare our results with those of Avery and Herakovich (1986). Finally, we find the stress distributions for five basic loadcases and effective material properties of a carbon fiber surrounded by two layers of PyC.

II.7.1. Hydrostatic loading and transverse shear in $r, \theta$-plane

Consider plane strain problem for an isotropic elastic inclusion with Young's modulus $E_T$, Poisson's ratio $\nu_T$ and radius $R_i = a$ surrounded by a concentric layer of
isotropic elastic material of radius $R_z = R$ with material properties $E_2$ and $v_2$. The solution of this problem in the case of an infinitely large outside layer ($R \to \infty$) can be found in Muskhelishvili (1977), see also Kachanov et al. (2003) for convenient formulae. Muskhelishvili's solution is provided for unidirectional in-plane tension only, however, the hydrostatic and pure in-plane shear cases can be obtained by superposition.

For illustration purposes, let us choose the following values of the material parameters: $E_1 = 10; E_2 = 1; v_1 = 0.2; v_2 = 0.4$. The stiffness matrices are then

$$
\begin{bmatrix}
1.11 & 2.78 & 2.78 & 0 & 0 & 0 \\
2.78 & 1.11 & 2.78 & 0 & 0 & 0 \\
2.78 & 2.78 & 1.11 & 0 & 0 & 0 \\
0 & 0 & 0 & 4.17 & 0 & 0 \\
0 & 0 & 0 & 0 & 4.17 & 0 \\
0 & 0 & 0 & 0 & 0 & 4.17 \\
\end{bmatrix}
$$

In the case of transverse hydrostatic loading, the solution is provided in section II.3.2. It can be observed that the material parameter ratios $\lambda^{(1)} = \lambda^{(2)} = 1$, and the longitudinal strain $\varepsilon_A = 0$ in this example. The coefficients of matrix $Q$ are given by equation (II.11) in terms of the components of matrices $N^{(1)}$. For the considered problem $Q = N^{(2)}$, so that:

$$
Q = \begin{bmatrix}
3.407 & -1.778 \\
-2.407 \frac{\alpha^2}{R^2} & 2.778 \frac{\alpha^2}{R^2} \\
\end{bmatrix}
$$

and matrices $P$ and $U$ are of no interest because they are multiplied by $\varepsilon_A = 0$ and $\Delta T = 0$ correspondingly. Now, all integration constants can be found from equation (II.13):
\[
A_2 = \frac{1}{3.57 + 0.504 \frac{a^2}{R^2}}, \quad B_2 = \frac{0.706}{3.57 \frac{R^2}{a^2} + 0.504}, \quad A_1 = \frac{0.294}{3.57 + 0.504 \frac{a^2}{R^2}}.
\]

Fig. II.6 shows variation of $\sigma_{rr}$ and $\sigma_{\theta\theta}$ with radius for several thicknesses of the outer layer. Note that the graphs for $a/R = 0.1$ coincide with the results obtained using Muskhelishvili (1977) formulae for circular inclusion in an infinite plane.

In the case of in-plane shear, the solution is given in section II.5. The coefficients of characteristic equation (II.36) can be calculated as $D_1 = 46.3, \quad E_1 = -462.96, \quad F_1 = 416.67, \quad D_2 = 0.77, \quad E_2 = -7.65, \quad F_2 = 6.89$. For both layers, the same values $\alpha_{1,2} = \pm 1, \quad \alpha_{3,4} = \pm 3$ for the equation roots are obtained. Note that any choice of isotropic material results in these roots of characteristic equation. Distribution of radial and hoop stresses along the line inclined at $45^\circ$ to $x$-axis is depicted at Fig. II.7. The plots are provided for $a/R \to \infty$; they are indistinguishable from Muskhelishvili (1977) results.
II.7.2. Remote loading of a cylinder surrounded by an orthotropic interphase layer in an isotropic matrix

In this test problem, we compare our predictions with the results provided in Shokrolahi-Zadeh and Shodja (2008), Example 1, p. 3570. We analyze stress distribution in a core cylinder of radius 1 surrounded by a shell of thickness 0.2 placed in an infinite solid and subjected to a remotely applied combination of tension $2\sigma_0$ in $x$-direction and compression $\sigma_0$ in $y$-direction. The core and the outside layer (matrix) are transversely isotropic. The shell is cylindrically orthotropic. To evaluate the influence of the shell's anisotropy, we consider three choices for its material parameters. The case considered by Shokrolahi-Zadeh and Shodja (2008) is denoted as "Shell 2". The material properties of constituent materials are given in Table II.1.
Table II.1. Mechanical properties of constituents in a composite cylinder

<table>
<thead>
<tr>
<th></th>
<th>( E_r ), GPa</th>
<th>( E_\theta ), GPa</th>
<th>( E_z ), GPa</th>
<th>( v_{r\theta} )</th>
<th>( v_{\theta z} )</th>
<th>( v_{rz} )</th>
<th>( G_{r\theta} ), GPa</th>
<th>( G_{\theta z} ), GPa</th>
<th>( G_{rz} ), GPa</th>
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</thead>
<tbody>
<tr>
<td>Core</td>
<td>5</td>
<td>5</td>
<td>10</td>
<td>0.4</td>
<td>0.3</td>
<td>0.3</td>
<td>25/14</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Matrix</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>0.3</td>
<td>0.3</td>
<td>0.3</td>
<td>5/13</td>
<td>5/13</td>
<td>5/13</td>
</tr>
<tr>
<td>Shell 1</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>0.2</td>
<td>0.2</td>
<td>0.2</td>
<td>5/4</td>
<td>5/4</td>
<td>5/4</td>
</tr>
<tr>
<td>Shell 2</td>
<td>6</td>
<td>3</td>
<td>3</td>
<td>0.3</td>
<td>0.2</td>
<td>0.3</td>
<td>1</td>
<td>5/4</td>
<td>1</td>
</tr>
<tr>
<td>Shell 3</td>
<td>12</td>
<td>3</td>
<td>3</td>
<td>0.4</td>
<td>0.2</td>
<td>0.4</td>
<td>3/4</td>
<td>5/4</td>
<td>3/4</td>
</tr>
</tbody>
</table>

Fig. II.8 presents distribution of radial and hoop stresses along the x-axis. As expected, our results for “Shell 2” model coincide with the solution of Shokrolahi-Zadeh and Shodja (2008). It can also be seen that the increase in the radial stiffness of the shell results in more radial stress transmitted to the inner cylinder, as well as bigger jumps in the hoop stresses.

II.7.3. Hydrostatic loading of a carbon fiber surrounded by four layers of pyrolytic carbon

This example is relevant for carbon/carbon composites manufactured by chemical vapor infiltration. The infiltration procedure results in carbon fibers being surrounded by concentric layers of pyrolytic carbon of different texture, see Reznik and Hüttlinger.
The level of texture determines the orthotropy of material stiffness tensor. In particular, one of the carbon/carbon material systems described in Reznik et al. (2003) and Piat et al. (2008) can be treated as a fiber surrounded by four layers of pyrolytic carbon with mechanical properties provided in Table II.2. We consider hydrostatic loading of such a material system. Solution of this problem is not only relevant to thermal treatment of carbon/carbon composites but can also be used to predict the composite’s overall bulk modulus, see Tsukrov et al. (2009). Fig. II.9 presents distributions of the radial and hoop components of stress. Note the significant jump in the hoop stress at the interface between the stiffer fiber and relatively soft pyrolytic carbon.

![Fig. II.9. Radial distribution of normalized $\sigma_r / \sigma_0$ and $\sigma_{\theta\theta} / \sigma_0$ stress components for hydrostatic loading $\sigma_0$ of a carbon fiber surrounded by layers of PyC](image)

<table>
<thead>
<tr>
<th>Layer</th>
<th>$R_{\text{ext}}$, $\mu$m</th>
<th>$C_{rr}$, GPa</th>
<th>$C_{\theta\theta}$, GPa</th>
<th>$C_{rr}$, GPa</th>
<th>$C_{\theta\theta}$, GPa</th>
<th>$C_{rr}$, GPa</th>
<th>$C_{\theta\theta}$, GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>5.8</td>
<td>25.166</td>
<td>37.189</td>
<td>37.189</td>
<td>22.362</td>
<td>19.004</td>
<td>22.362</td>
</tr>
<tr>
<td>3</td>
<td>9.8</td>
<td>23.863</td>
<td>38.496</td>
<td>38.496</td>
<td>22.272</td>
<td>18.466</td>
<td>22.272</td>
</tr>
<tr>
<td>4</td>
<td>10.8</td>
<td>24.953</td>
<td>37.300</td>
<td>37.300</td>
<td>22.332</td>
<td>18.911</td>
<td>22.332</td>
</tr>
<tr>
<td>5</td>
<td>11.18</td>
<td>24.189</td>
<td>38.096</td>
<td>38.096</td>
<td>22.309</td>
<td>18.609</td>
<td>22.309</td>
</tr>
</tbody>
</table>

Table II.2. Mechanical properties of constituents in C/C composite
II.7.4. Unconstrained thermal expansion of a carbon fiber surrounded by a layer of pyrolytic carbon

The unconstrained thermal expansion solution is validated here by comparing with the results of Avery and Herakovich (1986). We consider a cylindrically orthotropic fiber of $r = 38 \mu m$ with elastic parameters $E_r = E_\theta = 27.5 \text{ GPa}$, $E_z = 220 \text{ GPa}$, $\nu_\theta = 0.250$, $\nu_{\theta z} = \nu_{rz} = 0.025$ and thermal expansion coefficients $\alpha_r = \alpha_\theta = 5.56 \times 10^{-6} / ^\circ C$, $\alpha_z = 0.28 \times 10^{-6} / ^\circ C$ surrounded by matrix material (10 $\mu m$ thick). We investigate 3 possible choices for PyC matrix material: isotropic, transversely isotropic with axis of symmetry in the direction of fiber and cylindrically orthotropic. Table II.3 presents the considered material parameters: Matrix_1 was investigated in Avery and Herakovich (1986), the parameters for Matrix_2 and Matrix_3 are chosen based on the values for high-textured pyrolytic carbon reported in Gebert et al. (2010), Gross et al. (2011). Fig. II.10 shows variation of stresses with radius when $\Delta T = 1^\circ C$ is applied to the systems. Our results for isotropic matrix (Fig. II.10a) completely coincide with the solution of Avery and Herakovich (1986). Note that their solution was provided for the isotropic matrix only and cannot be used for the cylindrically-orthotropic case.

<p>| Table II.3. Thermoelastic properties of three considered matrix materials |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|</p>
<table>
<thead>
<tr>
<th>$E_r$, GPa</th>
<th>$E_\theta$, GPa</th>
<th>$E_z$, GPa</th>
<th>$\nu_{\theta r}$</th>
<th>$\nu_{\theta z}$</th>
<th>$\nu_z$</th>
<th>$\alpha_r, 10^{-6}$</th>
<th>$\alpha_\theta, 10^{-6}$</th>
<th>$\alpha_z, 10^{-6}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix_1</td>
<td>34.5</td>
<td>34.5</td>
<td>34.5</td>
<td>0.120</td>
<td>0.120</td>
<td>0.120</td>
<td>1.11</td>
<td>1.11</td>
</tr>
<tr>
<td>Matrix_2</td>
<td>5.2</td>
<td>34.5</td>
<td>34.5</td>
<td>0.040</td>
<td>0.120</td>
<td>0.040</td>
<td>3.50</td>
<td>1.11</td>
</tr>
<tr>
<td>Matrix_3</td>
<td>5.2</td>
<td>12.8</td>
<td>34.5</td>
<td>0.100</td>
<td>0.090</td>
<td>0.040</td>
<td>3.50</td>
<td>2.40</td>
</tr>
</tbody>
</table>
II.7.5. Stress distributions and effective thermomechanical properties of a C/C composite material unit

In the following example, the homogenization procedure presented in section II.6 is applied to a unidirectional composite consisting of carbon fibers surrounded by PyC matrix organized in low-textured (LT) and high-textured (HT) layers as shown in Fig. II.11. By varying CVI conditions, relative thickness of these layers can be manipulated, see Reznik et al. (2003). We investigate how the overall material parameters of unidirectional C/C composite depend on the relative volume fractions of two phases (LT
and HT) of PyC. Table II.4 summarizes the properties of C/C constituents used in the analysis. Properties of the T300 carbon fiber are taken from Wagoner and Bacon (1989). The HT PyC material is assumed to have the thermoelastic properties of highly-oriented pyrolytic graphite reported in Gebert et al. (2010). Mechanical properties of LT PyC are taken from Shavshukov et al. (2008), the CTEs are from Chen et al. (1990).

Fig. II.12 shows variation of the effective thermoelastic properties with volume fraction of the LT layer in the PyC matrix material. The volume fraction of fibers is 25%. The volume fraction of LT layer in the matrix is calculated as

\[ f(LT) = A_{LT} / (A_{LT} + A_{HT}) , \]

where \( A_{LT} \) and \( A_{HT} \) are the cross-sectional areas of LT and HT PyC, correspondingly.

As expected, the increase in concentration of LT PyC, characterized by less alignment of graphene planes and thus lower longitudinal stiffness, results in reduction of axial Young’s modulus \( E_A^* \). On the other hand, the radial stiffness of LT PyC is higher than that of HT PyC causing \( E_r^* \) and \( G_r^* \) to increase with \( f(LT) \). The longitudinal shear modulus \( G_A^* \) of the composite is controlled by the parameter \( G_n \) of the constituents which is greater for LT PyC than for HT PyC. The transverse CTE of the composite is mostly determined by the \( \alpha_r \)-values of the constituents (8.9 \( \cdot 10^{-6} \) °C\(^{-1} \) for fibers vs 3.3 \( \cdot 10^{-6} \) °C\(^{-1} \) for LT vs 21.4 \( \cdot 10^{-6} \) °C\(^{-1} \) for HT), however, contribution from \( \alpha_\theta \) (-1.7 \( \cdot 10^{-6} \) °C\(^{-1} \) for HT) is also significant. That is why for fiber/HT system \( f(LT) = 0 \), the value of \( \alpha_r^* \) (7.3 \( \cdot 10^{-6} \) °C\(^{-1} \)) is much lower than that of HT PyC.

Solid symbols in all graphs correspond to the previously available predictions for transversely isotropic matrix material (● - Schapery (1968); ◆ - Hashin (1983); ▲ - Chamis (1984); ■ - Tsukrov et al. (2005); ★ - Sevostianov et al. (2005)). It can be seen that our results are in good correlation with most of them even though in some of them different approaches (energy based or Mori-Tanaka scheme) were used. The only significant discrepancy is with the evaluation of \( G_A^* \) by Chamis (1984). Note, however,
that their formula produces value of $G^*_d$ higher than the Hashin upper bound (Hashin and Rosen (1964)).

Fig. II.13 presents stress distributions under basic loadcases in the Fiber/LT/HT PyC system described above with the radii of fiber equal to 5 $\mu m$, LT layer equal to 7.5 $\mu m$ and HT layer equal to 10 $\mu m$.

