Concretization of nonlinear constitutive relations by results of uniaxial compression and indentation experiments

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Abstract. A method for the experimental specification of the variant of the Hencky–Murnaghan constitutive relations is proposed. A series of compression and indentation experiments of a number of specimens made of highly elastic materials is carried out on the experimental stand. An iterative procedure for determining constants from experiments on inhomogeneous deformation is developed. The convergence of the iterative process is investigated. The values of the constants of the Hencky–Murnaghan model are found for materials of two types. It is found that the Hencky–Murnaghan model describes the results of indentation experiments on a wider range of deformations than the Hencky model.

1. Introduction

In [1, 2], the constitutive relations of the Hencky–Murnaghan hyperelastic material are proposed. The problem of experimental determination of the elastic constants included in the model is urgent. Most modern types of rubbers can be attributed to the class of incompressible materials, for which the specification of the constitutive relations of the Hencky–Murnaghan model is reduced to the determination of two elastic constants: the initial shear modulus $G$ and a constant $c_3$. The values of these constants for some elastomers in this work are determined from experiments on uniaxial compression using an analytical solution for the case of uniform deformation. However, in real experiments, inhomogeneity of the stress-strain state during compression is observed, due to friction between the sample and the supporting surfaces. The fundamental possibility of determining elastic constants $G$ and $c_3$ from experiments assuming inhomogeneous deformation of samples is demonstrated by the example of processing experiments on indentation, for which the effect of contact friction on the integral characteristic of the process is minimal [3]. The parameters of the model are determined by comparing the experimental and the integral characteristics of the process calculated using a numerical model. As a rule, such characteristics in mechanical experiments are the dependences of the force on the displacement.

If the values recorded in the experiment can be explicitly related by the analytical dependence, the model parameters can be determined using the least squares method. If it is difficult to obtain an analytical solution, but there is a numerical model of the experiment, the inverse problem can be formulated as follows: it is necessary to find the parameters of the model that
provide a minimum to the mean square deviation of the calculated values from the experimental ones. In this case, optimization methods and a variety of iterative procedures are widely used to determine the parameter values, including the methods of Hooke–Jeeves [4], Nelder–Mead [5], Levenberg–McGraft [6–9]. In [10, 11], a procedure is proposed for determining model parameters from experiments on inhomogeneous deformation using the direct free gradient method.

2. Processing of experiments on uniaxial compression of highly elastic materials

To carry out compression and indentation experiments, a kinematic stand shown in the figure 1 was designed. Description and main characteristics of the stand are presented in [12].

A series of experiments on uniaxial compression of cylindrical specimens of two types of soft materials was carried out at the kinematic loading stand. Samples of the first type were cylinders with \( r_0 = 19 \) mm, \( h_0 = 28 \) mm, made of a silicone compound Silagerm 2111 grade (hereinafter "material 1"). Samples of the second type were cylinders with \( r_0 = 13.3 \) mm, \( h_0 = 14 \) mm, made of a material that is a mixture of polyvinyl chloride with phthalate plasticizers (hereinafter "material 2"). The condition of quasi-stationarity of the process was achieved in experiments by using the multistep loading method, in which for each step the deformations increased by 5% at a rate 5 mm/min, and after each step a pause of 600 s was expected for stress relaxation [13]. To reduce the effect of friction in compression experiments, a polyamide film, coated with mineral oil, was placed between the test specimen and the reference planes.

