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Modeling of indentation processes

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1 INTRODUCTION

Knowledge of the mechanical properties of materials is important for various fields of human activities, such as engineering, transport or construction. In various applications, e.g. in vehicles, they are exposed to a variety of load conditions (vibrations caused by driving on uneven roads or engine running, sudden speed changes, collisions with foreign objects). Due to the increasing performance of computer technologies, these load cases can be included in the calculation model and the behaviour of the individual materials in the real conditions can be verified or predicted. The more accurate material information we have, the more accurate results we will get. This will streamline product development and reduce the cost on testing and prototyping.

One way to determine the mechanical properties of materials is the indentation test. The principle of the indentation process is the creation of the impression formed by the indenter into the tested sample. During the indentation, the impression depth is recorded as a function of force and time. The depth of impression can reach nanometer units up to tens of millimeters, so it is possible to examine thin layers of materials, very small samples or, for example, parts of the human organs that could not be characterized by other than indentative method. By means of indentation it is possible to investigate elastic, elastic-plastic and time-dependent force response. Indentation can also be used to measure the properties of heterogeneous materials and to measure fracture toughness in brittle materials. Another great advantage of this process is also that it does not require a number of specially treated samples. Thanks to these characteristics, indentation is a universal and perspective testing method.

Simulation of the indentation process using finite element method extends the possibilities of the test itself. It facilitates understanding of internal mechanisms, which are difficult to analyze only experimentally. The examined volume is divided into a finite number of elements. During the loading and the unloading it is possible to examine the behaviour of elements separately. It is possible to analyze stress and strain. If special elements or extended finite element method (XFEM) are used, crack propagation can be simulated.

The aim of this thesis is to create models by means of which it is possible to simulate indentation into the hard and brittle materials, such as glass, laminated glass or various types of ceramics. The created models are very useful as a support for the indentation tests, because by this way we obtain other material characteristics that are difficult or even impossible to obtain experimentally. Due to this models, it is also possible to detect eventual measurement inaccuracies. Another benefit is the ability to investigate the response of an unknown material strucure to assess the suitability of the application for a given purpose without the need to manufacture the sample in advance.

1.1 Current state of the problem

While current measurement methods allow the investigation of failure mechanisms in brittle materials as well as in laminates, either the method is very complex or only valid for a limited group of materials. For this reason, it is useful to create a model and simulate the process.

Analytical methods allow studying only of very simplified models [3], [12], [6]. The problem of indentation by the Vickers indenter into the elastic plastic material, including in addition the crack propagation process, can be modeled only by the numerical methods based on FEM [1], [2], [11], [13]. In [4] from 2014, the authors deal with FEM modeling of ceramic fractures. The aim of the research was to find the correction of relations for fracture toughness calculation using different indenter shapes. For this purpose, a model containing a cohesive zone in which crack propagation occurs was created. There are several studies based on the FEM model with a cohesive zone, for example [5], [8]. These models show that this approach well describes the real indentation process.

2 DISSERTATION AIM

Aim of this thesis can be divided into the following points:

- Creating a FEM model that simulates the indentation of Vickers indenter into the hard and brittle materials.
- Characterization of the mechanical properties of glass and laminated glass using indentation method.
- Model verification. Comparison of measured and calculated characteristics.

3 MODEL DESCRIPTION

3.1 Axisymetrical model

3.1.1 Geometry

The axially symmetrical model consists of an indenter and an axially symmetrical specimen. The specimen consists of either one layer or two layers so that it can also describe laminated structure. The indenter is conical at an apex angle of 140.6°, which at the given indentation depth gives the same projected area as the real Vickers indenter. The dimensions of the specimen are $R = 2350\ \mu m$ and the height $H = 2350\ \mu m$. The dimensions of the laminated strucure are $H_1 = 2000\ \mu m$, $H_2 = 350\ \mu m$ and $R = 2350\ \mu m$.

3.1.2 Mesh description

The indenter is modeled using a perfectly rigid analytical surface. The specimen is modeled by four-node axially symmetrical elements. The mesh is refined around the contact of the indenter with the specimen. Edge length of the smallest element $l = 0.35\ \mu m$.