![Fig. II.11. Configuration of C/C composite material unit (carbon fiber surrounded by layers of PyC)](image.png)

**Table II.4. Mechanical properties of constituents in C/C composite**

<table>
<thead>
<tr>
<th></th>
<th>$E_{11}$, GPa</th>
<th>$E_{22}$, GPa</th>
<th>$E_{12}$, GPa</th>
<th>$v_{12}$</th>
<th>$v_{21}$</th>
<th>$G_{12}$, GPa</th>
<th>$G_{13}$, GPa</th>
<th>$G_{23}$, GPa</th>
<th>$\alpha_{11}$, $10^{-4}$</th>
<th>$\alpha_{22}$, $10^{-4}$</th>
<th>$\alpha_{23}$, $10^{-4}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber</td>
<td>14.70</td>
<td>14.70</td>
<td>204.00</td>
<td>0.47</td>
<td>0.02</td>
<td>5.00</td>
<td>22.20</td>
<td>22.20</td>
<td>8.90</td>
<td>8.90</td>
<td>-0.67</td>
</tr>
<tr>
<td>LT</td>
<td>15.11</td>
<td>15.11</td>
<td>15.11</td>
<td>0.28</td>
<td>0.28</td>
<td>5.90</td>
<td>5.90</td>
<td>5.90</td>
<td>3.30</td>
<td>3.30</td>
<td>3.30</td>
</tr>
<tr>
<td>HT</td>
<td>12.48</td>
<td>26.92</td>
<td>26.92</td>
<td>0.22</td>
<td>0.35</td>
<td>1.80</td>
<td>10.00</td>
<td>1.80</td>
<td>21.4</td>
<td>-1.70</td>
<td>-1.70</td>
</tr>
</tbody>
</table>
Fig. II.12. Effective thermoelectric properties of the Fiber/LT/HT system: a) transverse and axial Young's moduli; b) transverse and axial shear moduli; c) transverse and axial CTEs
Fig. 11.13. Stress distributions in the Fiber/LT/HT PyC system for the basic loadcases: a) transverse hydrostatic tension \( \sigma_0 \); b) axial tension \( \sigma_a = 0.001 \); c) unconstrained thermal expansion \( \Delta T = 1^\circ C \); d) axial shear \( s = 0.001, \theta = 0^\circ \); e) transverse shear \( s = 0.001, \theta = 0^\circ \)
II.8. Discussion and conclusions of the microscale homogenization

This Chapter presents cylindrically orthotropic elasticity solutions for a laminated cylinder subjected to homogeneous loading applied to its external surface. Explicit expressions for displacement and stress components are given for five loading cases: transverse hydrostatic tension, axial elongation, unconstrained thermal expansion, longitudinal and transverse shear. All of these expressions contain sets of integration constants having different values for different layers of the cylinder. Due to the assumption of perfect bonding between layers, in all three loading cases the integration constants can be expressed in terms of the core cylinder constants. The core cylinder constants are related to the constants of the outer layer which are found from the corresponding boundary conditions.

Transversely isotropic thermoelastic properties of unidirectional bundles of multicoated fibers are predicted in terms of the properties of constituents which can be cylindrically orthotropic. The predictions of the elastic properties are based on the elasticity solutions for laminated cylinders presented in this Chapter.
III. MODELING OF CARBON/CARBON COMPOSITES WITH 3D PORES OF IRREGULAR SHAPES

III.1. Introduction

In this Chapter, we propose a computational procedure to determine the effective elastic properties of materials with pores of highly irregular shapes. We consider pores observed in carbon/carbon composites manufactured by chemical vapor infiltration of carbon fiber preforms.

Overall mechanical behavior of porous materials is influenced not only by the volume fraction of pores (porosity) but also by their shape and distribution. Fig. III.1 presents an example of a C/C composite characterized by X-ray computed microtomography as described in section III.3.1. The pores in the material appear to be distributed randomly and have highly irregular shapes. Traditional approaches to evaluate the contributions of pores to effective elastic properties are based on the Eshelby solution for ellipsoidal shapes (Eshelby (1957), Eshelby (1959), Mura (1987)). However, due to the irregularity of pore geometry, such approaches are not suitable in this case.

The choice of available results for irregular pore shapes is quite limited. For 2D geometries, the solutions based on conformal mapping were utilized by Zimmerman

For 3D inhomogeneities, analytical solutions for non-ellipsoidal shapes can be found in Faivre (1969), Lee and Johnson (1978), Wu and Du (1995), Rodin (1996), Markenscoff (1998), Nozaki and Taya (1997), Nozaki and Taya (2000). The results for cavities can be obtained from these solutions either by assuming the inhomogeneity stiffness to be zero, or by applying a limiting procedure for stiffness approaching zero. The relation between compliances of inhomogeneities having the same (regular or irregular) shape but different elastic constants is discussed in Sevostianov and Kachanov (2007b); an example of finite element analysis (FEA) calculation and subsequent analysis for a “generalized ellipsoid” is given in Sevostianov et al. (2008). Another commonly used technique involves FEA of a representative volume element filled with a sufficient number of inhomogeneities, see, for example, Arns et al. (2002), Böhme et al. (2004), González et al. (2004) and later publications by these research groups.

Our approach to micromechanical modeling of materials with irregularly shaped 3D pores combines numerical and analytical techniques. The compliance contribution tensor of individual pores is found by FEA as described in section III.3.2 (note that there are no constraints on the level of anisotropy of the matrix material). This tensor is then incorporated in the micromechanical procedure presented in section III.2. The results for an isotropic matrix material with irregularly shaped pores are presented in section III.4.1. The case in which pores are present in a unidirectional C/C composite (homogenized transversely isotropic material) is given in section III.5.
III.2. Contribution of pores to overall elastic properties

One of the approaches to characterize contribution of irregularly shaped pores to the effective elastic properties of PyC is based on the evaluation of their compliance and stiffness contribution tensors (Kachanov et al. (1994), Kachanov et al. (2003), Sevostianov and Kachanov (2002), Tsukrov and Novak (2002), Eroshkin and Tsukrov (2005)).

The approach is based on the concept of a representative volume element (RVE), see Hill (1963), Nemat-Nasser and Hori (1999). The presence of inhomogeneities in the RVE results in additional strains $\Delta \varepsilon$ (stresses $\Delta \sigma$) related to the externally applied homogeneous stresses $\sigma_0$ (strains $\varepsilon_0$) as

$$\Delta \varepsilon = H_{RVE} : \sigma_0, \quad \Delta \sigma = N_{RVE} : \varepsilon_0,$$

(III.1)

where $H_{RVE}$ and $N_{RVE}$ correspond to the compliance and stiffness contribution tensors (Kachanov et al. (1994), Kachanov et al. (2003)). The effective compliance and stiffness of the entire composite are expressed in terms of these tensors as

$$S = S_0 + H_{RVE}, \quad C = C_0 + N_{RVE},$$

(III.2)

where $S_0$ and $C_0$ are the compliance and stiffness of a matrix material.

The exact values for components of $H$ and $N$ are found by direct solution of the boundary value problem for an RVE with all interacting inhomogeneities. Such solutions are usually not obtainable analytically, while numerical solutions require significant computational effort (and often involve statistical processing of the data obtained on particular implementations of microstructure).

To perform homogenization analytically, several micromechanical schemes have been developed. Their implementation in terms of $H_{RVE}$ is discussed in Eroshkin and Tsukrov (2005) and is based on the compliance contribution tensors of individual inhomogeneities. If inhomogeneities are sufficiently far away from each other, the non-interaction approximation can be used. In this case, the corresponding contribution tensors are obtained by direct summation:
where $H^{(i)}$ and $N^{(i)}$ are the compliance and stiffness contribution tensors of individual inhomogeneities, and the summation is performed over all defects present in the RVE. Note that $H^{RVE}$ and $N^{RVE}$ (as well as $H^{(i)}$ and $N^{(i)}$) possess the symmetry of elasticity tensor, so that

$$H_{ijkl} = H_{klij} = H_{ilyk} = H_{ijkl}, \quad N_{ijkl} = N_{klij} = N_{ilyk} = N_{ijkl}. \quad \text{(III.4)}$$

As shown in Eroshkin and Tsukrov (2005), the predictions of more advanced micromechanical schemes can be readily obtained when the non-interaction approximation is known. For example, predictions for the overall elastic compliance by the Mori-Tanaka method (Mori and Tanaka (1973), Benveniste (1987)) is given by

$$S = S_0 + H^{MT}_{RVE}, \quad H^{MT}_{RVE} = H^{NI}_{RVE} : \left[ p(S, - S_0) + H^{NI}_{RVE} \right]^{-1} : (S, - S_0), \quad \text{(III.5)}$$

where $p$ is the volume fraction of inhomogeneities and $S_i$ is their compliance tensor. For the pores, the limiting procedure results in $H^{MT}_{RVE} = H^{NI}_{RVE} / (1 - p)$, see Kachanov et al. (1994).

In the case of regular pore shapes, the elasticity problem for a single pore can be solved analytically and explicit expressions for $H$ and $N$ tensors can be found. For the irregular pore shapes observed in C/C composites, the compliance and stiffness contribution tensors can be calculated by FEA as presented in section III.3.2.

III.3. Extraction of irregularly shaped pores from microtomography data and FEA analysis procedure

III.3.1. X-ray computed microtomography data acquisition and extraction of individual pore shapes

The three dimensional distribution of pores was determined using X-ray computed microtomography ($\mu$CT). $\mu$CT is a non-destructive method for obtaining detailed 3D
representation of material microstructure (see, for example, Salvo et al. (2003), Gebert et al. (2008)). The method is based on measuring attenuation of X-rays passing through a material. The level of attenuation depends on the atomic masses of material constituents, so information on their shape and location can be obtained.

The microtomographic studies were performed in the Institute of Materials Science and Engineering I, Karlsruhe Institute of Technology, Germany. The analyzed cubic 1x1x1cm specimen was cut from CVI infiltrated C/C laminate using high speed water cutting and examined in a desktop CT scanner Skyscan 1072 with a voxel edge length of 14.7μm. The C/C laminate consisted of four unidirectional C/C composite layers ([0°/90°]s) of 2.2 mm thickness each, separated by 0.4 mm thick layers of chemical vapor infiltrated random felt.

Prior to the evaluation of single pores the image was subjected to multiple data processing steps implemented using ITK and VTK libraries (www.vtk.org). First, the image was filtered using a 3D anisotropic Gaussian filter, which allowed for an edge-preserving filtering without smearing the geometrical properties of the pores. Following this step, a connected component region growing algorithm was used to binarize the image. The parameters of the binarization process were adjusted to provide optimal correspondence with the manually classified binarizations of several pores.

The resulting sequence of black and white images was imported into open source software ParaView 3.6.2 (www.paraview.org), where it was converted into 3D surfaces for the separation of individual cavities and exported to stereolithography (STL) format. The triangulated surfaces of pores from the corresponding STL files were then used to create tetrahedral mesh for finite element analysis.
III.3.2. Evaluation of contribution of a single pore by finite element analysis

Pore compliance contribution tensor $H$ of an individual pore was calculated numerically using FEA with Marc/Mentat 2008 software package (www.mscsoftware.com). The following procedure was used:

- The pore surface mesh (extracted from X-ray computed microtomography data as described in section III.3.1) was placed into a cube-shaped reference volume with sides five times larger than the largest dimension of the pore (Fig. III.2). This setup was auto meshed with tetrahedral 3D elements (#134 using Marc/Mentat classification). Typical pore shapes yield FE meshes with the number of elements on the order of 100,000;

- Boundary conditions were applied in displacements for convenience of preprocessing. To obtain all 21 independent components of $H$-tensor, the following 6 loadcases were considered:

Loadcase 1 (uniaxial tension in $x_1$ direction):
\[ X_1^- : u_1 = 0; \quad X_1^+ : u_1 = \varepsilon^{(0)} \cdot \alpha; \]
\[ X_1^-, X_1^+, X_2^-, X_2^+, X_3^-, X_3^+ : u_2 = 0; \quad u_3 = 0; \]

Loadcase 2 (uniaxial tension in $x_2$ direction):
\[ X_2^- : u_2 = 0; \quad X_2^+ : u_2 = \varepsilon^{(0)} \cdot \alpha; \]
\[ X_1^-, X_1^+, X_2^-, X_2^+, X_3^-, X_3^+ : u_1 = 0; \quad u_3 = 0; \]

Loadcase 3 (uniaxial tension in $x_3$ direction):
\[ X_3^- : u_3 = 0; \quad X_3^+ : u_3 = \varepsilon^{(0)} \cdot \alpha; \]
\[ X_1^-, X_1^+, X_2^-, X_2^+, X_3^-, X_3^+ : u_1 = 0; \quad u_2 = 0; \]

Loadcase 4 (shear deformation in $x_1x_2$ plane):
\[ X_1^- : u_2 = 0; \quad X_1^+ : u_2 = \varepsilon^{(0)} \cdot \alpha; \]
\[ X_1^-, X_1^+, X_2^-, X_2^+, X_3^-, X_3^+ : u_1 = 0; \quad u_3 = 0; \]

Loadcase 5 (shear deformation in $x_2x_3$ plane):
\[ X_2^- : u_3 = 0; \quad X_2^+ : u_3 = \varepsilon^{(0)} \cdot \alpha; \]
\[ X_1^-, X_1^+, X_2^-, X_2^+, X_3^-, X_3^+ : u_1 = 0; \quad u_2 = 0; \]
Loadcase 6 (shear deformation in $x_2x_1$ plane):

$$X_3^- : u_1 = 0; \quad X_3^+ : u_1 = e^{(0)} \cdot a;$$

$$X_i^- , X_i^+ , X_2^- , X_2^+ , X_3^- , X_3^+ : u_2 = 0; u_3 = 0;$$

where $X_i^+ , X_i^- , X_2^+ , X_2^- , X_3^+ , X_3^-$ denote the faces of the cube with outward normals directed in a positive or negative direction of the corresponding coordinate axes, $a$ is the side length of the reference volume cube, $e^{(0)}$ is the value of applied strain, $u_1, u_2, u_3$ are the displacements in $x_1, x_2, x_3$ directions correspondingly;

![Figure III.2. Reference volume, pore surface mesh and coordinate plane notation](image)

- The FEA simulations were performed and the output file was imported into MATLAB r2009b (www.mathworks.com);
- Stress volume averages $\left\langle \sigma_{ij}(t) \right\rangle_{RVE}$ ($k$ is the loadcase number) were calculated;
- Stiffness contribution tensor $N_{ijkl}$ was calculated: e.g. from the first loadcase (uniaxial tension in $x_1$ direction):

$$N_{ijkl} = \frac{\sigma_{ij}^{(0)} - \left\langle \sigma_{ij}^{(t)} \right\rangle_{RVE}}{e^{(0)}},$$  \hspace{1cm} (III.6)
where $\sigma^{(0)}_{ij}$ are the stresses in the matrix material subjected to $\varepsilon_{ij} = \varepsilon^{(0)}$, and $\langle \sigma^{(0)}_{ij} \rangle_{RVE}$ are the stress volume averages in the porous material subjected to $\varepsilon_{ij} = \varepsilon^{(0)}$. The remaining components can be determined from the symmetry conditions. Both stiffness and compliance contribution tensors are symmetric with respect to $i \leftrightarrow j$, $k \leftrightarrow l$, $(ij) \leftrightarrow (kl)$.

- Compliance contribution tensor $H_{ijkl}$ was then expressed in terms of $N_{ijkl}$:

$$H_{ijkl} = -S^{(0)}_{ijkl} N_{mnop} S^{(0)}_{mnop}, \quad (III.7)$$

where $S^{(0)}_{ijkl}$ are the components of the compliance tensor of the matrix material.

In the above procedure, the size of the reference volume was chosen to simulate remote loading and to eliminate boundary effects. We performed the sensitivity studies for different reference volume sizes (similar to the approach used in Tsukrov and Novak (2002), Teng (2010)) and determined that the reference volume with sides five times greater than the largest dimensions of the pore satisfies such requirements.

III.4. Pores in isotropic PyC matrix

III.4.1. Contribution of pores to effective elastic properties

The algorithm presented in section III.3.2 was used to evaluate the contributions of actual pores to the overall elastic properties of porous pyrolytic carbon. In this section we consider pores in the isotropic PyC with the properties derived using the method described in Böhlke et al. (2010) as $E_0 = 12.79 \text{ GPa}$, $\nu_0 = 0.39$. 