Preliminary tests were carried out for samples made of material 1. The purpose of these tests was to establish the range of deformations in which the material could be considered as elastic and incompressible. Preliminary experiments on compression of material 1 included a full cycle.
Preliminary experiments on compression of the material 1 show that the material 1 exhibits pronounced elastic properties in the medium and large range of compression deformations. At the magnitude of axial strains $| \varepsilon_z | < 0.15$, the sample visually retains its cylindrical shape, the coefficient of transverse deformations is close to 0.5, that can be considered a valid proof of the weak compressibility of the material 1 in this range of strains. At further loading, the effect of geometric distortion, inhomogeneity of the stress-strain state, caused by friction between the sample and the base plates, appears. In this case, the sample loses its cylindrical shape. Therefore, measurements of radial strains only in the middle section do not allow us to conclude that the sample volume is preserved or changed. Similar results have been repeatedly discussed in [14–16], devoted to experiments on uniaxial compression of rubbers. It is recommended to determine the initial bulk modulus from a pure bulk compression experiment [17]. The above results allow us to further consider the materials 1 and 2 as elastic incompressible and use the Hencky–Murnaghan constitutive relations for an incompressible material:

$$\Sigma_R = -pE + 2G\tilde{\Gamma} + c_3Q,$$

(1)

where $\Sigma_R = \frac{dV}{dV_0} R \cdot S \cdot R^{-1}$ is the generalized “rotated” stress tensor [18]; $\tilde{\Gamma}$ is the deviator of the logarithmic strain measure; $Q = \tilde{\Gamma}^2 - \frac{1}{3} e^2 E$ is the deviator of the tensor $\tilde{\Gamma}^2$; $p$ is the hydrostatic pressure; $e$ is the strain intensity; $G$ and $c_3$ are material constants; $E$ is the identity tensor.

The dependence of true stress $S_{zz}$ on the multiplicity of elongation $\lambda$ under uniaxial tension-compression of a material sample with constitutive relations (1) under the assumption of

**Figure 2.** Axial force $P^e$ versus displacement $D$ in uniaxial compression test for material 1.

**Figure 3.** Different stages of uniaxial compression of a sample of material 1.
uniformity of the stress-strain state has the form:

\[ S_{zz}(\lambda) = 3G \ln \lambda + \frac{3c_3}{4} \ln^2 \lambda. \]  

(2)

Note that relations (1) have the form of relations for the Hencky material model, which is a well-known generalization of Hooke’s law to the case of finite strains.

For each of the samples of materials 1 and 2, 3 compression cycles were carried out in the direction of the axis of symmetry on a stand using a round plate. In each experiment, the values of the axial displacement of the base plate \( D \) [mm] and the force \( P_e \) [N] corresponding to this displacement were recorded. The processing of the array of experimental data obtained from the \( m = 6 \) experiments was reduced to calculating the average values of the forces \( \langle P_e \rangle_i = \frac{1}{m} \sum_{j=1}^{m} P_{e,i,j} \) corresponding to the value of the displacement \( D_i \). For the average values of displacements, the multiplicities of axial elongation \( \lambda_i = 1 + \frac{D_i}{h} \) were calculated. The cross-sectional area in the current state \( A_i \) was calculated from the condition of incompressibility of the test materials \( \lambda_1 \lambda_2 \lambda_3 = 1: \)

\[ A_i = \frac{1}{\lambda_i} \pi r^2. \]

The results of compression experiments with three samples of each of the materials are presented in the figures 4 and 5 in the form of dependences of the average axial true stresses \( \langle S_{zz} \rangle_i \), shown by blue circles, on the elongation ratio \( \lambda_i \). The orange crosses in the figure 5 show the values of the absolute deviations \( \langle S_{zz} \rangle_i \pm \Delta S_{zz} \).

Let us determine the values of the constants \( G \) and \( c_3 \) proceeding from the requirement that the analytical dependence (2) approximates the array of experimental points \( \langle \lambda_i, \langle S_{zz} \rangle_i \rangle \) in the best way. We use the least squares method, which consists in finding the values of the model parameters \( \tilde{G} \) and \( \tilde{c}_3 \), delivering a minimum to the value of the standard deviation:

\[ f(G, c_3) = \sum_{i=1}^{n} (\langle S_{zz} \rangle_i - S_{zz}(\lambda_i, G, c_3))^2 \rightarrow \min. \]  

(3)

Using the necessary conditions for the extremum of the function of two variables (3), we obtain a system of equations for determining the parameters \( \tilde{G} \) and \( \tilde{c}_3 \), solving which we obtain the values of the elastic constants given in the table.