Part	Nodes	Elements	Element type
Specimen	4	12886	QUAD (CAX4)
Indenter	-	1	Analytical rigid surface

Table 1: Elements

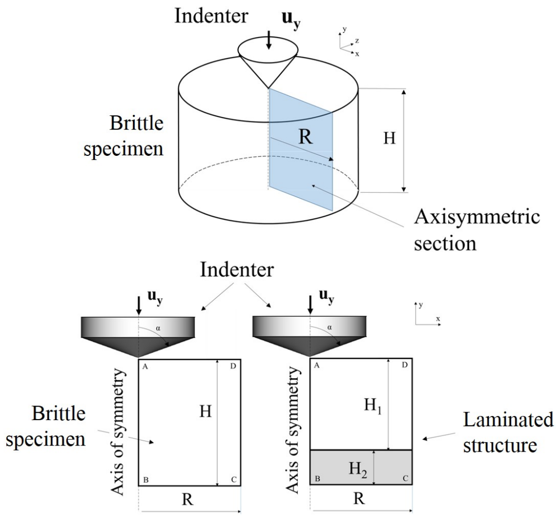

Figure 1: Axial symmetric model

3.1.3 Contact definition

A contact is prescribed between the indentor and the contact surface. Discretization of contact surfaces is surface to surface type. The mutual movement of the contact surfaces is solved by the finite sliding method. Contact stiffness is initially controlled by a nonlinear penalty method. Coefficient of shear friction between contact surfaces $f = 0.1$.

3.1.4 Boundary conditions

Nodes at the bottom of the specimen (BC boundary in fig.1) are prevented from moving in the normal direction to the bottom surface. Nodes in the symmetry axis (boundary AB in fig.1) are prevented

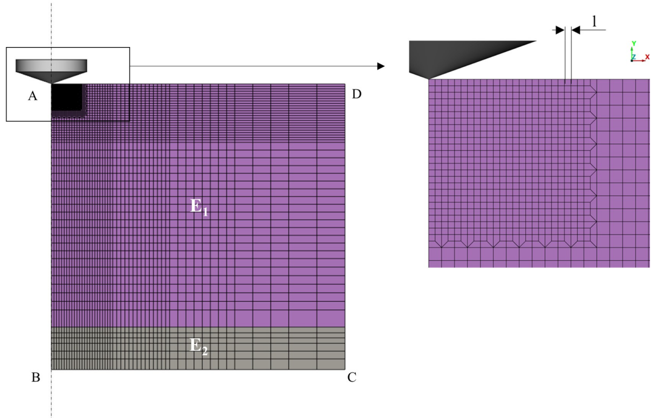

Figure 2: FEM mesh

from moving in the normal direction to the symmetry axis. The analytical surface is connected by a perfectly rigid RB binding to the control node. This node prescribes a forced indenter displacement in the y direction.

3.1.5 Definition of material properties

Glass is a brittle material. At normal temperatures, the extent of plastic deformation of the glass is negligible, but with concentrated contact, high shear stresses are produced, which are induced by pressure. These stresses allow atoms to pass to adjacent positions. Ions and smaller structural units are densified to adjacent sites. According to the theory developed by Marsh [9], plastic flow begins beyond the yield point. The boundary of plastic flow follows the material model Von Mises. Elastic behavior to yield strength σ_Y is governed by equation (1) and plastic behavior is described by equations (2).

The picture 3 shows the dependence of stress - strain. The material model describing glass is almost linear. Hardening module $E_{pl} = 50$ GPa. Young's module $E = 73.4$ GPa. Yield stress $\sigma_Y = 0.5$ GPa.

$$\sigma = E\epsilon \tag{1}$$

$$\sigma = E_{pl}\epsilon_{pl} + \sigma_Y \tag{2}$$

σ_Y is yield stress, ϵ_{pl} is plastic strain. E is Young modulus.