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As an illustration, all components of the H-tensor for a pore shape shown in Fig. III.3 can be found and presented in the matrix form (Voigt convention, see Kachanov et al. (2003)) as follows:

$$
\begin{pmatrix}
\bar{H}_{111} & \bar{H}_{112} & \bar{H}_{113} & 2\bar{H}_{112} & 2\bar{H}_{113} & 2\bar{H}_{111} \\
\bar{H}_{211} & \bar{H}_{222} & \bar{H}_{223} & 2\bar{H}_{222} & 2\bar{H}_{223} & 2\bar{H}_{221} \\
\bar{H}_{311} & \bar{H}_{322} & \bar{H}_{333} & 2\bar{H}_{322} & 2\bar{H}_{333} & 2\bar{H}_{331} \\
2\bar{H}_{121} & 2\bar{H}_{122} & 2\bar{H}_{123} & 4\bar{H}_{122} & 4\bar{H}_{123} & 4\bar{H}_{121} \\
2\bar{H}_{231} & 2\bar{H}_{232} & 2\bar{H}_{233} & 4\bar{H}_{232} & 4\bar{H}_{233} & 4\bar{H}_{231} \\
2\bar{H}_{311} & 2\bar{H}_{312} & 2\bar{H}_{313} & 4\bar{H}_{312} & 4\bar{H}_{313} & 4\bar{H}_{311}
\end{pmatrix}
$$

$$= \begin{pmatrix}
1.7527 & -0.5080 & -0.5088 & 0.0110 & -0.2838 & -0.2329 \\
-0.5080 & 2.3586 & -0.5658 & 0.1435 & -0.0953 & 0.0511 \\
-0.5088 & -0.5658 & 2.5908 & 0.0488 & 0.7214 & 0.3879 \\
0.0110 & 0.1435 & 0.0488 & 5.2280 & 0.1409 & 0.2653 \\
-0.2838 & -0.0953 & 0.7214 & 0.1409 & 6.1285 & -0.0245 \\
-0.2329 & 0.0511 & 0.3879 & 0.2653 & -0.0245 & 5.6228
\end{pmatrix}
$$

(III.8)

where the dimensionless components of H are defined as $\bar{H}_{ijkl} = \frac{V_{E0}}{V_p} H_{ijkl}$ ($V_p / V$ is the volume fraction of the pore in the reference volume). The resulting matrix should be symmetric from mechanical considerations. Numerical values of the corresponding off-diagonal terms obtained by processing FEA data were within 2% of each other; symmetrization was performed by taking the average of the two numbers.
As presented in section III.2, the overall compliance of porous material is given by

\[ S = S_0 + \mathbf{H}_{RVE}, \]

where \( S_0 \) is the compliance tensor of the PyC. Tensor \( \mathbf{H}_{RVE} \) for the representative volume element can be found in terms of the \( \mathbf{H} \)-tensors of individual pores. In particular, at low porosities, the non-interaction approximation can be used so that

\[ \mathbf{H}_{RVE} = \sum_i \mathbf{H}^{(i)} \]

where summation is performed over all pores present in the RVE.

In the case of parallel pores of the same shape, the non-interaction approximation yields

\[ \mathbf{H}_{RVE} = \frac{P}{E_0} \mathcal{H}, \quad (III.9) \]

where \( \mathcal{H} \) is the dimensionless compliance contribution tensor of the considered pore type and \( p \) is the porosity (volume fraction of pores) defined as \( p = \sum_i V^{(i)} / V_{RVE} \). Thus, all effective elastic parameters of porous material can be found. The effective Young's moduli, for example, are given by

\[ \frac{E_i}{E_0} = \frac{1}{1 + p\bar{E}_i}, \quad (III.10) \]

where \( E_i \) is the Young's modulus in the \( x_i \)-direction and \( \bar{E}_i \) is equal to \( \bar{H}_{uu} \) (no summation over repeating indices).

In the case of non-parallel orientation of pores, summation can be substituted by integration over the orientation angles multiplied by the corresponding orientation distribution density. For random orientational distribution of pores of the same type characterized by tensor \( \mathbf{H} \), this procedure results in an isotropic \( \mathbf{H}_{RVE} \)-tensor characterized by two invariants:

\[ \bar{H}_K = \frac{H_{uu}}{3}, \quad \bar{H}_G = \frac{3H_{uu} - H_{yy}}{15}. \quad (III.11) \]

It can be shown that these invariants are related to the changes in the overall bulk and shear moduli of the material containing randomly oriented pores of the same shape.
Utilizing Wu’s strain concentration tensor \( T \), related to \( H \) and \( S_0 \) as \( H = T : S_0 \) (David and Zimmerman (2011)), we obtain the following expression for the overall bulk and shear moduli:

\[
\frac{K}{K_0} = \frac{1}{1 + pK}, \quad \frac{G}{G_0} = \frac{1}{1 + pG},
\]

where \( K_0 = \frac{E_0}{3(1-2\nu)} \) and \( G_0 = \frac{G_0}{2(1+\nu)} \) are the bulk and shear moduli of the matrix material. The pore contributions \( \tilde{K} \) and \( \tilde{G} \) are found as in Wu (1966).

\[
\tilde{K} = \frac{T_{uu}}{3}, \quad \tilde{G} = \frac{3T_{uu} - T_{uu}}{15}.
\]

These parameters are sometimes called the pore compressibility and pore shear compliance.

Note that, as porosity increases, the non-interaction approximation becomes inaccurate and more advanced first order micromechanical schemes are usually used, for example, Mori-Tanaka (Mori and Tanaka (1973), Benveniste (1987)) or Generalized Self-Consistent (Christensen and Lo (1979), Benveniste (2008)). The predictions of these schemes can be obtained in terms of the non-interaction compliance contribution tensors (Eroshkin and Tsukrov (2005)).

Table III.1 presents values of \( \tilde{E}_i \) (contribution to effective Young’s moduli by parallel pores of the same shape), \( \tilde{K} \) and \( \tilde{G} \) (contributions to the bulk and shear moduli of the effective isotropic material in the case of randomly oriented pores) for several selected pores of irregular shapes observed in PyC. All of these pores are shown in a fixed coordinate system aligned with the edges of the cubic specimen described in section III.3.2. Relative deviations from the average values of the parameters \( \tilde{E}_1, \tilde{E}_2, \tilde{E}_3, \tilde{K}, \tilde{G} \) are denoted as \( \delta \tilde{E}_i, \delta \tilde{K}, \delta \tilde{G} \), e. g. \( \delta \tilde{E}_1 = 100\% \cdot (\tilde{E}_1 - \tilde{E}_1^{\text{av}}) / \tilde{E}_1^{\text{av}} \). In the end of the table, the corresponding values for spheroidal cavities (sphere, prolate spheroid with axes’ ratio 1:1:5 and oblate spheroid 1:1:0.2) are shown for comparison. It is obvious that
values of $\tilde{E}, \tilde{E}_2, \tilde{E}_3$ depend on the choice of the global coordinate system, while $\tilde{K}$ and $\tilde{\sigma}$ are invariant.

One immediate observation is that parameters characterizing contributions of the irregularly shaped pores in PyC are very close to each other, particularly pore compressibility $\tilde{K}$ and shear compliance $\tilde{\sigma}$. Most values of $\tilde{K}$ and all analyzed values of $\tilde{\sigma}$ are within 5% of their average values. At the same time, difference between these average values and the corresponding parameters for the considered spheroids is much greater: 21.2%, 12.5% and 58.6% for $\tilde{K}$, and 9.2%, 8.1% and 26.4% for $\tilde{\sigma}$. As previously mentioned in the literature (see, for example Zimmerman (1991a)) the spherical cavity is the stiffest possible pore shape ($\tilde{K} = 4.253$) while oblate spheroids (and, in the limiting case, penny-shaped cracks) are the most compliant objects.

We speculate that the observed closeness of the overall compliance contribution parameters of the considered PyC pores is caused by the chemical vapor infiltration used for the material manufacturing. The pore surface-to-volume ratio, which is one of the characteristic parameters of infiltration, can also be an essential parameter for compressibility and shear compliance of pores with similar aspect ratios (not crack-like). In the 2D case, this fact was previously observed by Zimmerman (1991a), Tsukrov and Novak (2002).
Table III.1. Contributions of selected pores to effective elastic properties

<table>
<thead>
<tr>
<th>Pore Shape</th>
<th>$\bar{E}_1$</th>
<th>$\delta \bar{E}_1$, %</th>
<th>$\bar{E}_2$</th>
<th>$\delta \bar{E}_2$, %</th>
<th>$\bar{E}_3$</th>
<th>$\delta \bar{E}_3$, %</th>
<th>$\bar{k}$</th>
<th>$\delta \bar{k}$, %</th>
<th>$\bar{\sigma}$</th>
<th>$\delta \bar{\sigma}$, %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1.753</td>
<td>-2.7</td>
<td>2.359</td>
<td>-8.5</td>
<td>2.591</td>
<td>14.1</td>
<td>5.507</td>
<td>2.0</td>
<td>2.012</td>
<td>1.0</td>
</tr>
<tr>
<td></td>
<td>1.711</td>
<td>-5.0</td>
<td>2.674</td>
<td>3.7</td>
<td>2.176</td>
<td>-4.2</td>
<td>5.327</td>
<td>-1.3</td>
<td>1.964</td>
<td>-1.3</td>
</tr>
<tr>
<td></td>
<td>1.831</td>
<td>1.7</td>
<td>2.348</td>
<td>-8.9</td>
<td>2.499</td>
<td>10.0</td>
<td>5.402</td>
<td>0.1</td>
<td>1.999</td>
<td>0.4</td>
</tr>
<tr>
<td></td>
<td>1.931</td>
<td>7.2</td>
<td>2.871</td>
<td>11.4</td>
<td>1.896</td>
<td>-16.5</td>
<td>5.354</td>
<td>-0.8</td>
<td>1.992</td>
<td>0.1</td>
</tr>
<tr>
<td></td>
<td>1.491</td>
<td>-17.2</td>
<td>2.765</td>
<td>7.3</td>
<td>2.112</td>
<td>-6.8</td>
<td>5.117</td>
<td>-5.2</td>
<td>1.932</td>
<td>-3.0</td>
</tr>
<tr>
<td></td>
<td>2.070</td>
<td>14.9</td>
<td>2.661</td>
<td>3.2</td>
<td>2.154</td>
<td>-5.2</td>
<td>5.605</td>
<td>3.8</td>
<td>2.056</td>
<td>3.2</td>
</tr>
<tr>
<td></td>
<td>1.994</td>
<td>10.7</td>
<td>2.377</td>
<td>-7.8</td>
<td>2.112</td>
<td>-7.0</td>
<td>5.276</td>
<td>-2.3</td>
<td>1.951</td>
<td>-2.0</td>
</tr>
<tr>
<td></td>
<td>1.630</td>
<td>-9.5</td>
<td>2.568</td>
<td>-0.4</td>
<td>2.626</td>
<td>15.6</td>
<td>5.609</td>
<td>3.9</td>
<td>2.024</td>
<td>1.7</td>
</tr>
<tr>
<td>Arith. mean</td>
<td>1.801</td>
<td></td>
<td>2.578</td>
<td></td>
<td>2.271</td>
<td></td>
<td>5.399</td>
<td></td>
<td>1.991</td>
<td></td>
</tr>
<tr>
<td>Std. deviation</td>
<td>0.194</td>
<td></td>
<td>0.199</td>
<td></td>
<td>0.265</td>
<td></td>
<td>0.169</td>
<td></td>
<td>0.041</td>
<td></td>
</tr>
</tbody>
</table>

| 1.905       | 5.8        | 1.904               | -26.1       | 1.905                | -16.1       | 4.253                | -21.2       | 1.808                | -9.2        |
| 2.379       | 32.1       | 2.381               | -7.6        | 1.121                | -50.6       | 4.725                | -12.5       | 1.830                | -8.1        |
| 1.266       | -29.7      | 1.266               | -50.1       | 5.918                | 160.6       | 8.561                | 58.6        | 2.517                | 26.4        |
III.4.2. Approximation of irregularly shaped pores by ellipsoids using principal components analysis

It is common practice in evaluating contribution to effective properties by three-dimensional pores (and other defects) to assume that the pores have ellipsoidal shapes. The main reason is that only such shapes possess the property of uniform eigenstrain under remotely applied loading, so that the analytical solutions for strains and stresses around them can be utilized (Eshelby (1957), Eshelby (1959), Mura (1987)).

In the case of highly irregular defect shapes, one possible approach is to find the bounds of individual pore contributions by considering the inscribed and circumscribed ellipsoids constructed for such a pore (Hill (1963)). However, for the shapes considered in Table III.1 of the previous section, such an approach would result in extremely wide bounds due to large differences between the dimensions of the inscribed and circumscribed ellipsoids.

When pores are approximated by ellipsoids, two major issues have to be addressed: (1) the choice of the best approximation of the real pore shape by an ellipsoid (orientations and lengths of principal axes) and (2) accuracy of the chosen approximation. An approach utilizing 2D micrographs to select the approximating spheroids is discussed in Laraia et al. (1995), Prokopiev and Sevostianov (2006). In this section of the Chapter, a principal component analysis (PCA) approach (Jolliffe (2002)) to calculate dimensions of approximating ellipsoids of 3D shapes is presented. This approach was implemented by the Karlsruhe Institute of Technology (Germany) researchers Romana Piat and Stefan Dietrich, who provided us with approximating ellipsoid dimensions for several pore shapes. In the text to follow we describe their approach and compare accuracy of the approximating ellipsoid predictions with the direct H-tensor calculations (see section III.3.2).

In the presentation of PCA approach, the notation \( x, y, z \) for the point coordinates will be used. Processing the µCT data, the surfaces of the pores were extracted to obtain
input points \((x_i, y_i, z_i)\) for the estimation of geometrical properties using PCA. Additionally, the volume and center of mass of each pore were determined for further use in the fitting process. A statistical method for describing variations or similarities in data is given by the variance or covariance of a data set (Jolliffe (2002)). In this case, the data set is comprised of all the surface points of the pore, which are a reduced representation of the complete body of the pore structure. It is possible to simplify the description of a pore in the composite to be represented by only a few characteristic parameters by applying the PCA methods to the data set. Therefore, it is necessary to compute the variance in 3D points and assemble the covariance matrix of the pore with all the necessary information to describe a simplified representation of the pore geometry. The covariance of two sets of variables, for example \((X,Y)\), is defined as:

\[
\text{cov}(X,Y) = \frac{1}{n-1} \sum_{i=1}^{n} (x_i - \bar{x})(y_i - \bar{y}) .
\] (III.14)

For the direct estimation of the geometrical parameters of the pore in a local coordinate system, it is advantageous to subtract the center of mass \((\bar{x}, \bar{y}, \bar{z})\) from each point in the point set before the covariance matrix is constructed. This sets the origin of the coordinate system to the center of mass and relates all geometrical parameters to the local coordinate system.

Using the definition of the covariance of the three dimension in space for the point set the covariance matrix is:

\[
\begin{bmatrix}
\text{cov}(X,X) & \text{cov}(X,Y) & \text{cov}(X,Z) \\
\text{cov}(Y,X) & \text{cov}(Y,Y) & \text{cov}(Y,Z) \\
\text{cov}(Z,X) & \text{cov}(Z,Y) & \text{cov}(Z,Z)
\end{bmatrix}
\]

This matrix is symmetric and we apply the eigenvector decomposition to rewrite the covariance matrix in the form suggested by Bronshtein et al. (2007):

\[
\textbf{C} = \textbf{Q} \Lambda \textbf{Q}^T
\] (III.16)

Matrix \(\Lambda = \text{diag}(\lambda_1, \lambda_2, \lambda_3)\) is the diagonal matrix of eigenvalues.
where $\sqrt{\lambda_1}$, $\sqrt{\lambda_2}$ and $\sqrt{\lambda_3}$ are the semi-axes of the approximating ellipsoid and $Q$ is the matrix of eigenvectors composed of the direction cosines of the ellipsoid's principal axes organized in columns. Thus, all parameters of the approximating ellipsoid are defined. The resulting surface has the same variance as the original set of surface data points.