Analytical dependences (2), constructed for materials 1 and 2, taking into account the values of the constants from the table, are shown in the figures 4 and 5 with red solid lines. The black solid lines show quasilinear solutions (2), in which \( c_3 = 0 \).

The assumption about the uniformity of the stress-strain state is the main one in processing the compression experiment. This assumption is in good agreement with the experimental data.
Figure 5. Average axial true stresses versus elongation ratio during the compression of a sample of material 2.

Table 1. Experimental data processing results.

| Material | Uniaxial compression test, $G, MPa$ | Indentation test, $R = 3$ mm, $G, MPa$, $c_3, MPa$ | Indentation test, $R = 7.5$ mm, $G, MPa$, $c_3, MPa$ |
|----------|-----------------------------------|-----------------------------------------------|-----------------------------------------------|
| 1        | 0.25 -2.03 0.23 -2.4 0.24 -2.82   |                                               |                                               |
| 2        | 0.51 -2.69 0.49 -3.34 – –         |                                               |                                               |

only at the initial stages of loading. Using the developed program for numerical simulation, finite element calculations [2] of loading of nonlinear elastic material 2 with characteristics from the table were performed. It can be concluded that friction has a lesser effect on the stress-strain state of specimens with a small value of the ratio $r_0/h_0$ than for specimens with a large value $r_0/h_0$. This conclusion fully confirms the conclusions made in the work [19].

The influence of the value of the friction coefficient $\mu$ at the initial loading section is small. Therefore, we can say that the constant $G$ that determines the initial slope of the loading curve is found quite accurately from the compression experiment. The correct determination of the constant $c_3$ from the uniaxial compression experiment is possible in the following cases:

- if we use a sample of material with the lowest possible ratio $r_0/h_0$. It should be noted, however, that experiments on uniaxial compression with samples with sufficiently small values $r_0/h_0$ are unfeasible in the region of large compression ratios $\lambda < 0.8$ due to the loss of stability by the sample;
- if we take into account the inhomogeneity of the stress-strain state, due to the influence of contact friction, in the analytical solution. Variants of analytical solutions of the problem of compression for linear elastic bodies, which take into account the curvature of the lateral surface of cylindrical samples are known [16, 20]. However, it is not possible to obtain a solution that takes into account the inhomogeneities arising from the interaction of the bases of a cylindrical specimen with the support planes for the problem of compression with constitutive relations in the form (1) in an analytical form;
- if we use the constructed numerical model of the finite nonlinear elastic deformation of cylindrical bodies. In this case, the inverse problem of determining the parameters of the model can be solved taking into account the inhomogeneity of the stress-strain state.
3. An iterative procedure for determining elastic constants from experiments on inhomogeneous deformation

As has been repeatedly noted, for the problem of inhomogeneous finite elastic deformation, it is difficult to obtain the dependence of the sample response under loading in an explicit form containing the model parameters. The numerical solution of the problem allows one to obtain all the characteristics of the stress-strain state, taking into account the given initial and boundary conditions for specific values of the model parameters. It is convenient to use as calculated values the integral power characteristics of the process, for which direct measurements are feasible. In this case, the identification of the Hencky–Murnaghan material (1) is reduced to finding the values of the model parameters \(\{G, c_3\}\) that provide a minimum of the total standard deviation of the calculated values of the forces \(P_i^s\) from the average values \(\langle P^e_i\rangle\) obtained from the experiment:

\[
\Delta = \sum_{i=1}^{n} (P_i^s - \langle P^e_i\rangle)^2 \rightarrow \min. \tag{4}
\]