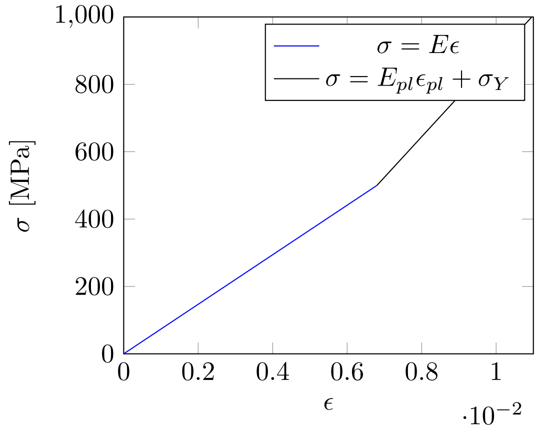

Figure 3: Bilinear material model

3.2 3D model of crack propagation

3.2.1 Geometry

The model geometry shown in fig. 4 contains only 1/4 of the loaded specimen. Model height $H = 2350\ \mu m$ and model radius $R = 2350\ \mu m$. The Vickers indentor model also contains 1/4 of the entire indenter. In order to show where and at what point cracks occur, the Cohesive Zone area was included into the model. This zone has a thickness of 0.02 μm.

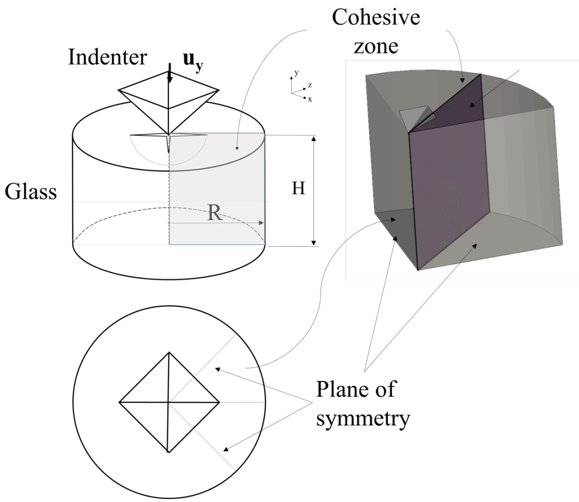

Figure 4: Scheme of 3D model

3.2.2 Mesh description

The mesh is created by rotating the 2D mesh along the y axis. In the radial direction, the mesh is divided into 16 elements. Very small elements will emerge around the concentrated contact. As the distance from the axis of rotation increases, the radial size of the elements increases. The minimum edge length of the smallest element around the contact $a_{min} = 0.3\ \mu m$, maximum edge length of the smallest element $a = 2.99\ \mu m$ (image 5). The indentor is modeled with three and four-node 2D elements with a thickness of $t = 1\mu m$. The glass sample is discretized by 3D linear elements C3D8 and C3D6, where 3D means three dimensional space, numbers 8 and 6 indicate the number of nodes of the element. It can be seen that the 3D model has larger elements than the 2D model. For smaller dimensions we encounter a problem with the time consuming calculation. The main purpose of the 3D model is to analyze the crack propagation process and not to evaluate absolute stress values. We are not interested in the exact stress value at the crack face, but we are satisfied with the stress value in its vicinity. The assumption is that under the surface there are cracks smaller than the size of the smallest elements. A critical stress intensity factor can be calculated from the presumed crack shape, which may be, for example, a penny-shaped crack and the stresses around it, to estimate the magnitude of the initiation stress.

The cohesive zone area consists of the COH3D8 cohesive elements, where the letter H indicates that it is a cohesive element. The material model is the same as in the 2D model.

3.2.3 Definition of material properties

The plastic flow follows the Von Mises material model similar to the 2D model. There is also used a more general model Drucker - Prager.