For the pore considered in section III.4.1 (Fig. III.3), the above procedure results in the approximating ellipsoid with semi-axes $a = 0.246d$, $b = 0.308d$, $c = 0.862d$, where $d$ is the length of the pore in $x$ direction, see Fig. III.4. The Euler angles defining orientation of the ellipsoid ($ZY'Z^*$ convention) are $-24.2^\circ$, $77.9^\circ$ and $-130.0^\circ$, correspondingly. The $\mathbf{H}$-tensor of the ellipsoid (calculated using formulas in Kachanov et al. (2003)) is

\[
\mathbf{H} = \begin{bmatrix}
\lambda_1 & 0 & 0 \\
0 & \lambda_2 & 0 \\
0 & 0 & \lambda_3
\end{bmatrix},
\tag{III.17}
\]

Comparing its components with the values for the original pore (III.8), we observe that diagonal terms are relatively close. No conclusive observation for the off-diagonal terms can be made. Introducing the Euclidean norm of the 4th rank tensor $\|\mathcal{S}\| = \sqrt{\mathcal{S}_{ij}\mathcal{S}_{jl}}$ (summation over the repeating indices), the relative distance between the compliance contribution tensor of the actual pore and its approximation is
\[
\Delta = \frac{\sum_{i=1}^{n} y_{i} \cdot (x_{i} - \bar{x})}{\sum_{i=1}^{n} x_{i}^2 - \bar{x}^2} = 0.2431. \tag{III.19}
\]

Note that this parameter is called error in Sevostianov and Kachanov (2008), who utilized it to analyze elastic symmetries.

To provide a more mechanically meaningful comparison, Table III.2 presents compressibility and shear compliance values for approximating ellipsoids compared to the corresponding parameters of the original shapes of the selected pores. The contribution of ellipsoids (H-tensor) was found by utilizing the analytical solutions of Eshelby (1957), Eshelby (1959) and formulas given in Kachanov et al. (2003).

Analyzing the relative error, defined as 
\[
\delta \bar{K} = 100\% \cdot \left( \bar{K}_p - \bar{K}_{ELL} \right) / \bar{K}_p,
\]
\[
\delta \bar{G} = 100\% \cdot \left( \bar{G}_p - \bar{G}_{ELL} \right) / \bar{G}_p,
\]
we conclude that for most shapes the PCA approximation of actual pores by ellipsoids produces discrepancy on the order of 10\% to 20\%. Also, there is no pronounced tendency of the approximation to over- or underestimate the change in stiffness of the porous C/C material.
Table III.2. Irregular pore shapes approximated by ellipsoids. $\tilde{K}_{ELL}$ and $\tilde{G}_{ELL}$ are the compressibility and shear compliance values of ellipsoids; these parameters for pores, $\tilde{K}_p$ and $\tilde{G}_p$ are copied from Table III.1 for comparison.

<table>
<thead>
<tr>
<th>Pore</th>
<th>$\tilde{K}_p$</th>
<th>$\tilde{K}_{ELL}$</th>
<th>$\delta \tilde{K}$, %</th>
<th>$\tilde{G}_p$</th>
<th>$\tilde{G}_{ELL}$</th>
<th>$\delta \tilde{G}$, %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>5.507</td>
<td>4.927</td>
<td>10.5%</td>
<td>2.012</td>
<td>1.933</td>
<td>3.9%</td>
</tr>
<tr>
<td></td>
<td>5.327</td>
<td>6.668</td>
<td>-25.4%</td>
<td>1.964</td>
<td>2.226</td>
<td>-13.3%</td>
</tr>
<tr>
<td></td>
<td>5.402</td>
<td>4.840</td>
<td>10.4%</td>
<td>1.999</td>
<td>1.914</td>
<td>4.3%</td>
</tr>
<tr>
<td></td>
<td>5.354</td>
<td>4.782</td>
<td>10.7%</td>
<td>1.992</td>
<td>1.901</td>
<td>4.6%</td>
</tr>
<tr>
<td></td>
<td>5.117</td>
<td>4.970</td>
<td>2.9%</td>
<td>1.932</td>
<td>1.934</td>
<td>-0.1%</td>
</tr>
</tbody>
</table>

III.5. Pores in unidirectional C/C composite

In this section, we investigate contributions of the pores to the effective elastic properties of unidirectional C/C composite. Fig. III.5 shows the entire unidirectional region of $\mu$CT-scanned sample with the complete set of pores (porosity $p_0 = 9.13\%$, appropriate for non-interaction approximation). This set includes all pores with the smallest dimension greater than 50\(\mu\)m (43 pores total). Smaller pores were excluded from consideration because their contribution to the overall response was minimal. Note that crack-like pores, even of negligible volume fraction, can contribute significantly to
the reduction of the overall stiffness; however, no such pores were observed in the region.

![Fig. III.5. Pore setup in a piece of unidirectional layer in C/C composite](image)

Each of the pores was processed separately to determine its compliance contribution tensor. The example of a pore observed in a unidirectional C/C composite is shown in Fig. III.6. Fibers of the specimen have a diameter of 10μm; the remaining space is filled with layers of PyC deposited on fibers.

![Fig. III.6. Elongated pore in unidirectional C/C composite](image)

In the modeling procedure, pores were assumed to be placed in a homogenized material consisting of unidirectional carbon fiber surrounded by concentric layers of cylindrically orthotropic PyC with two levels of texture, characterized by Fisher parameter $\kappa$ (see Chapter I), as schematically shown in Fig. III.7. This microstructure is described as Mat_B in Piat et al. (2008). Table III.3 provides typical dimensions, levels of
texture, and material properties of carbon fiber/PyC (FPC) system. The properties of the T300 carbon fiber are taken from Wagoner and Bacon (1989). Note that fiber volume fraction in this system is $V_f = 0.189$.

**Table III.3. Material properties of C/C composite constituents**

<table>
<thead>
<tr>
<th>Fiber</th>
<th>5</th>
<th>19.13</th>
<th>19.13</th>
<th>207.82</th>
<th>9.13</th>
<th>7.35</th>
<th>7.35</th>
<th>5.00</th>
<th>22.20</th>
<th>22.20</th>
</tr>
</thead>
<tbody>
<tr>
<td>Layer 1</td>
<td>6.5</td>
<td>0.1</td>
<td>25.02</td>
<td>25.01</td>
<td>25.01</td>
<td>15.48</td>
<td>15.48</td>
<td>4.78</td>
<td>4.75</td>
<td>4.78</td>
</tr>
<tr>
<td>Layer 2</td>
<td>11.5</td>
<td>100</td>
<td>18.12</td>
<td>38.80</td>
<td>38.80</td>
<td>13.15</td>
<td>13.15</td>
<td>1.94</td>
<td>9.66</td>
<td>1.94</td>
</tr>
</tbody>
</table>

**Fig. III.7. Carbon fiber surrounded by two layers of PyC**

The effective properties of the FPC are found utilizing the elasticity solutions and homogenization procedure presented in Chapter II. The application of the procedure to the considered material system results in the following prediction for the effective elastic properties of FPC:

\[
\begin{align*}
E_{1}^{\text{FPC}} &= 59.44 \text{ GPa}, \\
E_{2}^{\text{FPC}} &= E_{3}^{\text{FPC}} = 10.94 \text{ GPa}, \\
\nu_{23}^{\text{FPC}} &= 0.3973, \\
\nu_{12}^{\text{FPC}} &= \nu_{13}^{\text{FPC}} = 0.3229, \\
G_{12}^{\text{FPC}} &= G_{11}^{\text{FPC}} = 4.62 \text{ GPa}.
\end{align*}
\]
The compliance contribution tensor of the pore shown in Fig. III.6 placed in the homogeneous transversely isotropic material with properties given in (III.20) is calculated using procedure presented in section III.3.2 as

\[
\begin{bmatrix}
\bar{H}_{1111} & \bar{H}_{1122} & \bar{H}_{1133} & 2\bar{H}_{1112} & 2\bar{H}_{1123} & 2\bar{H}_{1131} \\
\bar{H}_{2111} & \bar{H}_{2222} & \bar{H}_{2233} & 2\bar{H}_{2123} & 2\bar{H}_{2223} & 2\bar{H}_{2231} \\
\bar{H}_{3111} & \bar{H}_{3222} & \bar{H}_{3333} & 2\bar{H}_{3131} & 2\bar{H}_{3233} & 2\bar{H}_{3331} \\
2\bar{H}_{1211} & 2\bar{H}_{1222} & 2\bar{H}_{1233} & 4\bar{H}_{1212} & 4\bar{H}_{1223} & 4\bar{H}_{1231} \\
2\bar{H}_{2311} & 2\bar{H}_{2322} & 2\bar{H}_{2333} & 4\bar{H}_{2312} & 4\bar{H}_{2323} & 4\bar{H}_{2331} \\
2\bar{H}_{3111} & 2\bar{H}_{3122} & 2\bar{H}_{3133} & 4\bar{H}_{3112} & 4\bar{H}_{3123} & 4\bar{H}_{3131}
\end{bmatrix}
\]

where components of \( \bar{H} \) are normalized with respect to the modulus \( E_i^{EPC} \) and volume fraction of the pore \( V_p \) in the reference volume \( V : \bar{H}_{ijkl} = \frac{E_i^{EPC} V_p}{V} H_{ijkl} \).

After calculating the cavity compliance tensors for all pores shown in Fig. III.5, we can assume the non-interaction approximation for the total porosity of less than 10%. Utilizing formulae (III.2) and (III.3), and extracting the engineering constants from the effective material compliance matrix, the following anisotropic material parameters are obtained:

\[
E_1 = 47.62 \text{GPa}, \quad E_2 = 8.72 \text{GPa}, \quad E_3 = 9.12 \text{GPa},
\]

\[
\nu_{12} = 0.3191, \quad \nu_{23} = 0.3610, \quad \nu_{13} = 0.3048,
\]

\[
G_{12} = 3.96 \text{GPa}, \quad G_{23} = 3.24 \text{GPa}, \quad G_{31} = 4.00 \text{GPa}.
\]

Note that the anisotropy of the effective material is of a general type; however, the deviation from transverse isotropy is not significant. The appropriate symmetrization produces transversely isotropic material with
\[ E_1 = 47.62 \text{GPa}, \quad E_2 = E_3 = 8.92 \text{GPa}, \]
\[ \nu_{12} = \nu_{13} = 0.3120, \quad \nu_{23} = 0.3610, \]
\[ G_{12} = G_{13} = 3.98 \text{GPa}, \]
\[ G_{23} = 3.28 \text{GPa}. \]  

The error of such approximation, expressed in terms of the Euclidean norm (as defined by equation (III.19)), is \( \Delta = 0.0054 \). Thus, the presence of irregularly shaped pores does not introduce a significant deviation from the transversely isotropic behavior of unidirectional C/C composites.

In micromechanical modeling of unidirectional composites pores are often assumed to be aligned with fibers and propagate continuously with constant cross-section (see Fig. III.8b). With these assumptions, the rule of mixtures can be used to predict longitudinal properties of the composite and plane strain solutions can be utilized for transverse response of a composite with cylindrical pore approximations. For the considered material system we obtain: \( E_1 = 54.01 \text{GPa} \) and \( E_2 = 8.59 \text{GPa} \). As can be seen, the rule of mixtures significantly overestimates the longitudinal and slightly underestimates the transverse stiffness of the material by assuming the arrangement of material that provides maximum resistance to unidirectional loading. For comparison, parallel 2:1 spheroidal pores randomly distributed in the same transversely isotropic matrix (non-interaction approximation, see Fig. III.8c) result in the effective moduli: \( E_1 = 50.40 \text{GPa} \) and \( E_2 = 9.03 \text{GPa} \).

Thus, even though the rule of mixtures provides a reasonably good estimate for longitudinal modulus of porous UD C/C composites, the approximation of pores by 2:1 aligned spheroidal shapes (this aspect ratio seems to be close to the average eccentricity of the pores) produces better overall predictions of the effective Young’s moduli.
III.6. Discussion and conclusions of the deterministic approach to evaluation of pore contributions on the mesoscale

CVI infiltrated C/C composites contain 3D pores of irregular shapes that can be extracted by X-ray computed microtomography. The deterministic procedure proposed in this Chapter to evaluate their contributions to the effective elastic properties is based on the compliance contribution tensors of individual pores calculated by FEA. The obtained tensors can then be used in a number of micromechanical schemes; explicit expressions for non-interaction approximation and Mori-Tanaka scheme are given in section III.2. The PyC matrix material is modeled assuming a von-Mises-Fisher distribution of highly textured transversely isotropic domains of submicron size (see Chapter I).

It was observed that the parameters characterizing contributions of individual pores to effective elastic moduli (especially, contributions to the bulk and shear moduli of the effective isotropic material in the case of randomly oriented pores of the same shape) were very similar. We speculate that this closeness in values is caused by the chemical vapor infiltration process used for the material manufacturing.

The principal component analysis approach to approximating the real pore geometry by ellipsoidal shapes is presented in section III.4.2. It was observed that for most shapes, the PCA approximation produces discrepancy on the order of 10% to 20% in the pore compressibility and shear compliance. This approach may be useful when
Bounding of the effective properties by analyzing inscribed and circumscribed ellipsoids is not practical due to large difference in the ellipsoids' dimensions.

Contribution of all pores present in a 3x3x1 mm region of unidirectional C/C composite was analyzed and is presented in section III.5. With porosity of 9.13% the reductions of Young's moduli $E_1$, $E_2$, $E_3$ were 19.9%, 20.3% and 16.6% correspondingly. It was determined that irregularity of elongated pore shapes does not introduce a significant deviation from the transversely isotropic behavior of unidirectional C/C composites. Also, comparing the approximations of pores by long cylinders and 2:1 spheroidal shapes (assuming the same porosity), we concluded that approximation by spheroids produces better overall predictions of the effective Young's moduli.
IV. CHARACTERIZATION AND STATISTICAL MODELING OF IRREGULAR POROSITY IN CARBON/CARBON COMPOSITES BASED ON X-RAY MICROTOMOGRAPHY DATA

IV.1. Introduction

In this Chapter we propose an approach to characterization and statistical modeling of contribution of irregularly shaped pores to the overall elastic properties of porous materials. We illustrate our approach by considering a sample of the carbon/carbon composite manufactured by chemical vapor infiltration of carbon fiber preform. The result of the manufacturing process is a porous material (typical porosities are in the 2-15% range) with pores having highly irregular shapes, as illustrated in Fig.IV.1 (Note that other C/C manufacturing methods also produce irregularly shaped pores, see Tomkova et al. (2008)). The shapes of the pores are influenced by the local arrangement of fibers, the infiltration parameters and carbon deposition rates, and, possibly, by the consecutive thermal treatment of the composite. Characterization and modeling of C/C material constituents including carbon fiber bundles (Hashin (1990), Tsukrov and Drach (2010)), pyrolytic carbon (Reznik and Hüttinger (2002), Böhlke et al. (2010), Gross et al. (2011)) and pores approximated by ellipsoids (Piat et al. (2006b), Piat et al. (2006a)) are discussed in previous Chapters. The objective of this Chapter is to characterize distribution of irregular pores in the material and develop a statistical micromechanical model to predict the overall stiffness based on the proper choice of morphological parameters reflecting pore shapes and orientations. Note that the spatial distribution effects, e.g. clustering of pores, are not included in the scope of this work as they were not observed in the considered specimen; the spatial distribution of the voids is assumed to be uniform.
Our studies are based on the X-ray microtomography (μCT) information obtained as described in Gebert et al. (2008). Microtomography is routinely utilized in micromechanical modeling to characterize microstructure of heterogeneous materials. The obtained data is usually either directly used to develop a finite element mesh reproducing the scanned microstructure as in Pahr and Zysset (2009) or processed to determine a typical ellipsoidal inhomogeneity and utilize it in a certain homogenization procedure as in Borbely et al. (2004). To the best of our knowledge, no statistical models directly incorporating the μCT-determined irregular heterogeneity shapes in the predictions of elastic properties have been reported in the literature.