The necessary conditions for the minimum of the sum (4) take the following form:

\[
\frac{\partial \Delta}{\partial G} = 2 \sum_{i=1}^{n} (P_i^s - \langle P^e_i\rangle) \frac{\partial P_i^s}{\partial G} = 0, \tag{5}
\]

\[
\frac{\partial \Delta}{\partial c_3} = 2 \sum_{i=1}^{n} (P_i^s - \langle P^e_i\rangle) \frac{\partial P_i^s}{\partial c_3} = 0.
\]

Using the resolving equations of the finite element method [2] and the fact that the constants \(G\) and \(c_3\) are linearly included in the constitutive relations (1), and also assuming that in the case of setting the boundary conditions of the kinematic type, the quantities \(\frac{\partial P^e_i}{\partial G}\) and \(\frac{\partial P^e_i}{\partial c_3}\) do not depend on the derivatives of the velocity field \([V_k]\) with respect to the model parameters \(G\) and \(c_3\), we obtain the minimum condition (4) in the form of a quasilinear system of equations with respect to unknown constants \(G\) and \(c_3\):

\[
\sum_{i=1}^{n} \left( \int_0^{t_i} \int_{\Sigma_e} \left( [C^r_{mk}] + [C^G_{mk}] G + [C^{c_3}_{mk}] c_3 \right) [V_k]d\Sigma_c d\tau - \langle P^e_i\rangle \right) \int_0^{t_i} \int_{\Sigma_e} [C^G_{mk}] [V_k]d\Sigma_c d\tau = 0, \tag{6}
\]

\[
\sum_{i=1}^{n} \left( \int_0^{t_i} \int_{\Sigma_e} \left( [C^r_{mk}] + [C^G_{mk}] G + [C^{c_3}_{mk}] c_3 \right) [V_k]d\Sigma_c d\tau - \langle P^e_i\rangle \right) \int_0^{t_i} \int_{\Sigma_e} [C^{c_3}_{mk}] [V_k]d\Sigma_c d\tau = 0, \tag{7}
\]

where \([C^r_{mk}], [C^G_{mk}], [C^{c_3}_{mk}]\) are the terms of the global stiffness matrix corresponding to the parameters \(G\) and \(c_3\).

The system (6), (7) can be solved using the following iterative procedure:

1) an initial approximation \(\{G^0, c_3^0\}\) is chosen;
2) from the numerical solution of the corresponding initial-boundary value problem \([C^r_{mk}], [C^G_{mk}], [C^{c_3}_{mk}]\) and \([V_k]\) are determined for the values \(\{G^0, c_3^0\}\);
3) the found values \([C^r_{mk}], [C^G_{mk}], [C^{c_3}_{mk}]\) and \([V_k]\) are substituted into the system (6);
Figure 6. Convergence of values of elastic constants $G$, $c_3$ at the initial approximation $G^0 = 0.1\, MPa$, $c_3^0 = 0$.

4) from the solution of the resulting system of linear algebraic equations, $G^1$ and $c_3^1$ are determined, which are used as an initial approximation for the next step.

The criterion for the convergence of the iterative procedure is the fulfillment of the inequality $|\Delta_k - \Delta_{k-1}| < \xi$, where $\xi$ is the required value of the relative error, $k$ is the iteration number.

The convergence of the computational procedure constructed to determine the elastic constants was investigated using the problem of determining the elastic constants from a compression experiment as an example. Data sets obtained from uniaxial compression experiments for material 2 were used.

The figures 6–8 show the dependences of the parameter values $G^k$, $c_3^k$ and $\Delta^k$ on the iteration number $k = 1 \ldots 12$.

Comparison of the values $G^{12}$ and $c_3^{12}$ obtained numerically for 12 iterations with the values $G$ and $c_3$ obtained using the analytical solution for an incompressible material, shows that for the material 2 the maximum relative difference is 3.3% for the constant $G$ and 2.5% for the constant $c_3$. This allows us to conclude that the constructed iterative procedure is applicable for the numerical solution of the inverse problem of determining the elastic constants of a weakly compressible material.