Part of the model	Number of Nodes	Number of elements	Element type
Glass	8	23008	C3D8
Glass	6	992	C3D6
Cohesive zone	8, 6	1500	COH3D8
Indenter	19643	4	S4
Indenter	345	3	S3R

Table 2: Elements

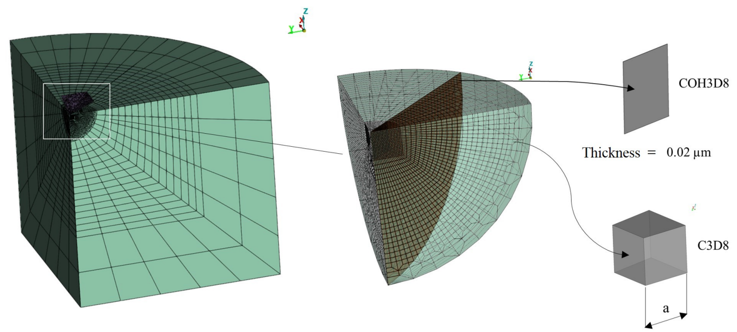

Figure 5: 3D mesh

The Drucker - Prager model is controlled by three parameters. The first parameter is the angle $\beta = 20°$. Another parameter is $K = 0.8$. The last parameter is the dilatation angle Ψ. At $\Psi = 0$ there is no change in volume (incompressible materials) during plastic flow. Materials that have an expansion angle greater than zero dilate. The model is set to $\beta = \Psi$. K, β, and Ψ have been estimated. The plastic behavior in compression is governed by equations (1) and (2).

3.2.4 Contact definition

A contact is defined between the indenter and the contact surface as defined in the 2D model. The contact pair is formed on the faces of the elements bordering the cohesive zone. Discretization of contact pairs is of the node to surface type. The mutual movement of the contact surfaces is controlled by the small sliding method. Zero clearance is forced between the contact surfaces. Friction is not considered in this contact.

3.2.5 Boundary conditions

Nodes in the lower surface of the specimen are prevented from moving in the normal direction to the surface. The same applies to the lateral surfaces of the symmetry planes, see figure 4. The nodes of the indenter elements are bounded by a perfectly rigid constraint to the control node where the boundary condition of the forced displacement is prescribed.

3.2.6 Failure criteria

The criteria of the elimination of cohesive elements are governed by the relation (3) and (4). If the condition (3) is met in the cohesive element, there is a phase of development of the fracture. The element is eliminated at the next load if the area under the curve (stress - displacement) corresponds to fracture energy G_c.

Failure criterion has the form:

$$\left\{\frac{\sigma_n}{\sigma_n^0}\right\}^2 + \left\{\frac{\tau_s}{\tau_s^0}\right\}^2 + \left\{\frac{\tau_t}{\tau_t^0}\right\}^2 = 1 \tag{3}$$

$$G_C = (G_s^C - G_n^C)\left(\frac{G_S}{G_T}\right)^\eta \tag{4}$$

σ_t is the tensile stress. τ_s and τ_t are shear stresses. It should be noted here that the cohesive elements do not carry compressive loads. Therefore, it is not possible to model the failure caused by

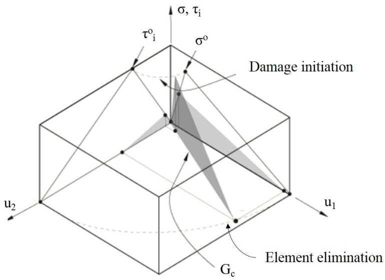

Figure 6: Damage initiation and evolution

pure pressure but also by shear from compressive load. Illustratively, the criteria of crack initiation and evolution are seen in the picture 6. The axis σ, τ shows the amount of tensile and shear stress. The axis u_1 shows the strain caused by the tensile stress. The axis u_2 shows the shear stress-induced deformation. If both the tensile stress component and the shear stress component are applied, then the energy required to eliminate the element is the area under the stress-strain curve (the gray area G_c in 6). The prescription of the crack development criterion is given by the equation (4) [1]. The stress in the model is $\sigma_t^o = \tau_t^o = \tau_s^o = 60$ MPa. The energy G_C is calculated by the relation (4). Where $G_S = G_s^C + G_t^C$, $G_T = G_n^C + G_S$. Tensile Fracture energy is G_n^C. Fracture energies induced by shear are G_s^C and G_t^C. For the crack development energy criterion, It was prescribed $G_n^C = G_s^C = G_t^C = 2.5$ N / m for the Von Mises model and $G_n^C = G_s^C = G_t^C = 4$ N / m for the model Drucker-Prager. Exponent $\eta = 2.284$

4 RESULTS AND DISCUSION

4.1 Comparison of measurement results with FEM calculations

Indentation test at maximum load $F = 50$ N was performed using a ZHU / zwickiLine + ZHU2.5 hardness tester. From the measurement of the glass and laminated glass, the load curve was analyzed using the Oliver and Pharr [10] method. The measured and calculated values are compared in the table 3.