One of the techniques developed for irregularly shaped pores involves combination of analytical micromechanical modeling with numerical approaches to evaluate contributions of individual pores to the effective elastic properties, see Tsukrov & Novak (2002), Sevostianov et al. (2008) and Drach et al. (2011). In particular, in Chapter III the finite element analysis (FEA) was utilized to find contributions of several pore shapes typical for carbon/carbon composites. However, performing FEA simulations for every individual pore in a typical specimen of interest may be prohibitively time consuming due to large numbers of the pores: the sample analyzed in this Chapter contained around 10,000 individual voids in 1 cubic centimeter volume. Thus, one of the main reasons for considering statistical approaches is the ability to construct prediction models based on such characteristics of pore shapes that can be obtained without the FEA simulations. Another reason is to determine the pore geometric parameters that are of the most significance for the overall mechanical response of the considered composite.
This Chapter is organized as follows. Section IV.2 provides description of the μCT data processing procedures. Statistical analysis of calculated pore geometrical parameters is presented in section IV.3. Section IV.4 introduces micromechanical parameters used for the quantification of pore contributions. Section IV.5 presents development and validation of pore compliance contribution statistical model. Section IV.6 provides the discussion of the results and conclusions.

IV.2. Microtomography data processing

The microtomography data considered in this Chapter was the same as the one discussed in section III.3.1 of this dissertation. It was obtained from the specimen of the CVI infiltrated C/C laminate of the size of 1x1x1 cm consisting of four unidirectional C/C layers ([0°/90°]s) 2.2 mm thick each, separated by 0.4 mm thick layers of chemical vapor infiltrated random felt. A schematic representation of the specimen including the choice of coordinate axes is shown in Fig.IV.2:

![Schematic of the scanned specimen including the coordinate axes and layer notations](image)

**Fig.IV.2.** Schematic of the scanned specimen including the coordinate axes and layer notations

The raw grayscale microtomography images contained information on all constituents: fibers, pyrolytic carbon and pores. For the purpose of this research, the images were binarized with predefined threshold to convert micro-CT data into series of black and white images with white regions representing pores and black – everything
else. The Visualization Toolkit file (VTK, http://www.vtk.org) format was chosen to store the black and white image series obtained from microtomography. The data contained 677 images representing evenly spaced slices along the Z direction, 678x685 pixels each, with 14.7 μm voxel unit length. They were then processed with a self-written MATLAB script as follows. VTK file is imported into MATLAB workspace resulting in a 3D matrix with dimensions equal to the dimensions of the VTK file – 678x685x677. Components in the matrix have integer values: '0' for the black color and '1' for the white color corresponding to the pores. The matrix is then processed using functions from MATLAB Image Processing Toolbox to obtain information about all connected objects (in our case, pores) in the input matrix. The information includes the number of objects and the coordinates of the components in the matrix comprising these objects. The criterion by which the components are determined to be connected to the objects is defined by the connectivity parameter. In our procedure we use the 6-connected neighborhood connectivity, meaning that the component is considered to be a part of the object if it is located in the same row or column as one of the object components and is adjacent to it. In the next part of the script, individual objects are processed to determine their geometric parameters. First, the triangular surface mesh of the pore is constructed in MATLAB. The vertex coordinates and the element connectivity matrix of surface elements of every pore are then used to calculate the inertia tensor, principal moments and principal directions, volume and surface area of the pore as follows.

The surface area of the pore is calculated as a sum of the areas of all triangular surface mesh elements comprising the pore. The area of the \(i\)-th triangular element is calculated as: \(S_i = \frac{|v_{i1} \times v_{i2}|}{2}\), where \(v_{i1}\) and \(v_{i2}\) are the vectors constructed from the vertices of the element. The pore volumes and inertia tensors are calculated using the method based on the mass properties of tetrahedral elements generated from the triangular surface mesh elements of the pores. To generate these elements, the fourth vertex can be chosen arbitrarily for as long as it is the same for all elements of the surface mesh. For simplicity, in our calculations this vertex was chosen to be at the origin of the
global coordinate system. This origin was defined during import of the μCT data into MATLAB to be located at the top left corner of the first image in the μCT data. The volume of the i-th element is calculated as: $V_i = \frac{\text{det}(L)}{6}$, where L is a 3x3 matrix consisting of components of the vectors connecting the vertex at the origin of coordinates to the vertices of the surface element (i.e. vertex coordinates of the surface element). To calculate the volume of a pore, all $V_i$ are summed. The tetrahedrons built on the surface elements with normal vectors directed towards the origin of the coordinates (fourth vertex) enter the sum with the minus sign, the volume elements built on the surfaces with normal vectors directed in the opposite direction enter the relation with the plus sign. The result of the overlapping tetrahedral volumes with opposite signs is the volume bounded by the surface mesh of the pore. For details on this method, see Zhang & Chen (2001).

The procedure of computing the mass properties of the volumes defined by triangular surface meshes is described in Blow & Binstock (2004). We introduce the inertia tensor represented in the matrix form as:

$$
I = \begin{bmatrix}
I_{xx} & I_{xy} & I_{xz} \\
I_{xy} & I_{yy} & I_{yz} \\
I_{xz} & I_{yz} & I_{zz}
\end{bmatrix},
$$

where $I_{xx}, I_{xy}, \ldots, I_{zz}$ are the moments of inertia of the considered 3D body in the $xyz$ coordinate system. This matrix can be decomposed as follows:

$$
I = \text{tr}C \cdot I_3 - C,
$$

where $I_3$ is the 3x3 identity matrix, $C$ is the covariance matrix defined as

$$
C = \int \rho \mathbf{x} \mathbf{x}^T d\mathbf{v} = \int \begin{bmatrix} x^2 & xy & xz \\
xy & y^2 & yz \\
xz & yz & z^2
\end{bmatrix} d\mathbf{v},
$$

where $\rho$ is the mass density (assumed to be unity in all subsequent calculations), $d\mathbf{v}$ is unit volume, $\mathbf{x} = [x, y, z]^T$ is the vector of global coordinates. In the text to follow, a
standard matrix notation is assumed with superscript "T" denoting a transpose of a matrix, and \( \mathbf{ab} \) being a matrix product of matrices or vectors \( \mathbf{a} \) and \( \mathbf{b} \). The idea of covariance is used in principal component analysis (Jolliffe (2002)). The method of Blow & Binstock (2004) is based on mapping of the canonical tetrahedron with vertices at \((0,0,0), (1,0,0), (0,1,0) \) and \((0,0,1)\) onto the one for which the covariance matrix needs to be calculated. The covariance matrix of the canonical tetrahedron is given by

\[
\mathbf{C}_C = \frac{\rho}{120} \begin{bmatrix} 2 & 1 & 1 \\ 1 & 2 & 1 \\ 1 & 1 & 2 \end{bmatrix}.
\] (IV.4)

Mapping of a canonical tetrahedron onto a given one results in the following relation for the covariance tensor:

\[
\mathbf{C}_i = \rho \int_V (\mathbf{Ax})(\mathbf{Ax})^T dV_A = \rho \int_V \mathbf{Ax}x^\top \mathbf{Ax}^T dV_C \det \mathbf{A} = \det(\mathbf{A}) \mathbf{C}_C \mathbf{A}^\top,
\]

where \( \mathbf{A} \) is the transformation matrix consisting of the vectors on which the tetrahedron is built (coincides with \( \mathbf{L} \) used in the element volume calculations), and \( V_C \) is the volume of the canonical tetrahedron. The multiplier \( \det(\mathbf{A}) \) comes from the distortion of the considered tetrahedron volume compared to the canonical one.

The covariance matrix of a pore is calculated as sum of covariance matrices of all its tetrahedrons: \( \mathbf{C}' = \sum_{i=1}^{N} \mathbf{C}_i \). At this stage, \( \mathbf{C}' \) is calculated relative to the origin of coordinates. With respect to the center of mass of the pore, the covariance matrix is given by

\[
\mathbf{C} = \mathbf{C}' + m \left( \Delta \bar{x} \Delta \bar{x}^\top + \bar{x} \Delta \bar{x}^\top + \Delta \bar{x} \Delta \bar{x}^\top \right),
\] (IV.5)

where \( m = \rho V \) is the total mass of the body, and \( \bar{x} \) is the position of center of mass of the pore found as \( \bar{x} = \frac{1}{V} \sum_{i=1}^{N} \bar{x}_i V_i \), \( \bar{x}_i = \frac{1}{4}(x_{i1} + x_{i2} + x_{i3} + x_{i4}) \), \( x_{ij} \) are the column vectors of the coordinates of the vertices comprising the \( i \)-th tetrahedral element, \( \Delta \bar{x} \) is the distance from the reference point of the initial covariance matrix to the center of mass.
Since the reference point was chosen at origin, $\Delta x$ is equal to $-\bar{x}$. When $C$ is known, we can compute the inertia matrix $I$ utilizing equation (IV.2). Eigenvalues and eigenvectors of the matrix $I$ are equal to the principal moments ($I_{11}$, $I_{22}$, $I_{33}$) and principal directions (1, 2, 3) correspondingly. For the obtained principal moments the convention $I_{11} < I_{22} < I_{33}$ is used, similar to Garboczi (2011). An example of orientations of the principal axes of inertia of a pore is provided in Fig.IV.3. MATLAB source files used for processing of $\mu$CT data are published at http://www.unh.edu/cc-composites/muct-processing/

![Fig.IV.3. Illustration of a pore geometry with principal axes of inertia (1,2,3) in the global coordinate system (x,y,z)](image)

### IV.3. Statistical processing of pore geometry dataset

#### IV.3.1. Pore volume distribution

The image processing procedure presented in section IV.2 resulted in 9,648 pores identified in the scanned specimen. However, not all of them were included in the dataset for statistical analysis. First, all pores adjacent to the external surfaces of the sample (858 pores) were removed from the initial set because they were cut during the specimen preparation. For the newly obtained dataset, the distribution of pore volumes
was analyzed by considering the cumulative empirical distribution function of pore volumes defined as:

\[ P(V) = \frac{1}{N_0} \sum_{i=1}^{N_0} \{ V_i \leq V \}, \quad \text{(IV.6)} \]

where \( N_0 \) is the total number of pores in the dataset and \( \sum_{i=1}^{N_0} \{ V_i \leq V \} \) is the number of pores with volume less than or equal to \( V \). The obtained plot of \( P(V) \) (Fig. IV.4a) shows two distinct slopes and a long tail at the upper end of the curve. This long tail is attributed to the presence of several “superpores” that either consist of an array of coalescent pores or many pores that were not distinguished as separate ones by the \( \mu \)CT data processing procedure. An example of such “superpore” is shown in Fig. IV.5. There is also a number of pores in the dataset that have only 1 voxel in one of the bounding box dimensions. The shape and orientation of these pores cannot be accurately determined with the given spatial resolution of \( \mu \)CT measurements. Thus, 2.5% tail from the beginning and 2.5% tail from the end of the distribution curve were removed from the analysis. This trimming resulted in a filtered dataset with \( N_f = 8,351 \) pores and pore volume range \( 2.9\,\text{vx} < V_i < 4.3\cdot10^1\,\text{vx} \), where \( \text{vx} \) is the volume element (voxel). Thus, 95% of the pores in the C/C sample have volumes between \( 9.2\cdot10^2 \) and \( 13.7\cdot10^6 \,\mu\text{m}^3 \). The cumulative pore volume distribution function of the filtered dataset is shown in Fig. IV.4b.

Fig. IV.4. Empirical distribution function for pore volumes: (a) before filtering; (b) after trimming 2.5% tails from each end.
Fig. IV.5. A cluster of coalescent pores treated as a single large pore by the pore extraction and labeling algorithm but excluded from the statistical pore analysis by the dataset filtering procedure

IV.3.2. Pore geometry and orientation distribution

We assume that orientation of a pore is given by vector $\mathbf{m}$ in the direction of its minor principal axis of inertia (the axis that corresponds to the smallest moment of inertia $I_{11}$, see Fig. IV.3). Vector $\mathbf{m}$ can be defined in a polar coordinate system as $\mathbf{m}(\theta, \phi)$ by two orientation angles (see Fig. IV.6): co-latitude ($0 \leq \theta \leq \pi$) and longitude ($0 \leq \phi \leq 2\pi$). It can be shown that for a slender pore with $I_{11} < I_{22} < I_{33}$, the principal axis $\mathbf{m}$ is aligned along the axis of the largest linear dimension. For the symmetrical bodies with $I_{11} \approx I_{22} \approx I_{33}$, the discussed approach would yield non-unique orientations of the principal axes which would be highly sensitive to the numerical and shape errors introduced by the finite resolution of \( \mu \)CT measurements.

The distribution of pore orientations is defined by the density of pore orientation vectors per angular area $dA(\theta, \phi)$, as shown in Fig. IV.6. The empirical spherical probability density function (ESPDF) is then given by

$$P(\theta, \phi) = \frac{1}{N} \sum_{i=1}^{N} \delta\left(\mathbf{m}_i(\theta, \phi) \in dA(\theta, \phi)\right),$$

(IV.7)
where \( \sum_{i=1}^{N} \{ \mathbf{m}_i(\theta, \phi) \in dA(\theta, \phi) \} \) is the number of pores with the orientation vector \( \mathbf{m}_i(\theta, \phi) \) that lie in the angular sector \( dA(\theta, \phi) \); and \( dA(\theta, \phi) \) is the angular sector with dimensions \( (d\theta, d\phi) \) on the surface of a unit sphere as illustrated by shaded gray in Fig.IV.6; \( N \) is the total number of pores in the considered dataset.

![Spherical coordinate system](image)

**Fig.IV.6.** Spherical coordinate system \((\theta, \phi)\) illustrated on the unit sphere. Orientation vector \( \mathbf{m} \) lies inside the differential angular sector \( dA(\theta, \phi) \) shaded in gray. Dimensions of the sector are \((d\theta, d\phi)\)

The issue of high sensitivity of vector \( \mathbf{m}(\theta, \phi) \) to the shape errors for small pores can be addressed by introducing the volume weighted ESPDF defined as:

\[
P_r(\theta, \phi) = \frac{1}{V_z} \sum_{i=1}^{N} \left[ V_i \cdot \{ \mathbf{m}_i(\theta, \phi) \in dA(\theta, \phi) \} \right],
\]

(IV.8)

where \( \sum_{i=1}^{N} \left[ V_i \cdot \{ \mathbf{m}_i(\theta, \phi) \in dA(\theta, \phi) \} \right] \) is the sum of volumes of pores with orientation vector \( \mathbf{m}_i(\theta, \phi) \) that lie in the angular sector \( dA(\theta, \phi) \), and \( V_z = \sum_{i=1}^{N} V_i \) is the total volume of all pores in the considered dataset. Note that for purposes of characterizing pore orientation distributions, there is no distinction between the positive and negative directions. The same pore can be described by either \( \mathbf{m} \) or \((-\mathbf{m})\) as its orientation.
vector. This fact was taken into account in the calculation of the ESPDFs \( P(\theta,\phi) \) and \( P_r(\theta,\phi) \).

The orientation distributions obtained for the unidirectional layers in X and Y directions, for the felt layers and for a full set of pores are plotted in Fig.IV.7. The plots represent the unweighted and volume-weighted ESPDFs calculated for equally spaced angular sectors with dimensions \( (d\theta = 10^\circ, \ d\phi = 10^\circ) \). For convenience, the sector boundaries are not shown and the data is smoothed using bicubic interpolation onto the equally spaced grid with dimensions \( (d\theta = 1^\circ, \ d\phi = 1^\circ) \). Also, due to the equivalency of \( m \) and \( (-m) \) orientations, the actual data is presented in the top hemisphere, and image of the bottom hemisphere is obtained by mirroring the top for visualization purposes.

Visual analysis of the plots allows to conclude that plots of the volume-weighted ESPDFs provide a clearer picture of the major orientations for all of the considered sets because of the reduced contribution of the orientations of small pores. Also, it can be observed that in the unidirectional layers X and Y pore orientations are coincident with the corresponding fiber orientations, exhibiting dependence of porosity on the morphology of preform.