4. Processing of experiments on indentation of highly elastic materials

As shown in [3], friction has an insignificant effect on the integral force characteristic of the indentation process of hyperelastic materials. In addition, during indentation, it is possible to obtain large deformations localized in the hearth under the indenter, with lower values of the forces than the same deformations during compression.

Experiments on the indentation of slabs made of the materials 1 and 2 were carried out at the stand. To reduce the influence of free boundaries, samples were made in the form of a cylinder with a radius $r_0 = 40\, mm$ and height $h_0 = 10\, mm$. For a sample made of the material 1, 3 indentation cycles were carried out in the direction of the symmetry axis on the stand using indenters with radii $R = 3\, mm$ and $R = 7.5\, mm$. For a sample made of the material 2, 3 indentation cycles were carried out in the direction of the axis of symmetry on the stand using only the indenter with radius $R = 3\, mm$, since for the indenter with radius $R = 7.5\, mm$ an excess of the permissible force for the stand was recorded.
In each experiment, the absolute values of the indenter displacement $D\ [mm]$ and the axial forces $P^e\ [N]$ corresponding to this displacement were recorded. The processing of the experimental data array obtained during the $m = 3$ experiments for each material sample was reduced to calculating the average values of the forces $\langle P^e \rangle_i = \frac{1}{m} \sum_{j=1}^{m} P^e_{i,j}$ corresponding to the displacement value $D_i$. For the experimental data arrays, a procedure for finding the zero point was required. The zero point corresponds to the displacement, at which the beginning of the growth of the axial force value is recorded. For the average displacement values, the values of the relative settlement, equal to the ratio of the displacement of the indenter to its radius $D_i/R$ were calculated.

The results of indentation experiments with samples of the materials 1 and 2 are presented in the figures 9, 10 in the form of dependences of the forces $P^e[N]$ shown by blue circles on the
Figure 9. Axial force $P_e$ versus relative precipitation $D/R$ during the indentation of a sample of the material 1.

Figure 10. Axial force $P_e$ versus relative precipitation $D/R$ during the indentation of a sample of the material 2.

value of the relative settlement $D_i/R$. The orange crosses in the figures 9, 10 show the values of the absolute deviations $P_e^i \pm \Delta P_e^i$.

Using the constructed iterative procedure for solving system (6), the values of constants $G$ and $c_3$ for the materials 1 and 2 are determined from indentation experiments. The convergence of the iterative procedure is demonstrated by the example of processing an experiment on the indentation of a sample of the material 1 with a sphere of radius $R = 3\, mm$ (see figure 11).

An analysis of the results of determining the elastic constants of the material 1 shows that the differences in the values obtained from experiments with different indenter radii are less than 5% for the constant $G$ and less than 15% for the constant $c_3$. Comparison of the values of the model constants for the material 1 determined in the indentation experiment and in the compression experiment shows that the difference in the determination of the constant $G$ is 8%, and for the constant $c_3$ it reaches 28%. For the material 2, the difference in the determination of the constant $G$ is 4%, and for the constant $c_3$ it is less than 20%. Thus, taking into account the inhomogeneity of the deformation during indentation makes it possible to refine the values of the model constants. This especially concerns the constant $c_3$, which is responsible for taking into account the deviation of material properties from quasilinear ones.

The dependences of axial forces $P_e$ on the value of the relative settlement $D/R$, constructed as a result of numerical modeling of the processes of indentation of materials, are shown in the figures 12–14. In the figures 12–14, quasilinear numerical solutions obtained when calculating according to relations (1) with $c_3 = 0$ are also shown.
Figure 11. The calculated values of the axial force $P_c$ in the iterative procedure for determining the elastic constants of the material 1.

Figure 12. Axial force versus $P_c$ relative precipitation $D/R$ during the indentation of the material 1 by the sphere with radius $R = 3\, mm$.