Fracture toughness was calculated according to Myioshi (5), Anstis (7) and Niihara (6). The relation (5) in this case gives the results closest to the values reported for [7]. From the results of numerical simulation, fracture toughness was also analyzed according to the given relations. In addition, a method based on the energy released during crack formation was used. In the table 3, fracture toughness based on the amount of energy released is denoted as K^*_{IC}. In the relation (5), c is half the crack length, F is the load force, E is the Young modulus, and H is the hardness. In the relation (6) there are also a and k parameters. The a parameter specifies half the length of the impression diagonal and the parameter $k \approx 3$ for radial cracks.

$$K_{IC} = 0.018 \frac{F}{c^{3/2}} \left(\frac{E}{H} \right)^{1/2} \tag{5}$$

$$K_{IC} = 0.129 \left(\frac{c}{a} \right)^{-\frac{3}{2}} \left(\frac{E}{H} \right)^{\frac{2}{5}} \frac{H a^{\frac{1}{2}}}{k} \tag{6}$$

$$K_{IC} = 0.016 \frac{P}{c^{3/2}} \cdot \left(\frac{E}{H} \right)^{0.5} \tag{7}$$

Parameter	Test	FEM
Young modulus E [MPa]	73400 $\pm$900	73400
Hardness H [MPa]	6576 $\pm$ 59	6780
Half length of impression diagonal [μm]	64 $\pm$ 2	59
Half crack lenght c [μm]	266 $\pm$ 22	270
K_{IC} [MPa $m^{1/2}$] (Niihara)	1 $\pm$ 0.13	0.97
K_{IC} [MPa $m^{1/2}$] (Anstis)	0.65 $\pm$ 0.12	0.59
K_{IC} [MPa $m^{1/2}$] (Myioshi)	0.699 $\pm$ 0.09	0.66
K^*_{IC} [MPa $m^{1/2}$] (Von Mises)	-	0.49
K^*_{IC} [MPa $m^{1/2}$] (Drucker Prager)	-	0.63
W_{unrel}/W_{tot}	0.47 $\pm$0.004	0.43

Table 3: Comparison of measurement results and FEM results

Fig. 8 shows the comparison of the measured specimen (left part of the picture) and the results of the numerical simulation (right part of the picture). In both cases it is a visualization of a fully lightened state. It is obvious that the simulation results are very similar to the measurement results.

For laminated glass, the ratio of Youg modules E_2/E_1 of individual layers was analyzed using a parametric study. The measurement itself provides only information about the reduced Young's module E^*. Assuming knowledge of E_1, only E^*/E_1 can be calculated and the E_2/E_1 ratio can be deducted from the parametric study results. Due to the deformation of the top layer it is necessary to model the whole laminated structure including contact with the contact surface of the hardnes tester. The model described in 3.1 has been modified to match the measured sample. This included increasing the sample diameter to 3 cm, adjusting each layer to a thickness corresponding to the actual sample, and adding a rigid substrate with which the sample is in contact during the measurement. Ratio $E_2/E_1 = 0.00013$, as is shown in the table 4.