The orientation distribution functions plotted in Fig.IV.7 provide input information for micromechanical modeling to predict anisotropic thermoeelastic properties of the composite. If heterogeneities (and, as a special case, pores) are approximated by ellipsoids, the famous Eshelby solution (Eshelby (1957), Eshelby (1959)) can be utilized in the appropriate micromechanical schemes, see Mura (1987) and Nemat-Nasser and Hori (1999) for convenient formulae, and Piat et al. (2006a) and Pettermann et al. (1997) for examples of applications. If more realistic pore shapes are assumed, the data on the orientation distribution can be combined, for example, with the cavity compliance contribution approach (Kachanov et al. (1994), Eroshkin and Tsukrov (2005)) or be used in generating representative unit cells with many pores for direct FEA simulations (as in Michailidis et al., 2010). Two limiting cases of pore orientation distributions, parallel and randomly oriented pores, are utilized as illustration in the following sections.
Fig IV.7. Original and volume weighted ESPDFs: (a) for a full set of pores; (b) for the X-UD layers 1 and 2, see Fig IV.2; (c) for the Y-UD layers 1 and 2; (d) for the felt layers 1,2 and 3
IV.4. Contribution of pores to the effective elastic properties

In the presentation below, we assume pores to be inserted in the homogenized isotropic material with Young's modulus $E_0$, bulk modulus $K_0$, and shear modulus $G_0$. A mixture of carbon fibers and PyC matrix in actual C/C composites is neither homogeneous nor isotropic, especially, for non-random fiber distributions. However, we utilize this assumption to emphasize contributions of various pore shapes without significant complication of the stochastic models. The more accurate approach would involve simultaneous multiscale modeling for various inhomogeneity types which is beyond the scope of this dissertation.

To evaluate contribution of individual pore shapes to the effective material properties we make use of the 5 dimensionless parameters: $\tilde{E}_1$, $\tilde{E}_2$, $\tilde{E}_3$, $\tilde{K}$, $\tilde{G}$ introduced in Chapter III. In the case of some regular pore shapes, the elasticity problem to find these parameters for a single pore can be solved analytically. For the irregular pore shapes observed in C/C, the compliance contribution tensors can be calculated by FEA as described in Chapter III. In analysis to follow, for consistency, the pore meshes are rotated so that their principal axes are aligned with the global coordinate system such that the longest dimension is aligned with X-axis and the shortest with Z-axis.

IV.5. Development and validation of pore compliance contribution statistical model

IV.5.1. Design of experiments approach

Micromechanical modeling procedure described in Chapter III provides an approach to evaluating contribution of pores to the overall elastic response utilizing FEA simulations for each pore shape. However, using this approach to model real-world material systems which may contain a large number of irregularly-shaped pores with high variability in geometric parameters and orientation is infeasible due to very high computational costs. A possible approach to account for variation of pore geometries is
to develop a stochastic model which takes pore geometric parameters as input variables and provides pore compliance contribution (PCC) parameters as an output.

One of the ways to develop such a model would be to perform FEA simulations on a large set of irregular pores with various geometries. Then, using statistical methods such as linear or non-linear regression, the obtained data can be processed to construct an empirical formula (usually a multivariate polynomial) that relates the input variables (pore geometry parameters) to their contribution to effective elastic properties. For the model to be accurate, a very large dataset is usually required to get good estimates (narrower confidence intervals) of the model coefficients and predictions.

To minimize the number of simulations required for construction of a statistical model, the design of experiments (DoE) approach can be applied. A special software tool (such as MATLAB, SAS, SAS JMP, etc.) is used to select the most efficient set of design points (combinations of input parameters) from the full design space (total number of possible combinations, or, in our case, total set of all pores), such that with the minimal number of simulations a good prediction model can be obtained. For the development of PCC model, we chose to construct and run the I-optimal design using DoE module in JMP software (SAS Institute Inc (2010)). The optimization criterion for I-optimal designs is minimization of the integrated variance of the model predictions over the entire design space. In other words, the width of confidence intervals for predicted values should not vary significantly across the design space. It should be noted that the width of confidence intervals grows in the vicinity of the design space boundaries (extreme values of design parameters).

After calculating the PCC factors for pores corresponding to the selected design points, their values were processed utilizing the Response Surface Methodology (Myers et al. (2009), Ryan (2007)) to develop the stochastic models of pore contributions to effective elastic moduli. The result is presented as a multivariate polynomial model. From the practical standpoint, there is always a trade-off when choosing between flexibility of the model (order of the polynomial), power of the model (confidence limits)
and the number of experiments required for the analysis. The second-order model was used because of its ability to capture nonlinearities in the response with a feasible number of experiments to get high-quality predictions.

IV.5.2. Design space parameters selection

Based on the previous studies (Taylor et al. (2006), Erdogan et al. (2007)), the following parameters were chosen as input for the development of the PCC model of an isotropic matrix material with irregularly shaped pores: the principal moments of inertia (PMIs) of the pore shapes \((I_{11}, I_{22}, I_{33})\) and the Poisson’s ratio of the matrix \((\nu)\). Since the values of moments of inertia are size-dependent, the ratios of PMIs \(I_{11}/I_{33}\) and \(I_{22}/I_{33}\) were considered to separate contribution of pore shapes from their volumes. The range of values for the input parameters was established using the information obtained in section IV.2. Similarly to the approach of determining the range of pore volumes, the empirical distribution functions for each of the parameters \((I_{11}/I_{33}\) and \(I_{22}/I_{33}\)) were analyzed and the range was chosen between the values corresponding to the 2.5% and 97.5% points of the curve. The range of values for the Poisson’s ratio was chosen to be \(0.0 - 0.4\), which includes values for carbon/carbon composite materials, as well as other types of ceramics and metals. The ranges of the input variables are summarized in Table IV.1. It should be noted that within the chosen range of parameters \(I_{11}/I_{33}\) and \(I_{22}/I_{33}\), some of the combinations of values are incompatible due to the properties of PMI. The values of PMI are limited by two constraints as illustrated in Fig. IV.8. The first constraint, given by the positive slope line in the plot, comes from the sorting of PMI according to \(I_{33} > I_{22} > I_{11}\). The second constraint is defined as \(I_{11}/I_{33} + I_{22}/I_{33} \geq 1\) and shown as the negative slope line in the plot. This is an inherent property of the PMI as can be shown by the generalized perpendicular axes theorem (McKelvey (1983)).
Table IV.1. The ranges of input variables for the I-optimal custom designed experiment

<table>
<thead>
<tr>
<th></th>
<th>Minimum value</th>
<th>Maximum value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$I_{11}/I_{33}$</td>
<td>0.10</td>
<td>0.70</td>
</tr>
<tr>
<td>$I_{22}/I_{33}$</td>
<td>0.67</td>
<td>0.98</td>
</tr>
<tr>
<td>$\nu$</td>
<td>0.00</td>
<td>0.40</td>
</tr>
</tbody>
</table>

To construct an I-optimal custom designed experiment, the following parameters were entered into the DoE module of JMP software:

- Input variables ($I_{11}/I_{33}$, $I_{22}/I_{33}$ and $\nu$) specified as continuous variables with ranges as shown in Table IV.1
- Constraints for input variables: $I_{22}/I_{33} \geq I_{11}/I_{33}$, $I_{11}/I_{33} + I_{22}/I_{33} \geq 1$
- All model responses without optimization goals: $\bar{E}_1$, $\bar{E}_2$, $\bar{E}_3$, $\bar{K}$, $\bar{G}$
- Prediction model type was set to the $2^{nd}$ order Response Surface Model
- Number of experimental runs (design points): 48
- Number of center points: 5
- Number of replicates: 0
- Number of random starts for search of optimal designs: 1,000

Note that the numbers of experimental runs and center points were chosen based on the analysis of the design evaluation parameters "Prediction Variance Profile", "Prediction Variance Surface" and "Average Variance of Prediction" provided by the JMP software. Center points are the experimental runs with all input variables set to the mean values of input ranges. These points are required for the 2nd order models to establish model curvature with respect to each of the input parameters (2nd order terms of the model).

The calculations resulted in a design table (see Table IV.2) containing combinations of input variables for each of the experimental runs. Using this table, geometries with the desired values of $I_{11}/I_{33}$ and $I_{22}/I_{33}$ were selected from the total dataset of pores obtained in section IV.2. Since the number of pores in the dataset is finite, it was impossible to find pores with parameters that exactly match the values of the input parameters. Therefore, the pores with the smallest deviation from these values were chosen. Average differences between the actual and design parameters were -0.0008 and 0.0084 for $I_{11}/I_{33}$ and $I_{22}/I_{33}$, respectively. Root mean square errors (RMSE) were found to be 0.0307 for $I_{11}/I_{33}$, and 0.0325 for $I_{22}/I_{33}$.

Once the experimental pore set was selected and pore surface meshes were extracted using the procedure described in section IV.2, a self-written MATLAB script was employed to create FEA models for MSC Marc/Mentat 2010 software (http://www.mscsoftware.com) and process the output files to determine pore compliance contribution parameters $\tilde{E}_1$, $\tilde{E}_2$, $\tilde{E}_3$, $\tilde{K}$, $\tilde{G}$. For details on the FEA and post-processing procedures see Chapter III.
### Table IV.2. Design table for the 3-factor designed experiment

<table>
<thead>
<tr>
<th>#</th>
<th>$I_{11}/I_{13}$</th>
<th>$I_{22}/I_{33}$</th>
<th>$\nu$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.100</td>
<td>0.900</td>
<td>0.2</td>
</tr>
<tr>
<td>2</td>
<td>0.100</td>
<td>0.900</td>
<td>0.4</td>
</tr>
<tr>
<td>3</td>
<td>0.100</td>
<td>0.900</td>
<td>0.4</td>
</tr>
<tr>
<td>4</td>
<td>0.100</td>
<td>0.980</td>
<td>0.0</td>
</tr>
<tr>
<td>5</td>
<td>0.100</td>
<td>0.980</td>
<td>0.0</td>
</tr>
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<td>6</td>
<td>0.100</td>
<td>0.980</td>
<td>0.2</td>
</tr>
<tr>
<td>7</td>
<td>0.100</td>
<td>0.980</td>
<td>0.4</td>
</tr>
<tr>
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<td>0.831</td>
<td>0.0</td>
</tr>
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<td>0.169</td>
<td>0.831</td>
<td>0.0</td>
</tr>
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<td>0.831</td>
<td>0.2</td>
</tr>
<tr>
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<td>0.330</td>
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<td>0.0</td>
</tr>
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<td>0.330</td>
<td>0.670</td>
<td>0.0</td>
</tr>
<tr>
<td>13</td>
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### IV.5.3. 3-factor designed experiment results

Results of the FEA simulations for the 3-factor designed experiment defined in Table IV.2 were processed and entered into the JMP software for statistical analysis. The “Fit Model” module was used with the following options:

- Standard least squares model
- Response variables defined as: $\ddot{E}_1$, $\ddot{E}_2$, $\ddot{E}_3$, $\dddot{K}$, $\dddot{G}$
• Input variables \( I_1 / I_3, I_2 / I_3 \) and \( \nu \) defined as the 2nd order “Response Surface Model Effects”

The results of the statistical analysis are presented in Fig.IV.10 as plots of actual vs predicted values. In these plots, the straight line represents the perfect fit when the predicted values coincide with the actual values \( (Actual = Predicted) \). The two curves below and above straight line represent the confidence intervals for the predicted values. After visual inspection, the outlier data point shown as an empty circle in the graph was removed and the model was rerun to improve the accuracy of predictions. Then the parameter estimates for each of the responses were inspected to determine which of the parameters were statistically significant. In the present study the significance level of 90% was used as a critical value. Insufficient parameters were excluded sequentially by removing one insignificant estimate at a time and re-running the model. It should be noted that exclusion of model effects obeys hierarchy of input variables in such a way that if a second-order effect or one of the interactions of a specific input variable is significant but the input variable by itself is determined as insignificant, it cannot be excluded from the analysis. Plots shown in Fig.IV.10 represent model predictions with excluded statistically insignificant model effects.

Accuracy of the model predictions was characterized using two indicators, \( R^2 \) and \( RMSE \). For the ideal model (perfect correlation with the experiment), \( R^2 = 1 \) and \( RMSE = 0 \). In contrast, for an inadequate model, points on the plot will be randomly scattered with no trend or a trend different from \( Actual = Predicted \) \( (R^2 \rightarrow 0, \ RMSE \gg 0) \). In an effort to improve accuracy of the proposed PCC model, visual analysis of the Actual vs Predicted plot (Fig.IV.10) was performed for each of the estimated responses. It was observed that in the midranges for responses \( \tilde{E}_1, \tilde{E}_2, \tilde{E}_3 \) there is a significant number of points which fall beyond the confidence intervals. These points correspond to pores with very similar predicted response, but different values of the actual response.
An example of two pores with virtually the same predicted response and input parameters $I_{11}/I_{33}$, $I_{22}/I_{33}$, and $\nu$ is shown in Fig. IV.9. Pore "a" is indicated on the plots (Fig.IV.10) as an empty square, and pore "b" as an empty triangle. Comparing the shape features of these two pores, it appears that pore "b" has more surface features and looks less convex than pore "a". One of the ways to account for this difference would be to introduce in the model a non-dimensional surface area to volume ratio $S_v = S^{12}/V^{13}$, which is analogous to one of the shape coefficients in Rauch et al. (2012) or the sphericity parameter defined by Wadell as $\pi^{10}(6\nu)^{22}/S$ (Wadell (1935)). The range of values for $S_v$ parameter was chosen using the same approach as for pore volumes and moments of inertia. The empirical distribution function for $S_v$ was analyzed and the range was chosen between the values corresponding to the 2.5% and 97.5% points of the curve (see Table IV.3).

Fig. IV.9. Two pores with very similar geometric parameters $I_{22}/I_{11}$, $I_{33}/I_{11}$, but noticeably different mechanical responses

IV.5.4. 4-factor designed experiment results

For the development of a PCC model incorporating the $S_v$ factor, we constructed a new 4-factor experimental design using the DoE module of JMP software with the following parameters:
- Input variables \((I_{11}/I_{33}, I_{22}/I_{33}, \nu \text{ and } S_{\nu})\) specified as continuous variables with ranges as shown in Table IV.3
- Constraints for input variables: \(I_{22}/I_{33} \geq I_{11}/I_{33}, I_{11}/I_{33} + I_{22}/I_{33} \geq 1\)
- All model responses without optimization goals: \(\bar{E}_1, \bar{E}_2, \bar{E}_3, \bar{K}, \bar{G}\)
- Prediction model type was set to the 2nd order Response Surface Model
- Number of experimental runs (design points): 145
- Number of center points: 5
- Number of replicates: 0
- Number of random starts for search of optimal designs: 1,000

Table IV.3. The ranges of input variables for the 4-factor designed experiment

<table>
<thead>
<tr>
<th>Input Variable</th>
<th>Minimum value</th>
<th>Maximum value</th>
<th>Average value (AVG)</th>
<th>Midrange value (MID)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(I_{11}/I_{33})</td>
<td>0.10</td>
<td>0.70</td>
<td>0.40</td>
<td>0.30</td>
</tr>
<tr>
<td>(I_{22}/I_{33})</td>
<td>0.67</td>
<td>0.98</td>
<td>0.825</td>
<td>0.155</td>
</tr>
<tr>
<td>(S_{\nu})</td>
<td>2.30</td>
<td>3.30</td>
<td>2.80</td>
<td>0.50</td>
</tr>
<tr>
<td>(\nu)</td>
<td>0.00</td>
<td>0.40</td>
<td>0.20</td>
<td>0.20</td>
</tr>
</tbody>
</table>

The design space of the experiment is presented in Table IV.4. Similarly to the previous design, it was impossible to find pores with exactly matching parameters, and pores with the smallest deviations in parameters were selected. Average deviations of the design parameters were -0.0039 for \(I_{11}/I_{33}\), 0.0071 for \(I_{22}/I_{33}\), and 0.0129 for \(S_{\nu}\). The root mean square errors (RMSE) were calculated as 0.0516, 0.0619 and 0.1562 for \(I_{11}/I_{33}\), \(I_{22}/I_{33}\) and \(S_{\nu}\) respectively.