Figure 13. Axial force versus $P_c$ relative precipitation $D/R$ during the indentation of the material 1 by the sphere with radius $R = 7.5\, mm$. 
Figure 14. Axial force versus $P^e$ relative precipitation $D/R$ during the indentation of the material 2 by the sphere with radius $R = 3\ mm$.

At the values of the relative settlement of the rigid sphere $D/R > 0.5$, the axial forces calculated using the linear model of the Hencky material are underestimated in comparison with the experimental data by 30–50% for the material 1. At the same level of relative displacement for samples of the material 2, an underestimation of the values of axial forces by 25% is observed in comparison with the experimental data when using a linear model.

5. Conclusion
Based on the approximation of the experimental points by the dependence of the true stresses on the elongations under uniform compression, which follows from the constitutive relations (1), the values of the constants $G$ and $c_3$ were obtained. The use of Hencky quasilinear model leads to significant deviations of the theoretical dependence from the experimental one at $\lambda < 0.9$. The nonlinear two-constant Hencky–Murnaghan model approximates the experimental data well over the entire measurement range (up to $\lambda \approx 0.7$).

Despite the difficulties in processing indentation experiments, which involve the need to solve the initial-boundary value problem, the indentation experiment is one of the least demanding on the size and shape of the samples under study in comparison with classical experiments. Using the example of highly elastic materials, the fundamental possibility of determining the constants of the material model from the indentation experiment is shown. It is shown that the numerical Hencky–Murnaghan model with constitutive relations (1) describes the processes of indentation in a wider range of deformations than the physically linear Hencky model.

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References
[1] Markin A and Sokolova M Yu 2019 Mechanics of Solids 54(8) 1182–1188.
[2] Markin A, Sokolova M Yu, Khristich D V and Astapov Yu V 2019 International Journal of Applied Mechanics 11(7) 1950064-1–1950064-13
[3] Astapov Yu V and Khristich D V 2018 International Journal of Applied Mechanics 10(3) 1850026-1–1850026-12
[4] Hooke R and Jeeves T A 1961 Journal of the ACM 8(2) 212–229
[5] Nelder J A and Mead R 1965 The Computer Journal 7(4) 308–313
[6] Marquardt D. 1963 SIAM Journal on Applied Mathematics 11(2) 431–441
[7] Wu Y, Wang H and Li A 2016 Applied Sciences 6(386) 1–13
[8] Mesa-Munera E, Ramirez-Salazar J-F, Boulanger P and Branch J W 2012 Ingeniería y Ciencia 8 11–36
[9] Madireddy S, Sista B and Vemaganti K 2016 Journal of the Mechanical Behavior of Biomedical Materials 59 108–127
[10] Hartmann S and Neff P 2003 International journal of solids and structures 40 2767–2791
[11] Seibert D J and Schöche N 2000 Rubber Chemistry and Technology 73(2) 366–384
[12] Astapov Yu V and Khristich D V 2019 IOP Conf. Series: Journal of Physics: Conf. Series 1203 012015-1–012105-10
[13] Neff P, Ghiba I-D and Lankeit J 2015 Journal of Elasticity 121 143–234
[14] Forster M J 1955 Journal of Applied Physics 26(9) 1104–1106
[15] Anderson M L, Mott P H and Roland C M 2004 Rubber Chemistry and Technology 77(2) 293–302
[16] Williams J G and Gamonpilas C 2008 International Journal of Solids and Structures 45 4448–4459
[17] Gehrmann O, Kröger N H, Erren P and Juhre D 2017 Technische mechanic 37(1) 28–36
[18] Markin A A and Sokolova M Yu 2015 Thermomechanics of Elastoplastic Deformation (Cambridge: Cambridge International Science Publishing)
[19] Kim S, Kim M, Shin H and Rhee K-Y 2018 Experimental Mechanics 58 1479–1484
[20] Gent A N 1969 Rubber Chemistry and Technology 69(1) 59–61