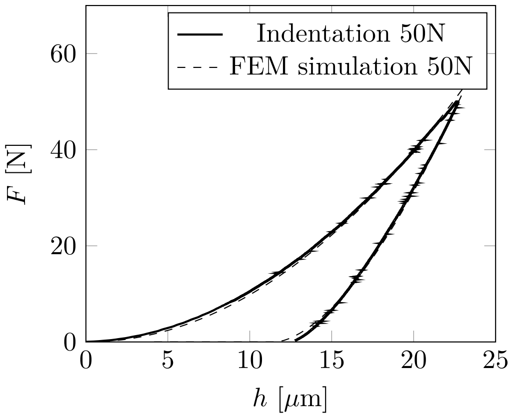

Figure 7: Comparison of measurement results and FEM simulation (indentation into glass)

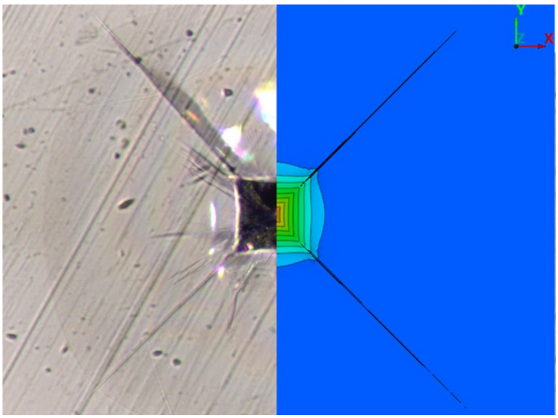

Figure 8: Comparison of measured and calculated cracks

Parametr	Měření	MKP výpočet
E^*/E_1	0.58 $\pm$0.01	0.55
E_2/E_1	-	0.00013

Table 4: Comparison of calculated and measured data

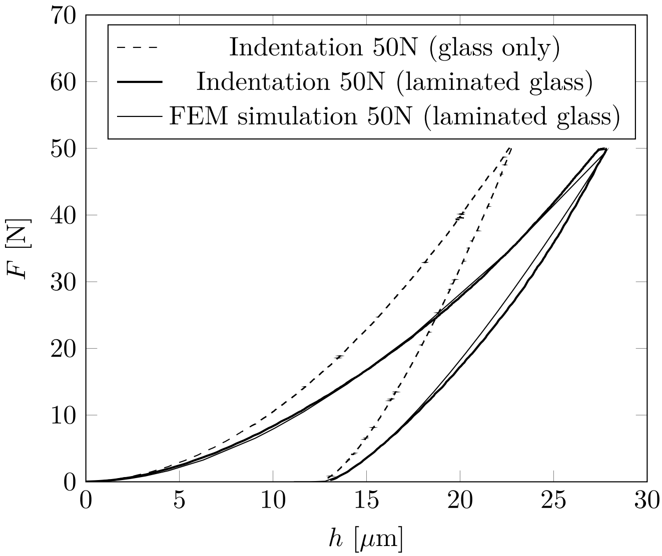

Figure 9: Comparison of measurement results and FEM simulation of laminated structure and glass itself

5 CONCLUSION

5.1 List of Activities

Several FEM models have been created to simulate indentation into hard and brittle materials. These are simplified axially symmetrical models and a 3D model allowing simulation of crack propagation. Simplified models are controlled (automated) by a program written in PYTHON. Axially symmetric models are used to determine the material model parameters of the specimen to be examined, where the specimen can be either single material or layered structure. 3D model uses characteristics based on 2D model results. This model is used to simulate crack propagation in hard and brittle materials.

These models were verified by simulation of indentation into glass and laminated glass. The results of the simulations show a very good agreement with the measurement, which was realized by instrumented hardness tester ZHU / zwickiLine + indentor Vickers.

The response (force dependence on indentation depth) of the glass sample and the laminated glass sample was measured. Hardness, Young's modulus and indentation work were calculated from the response. Fracture toughness was calculated from the size of the cracks. The calculation of fracture toughness is based on Myioshi, Anstis and Niihara relations. The results of the fracture toughness measurements show that the results are quite sensitive to the size of the crack, which shows a considerable dispersion. Measurement of fracture toughness by this technique is very simple and does not require a number of specially treated specimens, but a large dispersion of crack lengths is a major disadvantage. Fracture toughness was also calculated from the amount of energy released during crack formation. This calculation was performed on FEM simulation results with the Von Mises material model and the Drucker - Prager model. In both cases, a crack of the same area was formed, but in Von Mises the energy was half. The Von Mises response is less than 10% lower than the Drucker - Prager response. Thus, it appears that a small change in response causes a significant change in fracture energy. Fracture toughness calculated from the released energy shows lower values for both material definitions. This is probably due to the compliance of the measuring device or an imperfectly cleaned specimen. In fact, the tensile stresses below the contact surface reach higher values.