Once the experimental pore set was selected and pore surface meshes were extracted, FEA simulations were performed for all pores and the results were imported into JMP software. The following options were selected in the "Fit Model" analysis module:
• Standard least squares model  
• Response variables defined as: \( \bar{E}_1, \bar{E}_2, \bar{E}_3, \bar{K}, \bar{G} \)  
• Input variables \( I_{11}/I_{33}, I_{22}/I_{33}, S_\nu \) and \( \nu \) defined as the 2nd order "Response Surface Model Effects"

The results of statistical modeling are presented in Fig.IV.11. It can be seen that the overall fit of the 4-factor model is better than that of the 3-factor model. This is evidenced by the increased values of \( R^2 \) shown in Table IV.5. The scatter has not changed significantly (similar RMSE values) for all model responses except \( \bar{K} \), for which an increased scatter was observed. Also, due to the larger number of experimental runs, the new model has narrower confidence intervals for predicted values of pore compliance contribution parameters. Goodness of fit (\( R^2 \) and RMSE values) for both models is summarized in Table IV.5.

The parameter estimates of the 4-factor PCC model are presented in Table IV.6. These parameters are coefficients in the polynomial model predicting dependence of pore contributions to elastic properties on the chosen geometric factors:

\[
PCC = \alpha_0 + \alpha_1 \bar{I}_{11} + \alpha_2 \bar{I}_{22} + \alpha_3 \bar{S}_\nu + \alpha_4 \bar{\nu} + \alpha_5 \bar{I}_{11}^2 + \alpha_6 \bar{I}_{11} \bar{I}_{22} + \alpha_7 \bar{I}_{22} + \alpha_8 \bar{I}_{11} \bar{S}_\nu + \\
+ \alpha_9 \bar{I}_{22} \bar{S}_\nu + \alpha_{10} \bar{S}_\nu^2 + \alpha_{11} \bar{I}_{11} \bar{\nu} + \alpha_{12} \bar{I}_{22} \bar{\nu} + \alpha_{13} \bar{S}_\nu \bar{\nu} + \alpha_{14} \bar{\nu}^2
\]

where \( PCC \) is one of the following parameters \( \bar{E}_1, \bar{E}_2, \bar{E}_3, \bar{K} \) or \( \bar{G} \) characterizing pore contribution to effective Young’s moduli, bulk modulus and shear modulus; and the input factors are normalized to the range \([-1; 1]\) based on the values provided in Table IV.3:

\[
\bar{I}_{11} = \frac{I_{11}/I_{33} - AVG(I_{11}/I_{33})}{MID(I_{11}/I_{33})}, \quad \bar{I}_{22} = \frac{I_{22}/I_{33} - AVG(I_{22}/I_{33})}{MID(I_{22}/I_{33})}, \\
\bar{S}_\nu = \frac{S_\nu - AVG(S_\nu)}{MID(S_\nu)}, \quad \bar{\nu} = \frac{\nu - AVG(\nu)}{MID(\nu)}.
\]
Fig. IV.10. Results of the 3-factor experiment. Actual vs predicted plots for PCC parameters
(a) $\tilde{E}_1$, (b) $\tilde{E}_2$, (c) $\tilde{E}_3$, (d) $\tilde{K}$, (e) $\tilde{G}$
Table IV.4. Design table for the 4 factor designed experiment.

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<td>46</td>
<td>0.192</td>
<td>0.808</td>
<td>3.100</td>
<td>0.4</td>
<td>0.2</td>
</tr>
</tbody>
</table>

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Fig. IV.11. Results of the 4-factor experiment. Actual vs predicted plots for PCC parameters
(a) $\tilde{E}_1$, (b) $\tilde{E}_2$, (c) $\tilde{E}_3$, (d) $\tilde{K}$, (e) $\tilde{G}$
### Table IV.5. Accuracy of model predictions of 3-factor and 4-factor PCC models.

<table>
<thead>
<tr>
<th>Model response</th>
<th>3-factor PCC model</th>
<th>4-factor PCC model</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( R^2 )</td>
<td>( RMSE )</td>
</tr>
<tr>
<td>( \tilde{E}_1 )</td>
<td>0.56</td>
<td>0.10</td>
</tr>
<tr>
<td>( \tilde{E}_2 )</td>
<td>0.73</td>
<td>0.11</td>
</tr>
<tr>
<td>( \tilde{E}_3 )</td>
<td>0.80</td>
<td>0.26</td>
</tr>
<tr>
<td>( \tilde{K} )</td>
<td>0.99</td>
<td>0.12</td>
</tr>
<tr>
<td>( \tilde{G} )</td>
<td>0.90</td>
<td>0.08</td>
</tr>
</tbody>
</table>

### Table IV.6. Parameter estimates for 5 responses (contributions to elastic moduli) of the 4-factor PCC model. Standard errors are given in parentheses.

<table>
<thead>
<tr>
<th>Coeff.</th>
<th>Term</th>
<th>( \tilde{E}_1 )</th>
<th>( \tilde{E}_2 )</th>
<th>( \tilde{E}_3 )</th>
<th>( \tilde{K} )</th>
<th>( \tilde{G} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \alpha_0 )</td>
<td>Intercept</td>
<td>1.673 (0.013)</td>
<td>1.997 (0.018)</td>
<td>3.077 (0.033)</td>
<td>2.484 (0.030)</td>
<td>2.226 (0.011)</td>
</tr>
<tr>
<td>( \alpha_1 )</td>
<td>( \tilde{I}_{11} )</td>
<td>0.466 (0.020)</td>
<td>0.291 (0.026)</td>
<td>-1.122 (0.074)</td>
<td>-0.487 (0.051)</td>
<td>-0.155 (0.015)</td>
</tr>
<tr>
<td>( \alpha_2 )</td>
<td>( \tilde{I}_{22} )</td>
<td>0.161 (0.017)</td>
<td>0.405 (0.023)</td>
<td>-0.857 (0.064)</td>
<td>-0.349 (0.043)</td>
<td>-0.107 (0.014)</td>
</tr>
<tr>
<td>( \alpha_3 )</td>
<td>( \tilde{S}_y )</td>
<td>0.242 (0.014)</td>
<td>0.125 (0.018)</td>
<td>0.265 (0.053)</td>
<td>0.339 (0.034)</td>
<td>0.200 (0.013)</td>
</tr>
<tr>
<td>( \alpha_4 )</td>
<td>( \tilde{\nu} )</td>
<td>0.010 (0.009)</td>
<td>-0.008 (0.012)</td>
<td>-0.174 (0.033)</td>
<td>1.884 (0.024)</td>
<td>-0.274 (0.009)</td>
</tr>
<tr>
<td>( \alpha_5 )</td>
<td>( \tilde{I}<em>{11}^{\tilde{I}</em>{22}} )</td>
<td>-0.089 (0.023)</td>
<td>-0.207 (0.031)</td>
<td>0.369 (0.101)</td>
<td>0.215 (0.057)</td>
<td>0</td>
</tr>
<tr>
<td>( \alpha_6 )</td>
<td>( \tilde{I}_{11}^{\tilde{S}_y} )</td>
<td>-0.212 (0.028)</td>
<td>-0.267 (0.038)</td>
<td>0.859 (0.096)</td>
<td>0.502 (0.064)</td>
<td>0.120 (0.019)</td>
</tr>
<tr>
<td>( \alpha_7 )</td>
<td>( \tilde{I}_{22}^{\tilde{S}_y} )</td>
<td>-0.072 (0.022)</td>
<td>0.053 (0.029)</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>( \alpha_8 )</td>
<td>( \tilde{I}_{11}^{\tilde{S}_y} )</td>
<td>0</td>
<td>0</td>
<td>-0.212 (0.083)</td>
<td>0</td>
<td>-0.074 (0.017)</td>
</tr>
<tr>
<td>( \alpha_9 )</td>
<td>( \tilde{I}_{22}^{\tilde{S}_y} )</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>( \alpha_{10} )</td>
<td>( \tilde{S}_y^{\tilde{S}_y} )</td>
<td>-0.118 (0.026)</td>
<td>-0.125 (0.036)</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>( \alpha_{11} )</td>
<td>( \tilde{I}_{11}^{\tilde{\nu}} )</td>
<td>0</td>
<td>0</td>
<td>0.101 (0.056)</td>
<td>-0.155 (0.041)</td>
<td>0.044 (0.015)</td>
</tr>
<tr>
<td>( \alpha_{12} )</td>
<td>( \tilde{I}_{22}^{\tilde{\nu}} )</td>
<td>0</td>
<td>0</td>
<td>0.105 (0.058)</td>
<td>-0.125 (0.043)</td>
<td>0.037 (0.016)</td>
</tr>
<tr>
<td>( \alpha_{13} )</td>
<td>( \tilde{S}_y^{\tilde{\nu}} )</td>
<td>0</td>
<td>0</td>
<td>-0.111 (0.054)</td>
<td>0.273 (0.040)</td>
<td>-0.060 (0.015)</td>
</tr>
<tr>
<td>( \alpha_{14} )</td>
<td>( \tilde{\nu}^{\tilde{\nu}} )</td>
<td>-0.062 (0.014)</td>
<td>-0.097 (0.019)</td>
<td>0</td>
<td>1.199 (0.035)</td>
<td>-0.025 (0.013)</td>
</tr>
</tbody>
</table>
IV.5.5. Validation of the 4-factor PCC model and its comparison with approximation of pore shapes by ellipsoids

For validation of the proposed stochastic 4-factor PCC model, we compared the model estimates to the direct FEA simulations on a new set of 150 pores which were not used for the model construction. Pore geometries were selected from the original dataset with all available pores. For this experiment, the design space was constructed using uniform distribution of the input variables within the boundaries given in Table IV.3. The results are shown in Fig.IV.12. For all five responses, the model shows good prediction characterized by the low bias and moderate scatter (Table IV.7, the first two columns). It can be observed that RMSE values for all responses are similar to those obtained for the original dataset. Low mean error (ME) and moderate RMSE values allow to conclude that the model is satisfactory for the new dataset.

In another study, the accuracy of approximations of pore shapes by ellipsoids was evaluated by comparing their response with direct FEA calculations for the materials with irregular shapes. Approximation of irregular shapes by ellipsoids was performed based on the values of principal moments of inertia and volumes as suggested, for example, in Borbely et al. (2004) and Erdogan et al. (2007). Assuming mass density equal to unity, the expressions for the ellipsoids’ semi-axes are:

\[ a = \sqrt{\frac{5 I_{22} + I_{33} - I_{11}}{2V}}, \quad b = \sqrt{\frac{5 I_{33} + I_{11} - I_{22}}{2V}}, \quad c = \sqrt{\frac{5 I_{11} + I_{22} - I_{33}}{2V}}, \]  

(IV.11)

where \( I_{11}, I_{22}, I_{33} \) are the principal moments of inertia of the considered pore (see section IV.2), and \( V \) is the volume of the pore. The semi-axis \( a \) is aligned with the principal direction 1, \( b \) with direction 2, and \( c \) with direction 3. The pore compliance contribution parameters for ellipsoids were calculated using Eshelby solutions (Eshelby (1957), Eshelby (1959)) as described in Kachanov et al. (2003), and formulas presented in Chapter III.
The results of the comparison are presented in Fig. IV.13. It can be observed that the method of approximating ellipsoids shows larger scatter and higher bias than the proposed PCC approach (Table IV.7). Thus, for the considered dataset, approximation of irregular shapes by ellipsoids based on the principal moments of inertia produces less accurate predictions than utilization of the proposed 4-factor PCC model. Note that approximations by ellipsoids seem to underpredict values of $E_1$, $E_2$, and $K$, which means that material with irregularly shaped pores will have lower values of the corresponding elastic parameters $E_1$, $E_2$, and $K$ (see equations (III.10) and (III.12)) as compared to the material with ellipsoidal pores.
Fig. IV.12. Results of the validation study of the 4-factor experiment. Actual vs predicted plots for PCC parameters (a) $\bar{E}_1$, (b) $\bar{E}_2$, (c) $\bar{E}_3$, (d) $\bar{K}$, (e) $\bar{G}$
Fig. IV.13. Comparison of the pore approximations by ellipsoids with FEA calculations for actual shapes. Actual vs predicted plots for PCC parameters (a) $\bar{E}_1$, (b) $\bar{E}_2$, (c) $\bar{E}_3$, (d) $\bar{K}$, (e) $\bar{G}$
Table IV.7. Accuracy of model predictions for the validation studies.

<table>
<thead>
<tr>
<th>Model response</th>
<th>4-factor PCC model</th>
<th>Ellipsoidal Approximation Model</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>ME</td>
<td>RMSE</td>
</tr>
<tr>
<td>$\tilde{E}_1$</td>
<td>-0.06</td>
<td>0.14</td>
</tr>
<tr>
<td>$\tilde{E}_2$</td>
<td>-0.06</td>
<td>0.16</td>
</tr>
<tr>
<td>$\tilde{E}_3$</td>
<td>0.05</td>
<td>0.31</td>
</tr>
<tr>
<td>$\tilde{K}$</td>
<td>0.08</td>
<td>0.30</td>
</tr>
<tr>
<td>$\tilde{G}$</td>
<td>-0.02</td>
<td>0.08</td>
</tr>
</tbody>
</table>

IV.6. Discussion and conclusions of the statistical approach to evaluation of pore contributions on the mesoscale

Processing of µCT data on porosity in C/C shows that orientation of pores can be characterized by directions of their principal axes of inertia. As can be seen in Fig.IV.7, the pore orientation distributions are highly dependent on the morphology of carbon fiber preform.

Contribution of pores to the effective elastic properties can be estimated using a stochastic model based on their geometric parameters including ratios of their principal moments of inertia ($I_{11} / I_{13}$, $I_{22} / I_{13}$). Analysis shows that an improved accuracy can be achieved by incorporating a non-dimensionalized surface-to-volume ratio ($S_v$) as one of the model input variables.

The major result of this work is the 4-factor pore compliance contribution model given by equation (IV.9) and Table IV.6. For the dataset of pores considered in this Chapter, the model provides better predictions of the material's effective elastic properties than the approach based on approximation of pore shapes by ellipsoids, as can be seen in Fig.IV.12, Fig.IV.13 and Table IV.7. The potential improvements of the model can be achieved by identifying other statistically significant factors which characterize pore shapes and their distribution.
V. EXPERIMENTAL TESTING OF C/C COMPOSITES

In this section tension and compression testing performed on C/C composite felt specimens is presented. Overall, three specimens were tested to measure Young's moduli and Poisson's ratios of the material in three perpendicular directions. The small number of the test specimens is the result of the limited availability of C/C composite specimens due to their high cost.

V.1. Specimen preparation

The original felt specimen (see Fig. V.1) was provided by the Institute of Materials Science at Karlsruhe Institute of Technology (Germany). The specimen was roughly cut by a diamond saw and then finished at the UNH Kingsbury Machine Shop to ensure that the opposite sides are parallel to each other, which is crucial for compression tests. Three specimens were cut from the original specimen to determine Young's moduli and Poisson's ratios in three perpendicular directions (see Fig. V.2). The specimens were denoted as: “specimen 11”, “specimen 22” and “specimen 33” to measure properties in the first, second and third directions correspondingly. The final dimensions of the specimens are summarized in Table V.1.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Dimension 1, mm</th>
<th>Dimension 2, mm</th>
<th>Dimension 3, mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>11</td>
<td>28.1</td>
<td>13.0</td>
<td>14.4</td>
</tr>
<tr>
<td>22</td>
<td>14.6</td>
<td>33.6</td>
<td>14.6</td>
</tr>
<tr>
<td>33</td>
<td>14.6</td>
<td>12.4</td>
<td>14.7</td>
</tr>
</tbody>
</table>
Two types of the strain gage rosettes were selected for the testing:

- Vishay PG Micro-Measurements CEA-06-250WQ-350, which corresponds to the series of two stacked strain gage rosettes with nominal resistance of $350\Omega \pm 0.3\%$, gage length of 0.25" (6.35 mm), self-temperature-compensation factor of 6 (designed for steel specimens), and strain gage factor of 2.12;
- Vishay PG Micro-Measurements CEA-06-375UW-350, which corresponds to a single strain gage with nominal resistance of $350\Omega \pm 0.3\%$, gage length of 0.375" (9.53 mm), self-temperature-compensation factor of 6 (designed for steel specimens), and strain gage factor of 2.12.

Note, that since there was no significant temperature change in the course of the experiment, the temperature compensation factors did not affect the results of the experiment.

The strain gages were bonded (using M-BOND 200 adhesive) to the prepared surfaces of the specimens according to the instructions provided by the manufacturer (Vishay PG website: http://vishaypg.com/micro-measurements/videos). The resistance of the strain gages with lead wires was checked using a digital six-digit multimeter and found to be $350\pm1\ \Omega$ which is within the limits provided by the manufacturer.

V.2. Uniaxial compression testing of C/C composite felt specimens

V.2.1. Experimental setup and data processing

All experiments were performed at the UNH Kingsbury Manufacturing Lab on the universal testing machine INSTRON 1350 with a servohydraulic load frame and a digital controller. Force measurements were obtained using a foil strain gage force sensor with the capacity of 20,000 lbs (100 kN). The compression experimental setup is shown in Fig. V.3.

After the application of the strain gage rosettes the specimens with bonded strain gages were positioned in the center of the bottom compression platen (mounted on the spherical support to adjust for any misalignment present in the system). Strain gage lead wires (triple wires twisted to eliminate self-induced currents) were connected to the Wheatstone bridges (quarter-bridge configuration) and then the data acquisition system was started to check the wiring. The bridges were assembled from the precision resistors
and attached to the data acquisition board Keithley KUSB-3108. The power was supplied by the DC power source with adjustable voltage. The data was recorded using the LabView software set to 1,000 Hz with sequential acquisition of the input channels which included: excitation voltage, four strain gage signals and load sensor signal. High acquisition rate was chosen to improve resolution and reduce high-frequency noise during the data processing state via oversampling method (Meyer-Baese (2007)). The excitation voltage was chosen according to the guidelines provided by the strain gage manufacturer and set to 2 V to avoid self-heating of the strain gages. The input gain of 1 for excitation voltage and load sensor channels, and an input gain of 500 (maximum value) for the strain gage channels were selected.

![Fig. V.3. Compression testing setup](image)

The testing was performed in displacement control mode with strain rate of $3 \cdot 10^{-3} / s$. The obtained data was processed in two steps. First, the raw voltage data was input into MATLAB and smoothed using an averaging filter with a window of 100
samples, thus transforming the output frequency to 10 Hz to reduce high-frequency noise and improve resolution (Meyer-Baese (2007)). In the next step, the tabulated data was imported into Microsoft Excel and trimmed to the actual duration of the experiment. The trimmed dataset was used to produce the plots and perform the linear regression analysis. As a result, Young’s modulus and Poisson’s ratio were determined.

Voltage values were converted into the engineering strain values using the simplified formulas for the quarter-bridge strain gage configuration (Sharpe (2008)):

$$\varepsilon' = \frac{4 \Delta V}{GF \cdot V_{ex}}$$

where $\Delta V$ is the strain gage bridge voltage, $V_{ex}$ is the excitation voltage, $GF = 2.12$ is the strain gage factor.

Note that this formulation does not account for nonlinearity of the quarter-bridge configuration and does not compensate for the temperature and transverse sensitivity effects. To account for nonlinearity (required for large strains $\varepsilon > 0.1\%$) the following formula was used:

$$\varepsilon = \frac{2\varepsilon'}{2 - GF \cdot \varepsilon'}$$

where $\varepsilon'$ is the measured strain, $F$ is the strain gage factor, $\varepsilon$ is the corrected strain.

In the Poisson’s ratio measurements after nonlinearity correction, transverse sensitivity correction (TS) was also applied. TS effects occur due to mismatch of the Poisson’s ratio of the material used for strain gage calibration and the material to which the strain gage is applied. The calibration material’s Poisson’s ratio was that of steel $\nu_s = 0.285$ for all strain gages used in the experiment. The following formulas were utilized to correct for TS:

$$\varepsilon_1 = \varepsilon'_1 \frac{1 - \nu_s K_n}{1 - K_{n1} K_{12}} - K_{n2} \varepsilon'_1 \frac{1 - \nu_s K_{12}}{1 - K_{n1} K_{12}}$$

$$\varepsilon_2 = \varepsilon'_2 \frac{1 - \nu_s K_{12}}{1 - K_{n1} K_{12}} - K_{n2} \varepsilon'_2 \frac{1 - \nu_s K_n}{1 - K_{n1} K_{12}}$$

(V.2)
where $\varepsilon'_1$ and $\varepsilon'_2$ are measured strains (before the correction), $\varepsilon_1$ and $\varepsilon_2$ are corrected strains, $K_{n1} = 0.004$ and $K_{n2} = 0.004$ are transverse sensitivity correction factors of the strain gages in direction 1 and 2, and $\nu$ is the Poisson’s ratio of the calibration material.

Force values were converted to the engineering stress values using the following formula:

$$\sigma = \frac{F}{A'}$$

where $F$ is the applied force, and $A$ is the initial cross-sectional area.

**V.2.2. Results of compression testing**

The resulting stress-strain plots are shown in Fig. V.4-Fig. V.9. In all plots, SG-1,...,SG-4 stand for Strain Gage 1,..., Strain Gage 4 correspondingly (see Fig. V.2). Note that for all tested specimens, SG-1 was located on the opposite face of the specimen from SG-3, and SG-2 was located on the opposite face from SG-4. The Young’s moduli and Poisson’s ratios measured in compression are summarized in Table V.2 and Table V.3 correspondingly.

In all stress-strain plots, the non-linear behavior in the beginning of the tests can be explained by the compliance of the experimental setup itself. The more evident non-linear behavior observed for SG-2 and SG-4 for stresses below 8 MPa in the “specimen 11” (Fig. V.4) is consistent with the presence of a crack in the specimen. If the hypothesized crack is indeed there, in the beginning of the test the response is characterized by crack closure rather than the bulk material compression; once the crack is fully closed, the compression of the bulk materials starts. Note that no microscopic studies of the specimen have been conducted yet to confirm the presence of the crack. The non-linear behavior of the “specimen 11” is observed in tension as well (see Fig. V.11).
Fig. V.4. Stress-strain curves obtained from the compression of the "specimen 1" using 4 strain gages (SG-1 ... SG-4) bonded on the lateral surfaces of the specimen. Slopes of the curves represent Young's modulus $E_1$.

Fig. V.5. Stress-strain curves obtained from the compression of the "specimen 2" using 4 strain gages (SG-1 ... SG-4) bonded on the lateral surfaces of the specimen. Slopes of the curves represent Young's modulus $E_2$. 

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Fig. V.6. Major-minor strain curves obtained from the compression of the "specimen 22" using 2 strain gage rosettes bonded on the opposite faces of the specimen. Slopes of the curves represent Poisson's ratio \( \nu_{21} \).

Fig. V.7. Stress-strain curves obtained from the compression of the "specimen 33" using 4 strain gages (SG-1 ... SG-4) bonded on the lateral surfaces of the specimen. Slopes of the curves represent Young's modulus \( E_3 \).
Fig. V.8. Major-minor strain curves obtained from the compression of the "specimen 33" using 2 strain gage rosettes bonded on the opposite faces of the specimen. Slopes of the curves represent Poisson's ratio $\nu_{11}$.

Fig. V.9. Major-minor strain curves obtained from the compression of the "specimen 33" using 2 strain gage rosettes bonded on the opposite faces of the specimen. Slopes of the curves represent Poisson's ratio $\nu_{22}$. 
Table V.2. Results of the compression testing: Young’s moduli

<table>
<thead>
<tr>
<th>Strain gage #</th>
<th>Young’s modulus $E_1$, GPa</th>
<th>Strain gage #</th>
<th>Young’s modulus $E_2$, GPa</th>
<th>Strain gage #</th>
<th>Young’s modulus $E_3$, GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>17.57</td>
<td>1</td>
<td>19.63</td>
<td>1</td>
<td>5.53</td>
</tr>
<tr>
<td>2</td>
<td>19.32</td>
<td>2</td>
<td>17.76</td>
<td>2</td>
<td>5.78</td>
</tr>
<tr>
<td>3</td>
<td>18.37</td>
<td>3</td>
<td>16.96</td>
<td>3</td>
<td>5.72</td>
</tr>
<tr>
<td>4</td>
<td>17.57</td>
<td>4</td>
<td>20.27</td>
<td>4</td>
<td>5.94</td>
</tr>
<tr>
<td>Average</td>
<td>18.21</td>
<td>Average</td>
<td>18.66</td>
<td>Average</td>
<td>5.74</td>
</tr>
</tbody>
</table>

Table V.3. Results of the compression testing: Poisson’s ratios

<table>
<thead>
<tr>
<th>Strain gage rosette #</th>
<th>Poisson’s ratio $v_{11}$</th>
<th>Strain gage rosette #</th>
<th>Poisson’s ratio $v_{12}$</th>
<th>Strain gage rosette #</th>
<th>Poisson’s ratio $v_{21}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.1000</td>
<td>1</td>
<td>0.1230</td>
<td>1</td>
<td>0.2985</td>
</tr>
<tr>
<td>2</td>
<td>0.1220</td>
<td>2</td>
<td>0.1063</td>
<td>2</td>
<td>0.3103</td>
</tr>
<tr>
<td>Average</td>
<td>0.1110</td>
<td>Average</td>
<td>0.1147</td>
<td>Average</td>
<td>0.3044</td>
</tr>
</tbody>
</table>

V.3. Uniaxial tension testing of C/C composite felt specimens

V.3.1. Tension test setup

Tension testing was performed on the specimens previously used for compression testing to measure Young’s moduli and evaluate (if any) tension-compression asymmetry of felt layers in C/C composites. Instead of compression platens, cylindrical rods were screwed on to the fixtures. The tension testing setup is shown in Fig. V.10. Specimens were glued to the rods using M-BOND 200 adhesive as follows: thin film of adhesive was applied to the top rod, then preload of 1kN in compression was applied for 2-5 minutes; the same procedure was repeated for the bottom rod. Displacement control was used with strain rate of $3 \cdot 10^{-3} / s$. Data acquisition and post-processing is described in section V.2.
V.3.2. Results of tension testing

The resulting stress-strain plots of the tension testing are shown in Fig. V.11-Fig. V.13. The measured Young’s moduli are summarized Table V.4.

![Stress-strain curves](image)

**Fig. V.11.** Stress-strain curves obtained from the tension of the "specimen 11" using 4 strain gages (SG-1 ... SG-4) bonded on the lateral surfaces of the specimen. Slopes of the curves represent Young’s modulus $E_1$.
Fig. V.12. Stress-strain curves obtained from the tension of the "specimen 22" using 4 strain gages (SG-1 ... SG-4) bonded on the lateral surfaces of the specimen. Slopes of the curves represent Young's modulus $E_2$.

Fig. V.13. Stress-strain curves obtained from the tension of the "specimen 33" using 4 strain gages (SG-1 ... SG-4) bonded on the lateral surfaces of the specimen. Slopes of the curves represent Young's modulus $E_3$. 

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Table V.4. Results of the tension testing: Young’s moduli

<table>
<thead>
<tr>
<th>Strain gage</th>
<th>Young’s modulus $E_1$, GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>16.80</td>
</tr>
<tr>
<td>2</td>
<td>18.80</td>
</tr>
<tr>
<td>3</td>
<td>21.23</td>
</tr>
<tr>
<td>4</td>
<td>19.69</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Strain gage</th>
<th>Young’s modulus $E_2$, GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20.50</td>
</tr>
<tr>
<td>2</td>
<td>20.63</td>
</tr>
<tr>
<td>3</td>
<td>17.02</td>
</tr>
<tr>
<td>4</td>
<td>16.25</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Average (tension)</th>
<th>19.13</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average (compr.)</td>
<td>18.21</td>
</tr>
<tr>
<td>Difference</td>
<td>5.05%</td>
</tr>
</tbody>
</table>

V.4. Discussion and conclusions of experimental measurements on the macroscale

Tension and compression tests were performed on the C/C composite felt specimens. Although significant variations in Young’s moduli (up to 10%) measured from four strain gages of the same specimen were observed, the average values of the moduli were consistent throughout repeated measurements. The following possible sources of the variations are suggested: a) inhomogeneity of the specimen; b) bending of the specimen due to misalignment of the compression platens/tension rods; and c) misalignment of the strain gages. Based on the results (Table V.4) it is reasonable to conclude that there is no significant tension/compression asymmetry exhibited by the C/C composite felt specimens. The material also appears to be transversely isotropic ($E_1 \approx E_2$).

Because only one specimen was tested to measure properties in each direction, the obtained values of Young’s moduli and Poisson’s ratios cannot be considered representative of the C/C composite felt specimens. However these values provide initial estimates of the material’s mechanical properties and allow to make preliminary conclusions about its tension/compression asymmetry.
VI. CONCLUSIONS

Carbon/carbon composites manufactured by chemical vapor infiltration possess a complicated hierarchical microstructure as seen in Fig. 1.2. Characterization and modeling approaches on multiple length scales (micro, meso and macro) have been presented in the preceding Chapters and are summarized below.

On the microscale, a new set of thermo-elasticity solutions for cylindrically orthotropic multicoated cylinders representing the basic material unit in the C/C composites (fiber surrounded by layers of PyC) is presented, see Tsukrov and Drach (2010). Explicit expressions for displacement and stress components are given for five loading cases: transverse hydrostatic tension, axial elongation, unconstrained thermal expansion, longitudinal and transverse shear. The new elasticity solutions are utilized to predict the following effective thermo-mechanical properties of the basic material unit of the C/C composite (Tsukrov et al. (2012)): Young’s and shear moduli in the longitudinal and transverse directions, Poisson’s ratio in the longitudinal direction and coefficients of thermal expansion in longitudinal and transverse directions. The homogenization approach is based on the Hashin’s composite cylinder assemblage model (Hashin (1983)) and self-consistent scheme (Christensen and Lo (1979)).

On the mesoscale, an automated numerical approach to evaluate contributions of irregularly shaped pores to the effective material properties of C/C composites is presented, see Drach et al. (2011). The procedure combines finite element analysis and the pore compliance contribution tensor approach (Kachanov et al. (1994)). The pore shapes utilized in the analysis were extracted from the X-ray computed microtomography data kindly provided by the Institute of Materials Science and Engineering I at Karlsruhe Institute of Technology (Germany). The method is demonstrated for isotropic and transversely isotropic matrices. In both cases the

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homogenized properties of PyC matrix are found based on the nanoscale modeling predictions given in Böhlke et al. (2010).

A statistical modeling approach (Drach et al. (2013)) is proposed as an alternative to the direct calculations of pore compliance contribution tensors of a large number of pores on mesoscale. The method is based on the development of a statistical model that correlates pore geometric parameters with pore compliance contribution parameters. The model is developed using design of experiments approach and statistical analysis of pore volume and orientation distributions performed on the X-ray computed microtomography data from a laminated C/C composite sample. It is found that a reasonably good correlation can be established between principal moments of inertia of the pores and their contributions to the effective bulk and shear moduli ($R^2 = 0.99$ and $R^2 = 0.90$ correspondingly). It is also observed that by adding the non-dimensional surface-to-volume ratio $S_y = S^{1/2}/V^{1/3}$ the accuracy of statistical model predictions can be significantly improved, e.g. $R^2 = 0.56$ vs $R^2 = 0.87$ in the case of the pore contributions to the effective Young’s modulus $E_1$.

On the macroscale, a set of tension/compression tests using strain gages was performed and the results are presented in Chapter V. The test specimens were prepared from a C/C composite sample provided by the Institute of Materials Science at Karlsruhe Institute of Technology (Germany). It is observed that the tested specimens exhibit transverse isotropy ($E_1 \approx E_2$) and some degree of preferential orientation of the carbon fiber reinforcement ($E_1 \approx E_2 > E_3$). No tension/compression asymmetry is observed. The details of the experiments are presented in Table V.2, Table V.3 and Table V.4. Because only one specimen was tested to measure properties in each direction, the obtained values of Young’s moduli and Poisson’s ratios cannot be considered representative of C/C composites.
REFERENCES