On the basis of input data from tensile and indentation test of bainitic steel, the indentor frame stiffness was calculated. It turned out that at the same indentation depth, the response with a perfectly rigid machine frame showed 25% higher force.

5.2 Summary of knowledge

To determine accurately the mechanical properties of the material by indentation, it is necessary to measure the exact response. Measuring a precise response requires a perfectly cleaned sample with a smooth contact and contact surface and an unused indenter. It is necessary to check that the device is not compliant. We can do this by testing a homogeneous specimen whose Young's modulus we know. If we measure more compliant characteristics, the device appears to be compliant. Very useful is the FEM model of the indentation process, which can reveal possible problems related to the interpretation of measured data. For example, a sample that is too thin to bend or a thin layer containing a compliant substrate.

5.2.1 Determination of Young's modulus of laminated material

Accurate determination of Young modulus of laminate cannot be done without the support of FEM model. If Young modulus of one of the E_1 layers is known, we calculate the E^*/E_1 ratio and subtract the unknown E_2/E_1 ratio from the parametric study results. E^* is a reduced Young's module calculated by [10]. The indentation depth of the laminated glass differs significantly from the indentation depth of the glass itself, although similar values should be measured by sensing the relative indentation depth (from the top of the indentor to the contact area). This is due to the deflection of the glass layer on the flexible substrate. In fact, the measured maximum depth also includes the deflection of the glass layer, as shown in fig. 10. When modeling indentation into a laminate and modeling indentation into thin samples, it is necessary to model the whole layered structure, including contact of the test specimen with the bearing surface.

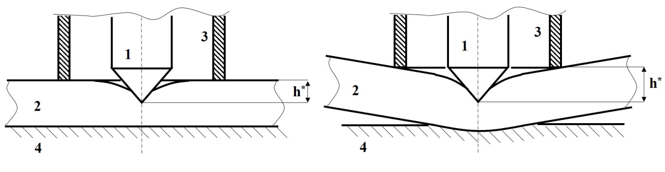

Figure 10: Measured indentation depth for perfectly rigid substrate (left), measured indentation depth for flexible substrate (right)

5.2.2 Determination of characteristics of brittle materials

Precisely measured indentation curve is a prerequisite for correct determination of material characteristics. The [10] method is then used to compute Young modules E. From the indentation curve, the ratio of the indentation work W_{unrel}/W_{tot} is calculated, ie the ratio of non-released energy to total energy. For this W_{unrel}/W_{tot} ratio, E_{pl}/E and σ_Y/E can be deducted from the results of the parametric study. In this way we define quite simply the bilinear model of reinforcement $\sigma = E_{pl}\epsilon_{pl} + \sigma_Y$ from the indentation measurement.

The results of the simulations show very good match with the measurement. It should be noted that the measurement was affected by an error caused by the compliance of the measuring device. As a result, the FEM results show simulations of lower values for crack driving force, lower initiation stresses, and lower values of expected yield stress.

5.3 Fulfillment of the aim of the dissertation

The aim of the dissertation was fulfilled. A group of models has been created to simulate indentation into hard and brittle materials. The models were verified by comparing the results of calculations with real measurements. The created model can be used not only to simulate indentation into the hard and brittle materials. Another applications can be seen in the simulation of indentation into layered materials, where, among other things, the energy needed to delaminate layers can be investigated.

The created model has practical benefit. The methods of examining fracture induced by concentrated contact are valid for a particular group of materials. For this reason, it is useful to support measurement by simulation and analysis of results, and explain any anomalies. The simulation can also predict what the response will be when indenting into an unknown combination of layers of materials. It may be shown that some combinations are not suitable for the purpose without being produced in advance.

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