Determination of mechanical properties from depth-sensing indentation data and results of finite element modeling

M G Isaenkova, Yu A Perlovich, O A Krymskaya and D I Zhuk

National Research Nuclear University MEPhI (Moscow Engineering Physics Institute)
Kashirskoe highway 31, Moscow, 115409, Russia

E-mail: dimazhuk@gmail.com

Abstract. 3D finite element model of indentation process with Berkovich tip was created. Using this model with different type of test materials, several series of calculations were made. These calculations lead to determination of material behavior features during indentation. Relations between material properties and its behavior during instrumented indentation were used for construction of dimensionless functions required for development the calculation algorithm, suitable to determine mechanical properties of materials by results of the depth-sensing indentation. Results of mechanical properties determination using elaborated algorithm for AISI 1020 steel grade were compared to properties obtained with standard compression tests. These two results differ by less than 10% for yield stress that evidence of a good accuracy of the proposed technique.

1. Introduction

Indentation is one of the most widely used techniques for determination of mechanical properties of materials. This method has many advantages compared to standard tension/compression tests [1]. It is not required to produce a standard size specimen for testing; any piece of material which can be glued to surface will be enough. In addition, it is possible to get properties of very small (eg. 100 nm) region [2] of the material or in thin films. In classic indentation, the hard indenter is pressed into material with specified force. Then, force is removed and indenter print’s size is measured. Usually it is done by using optical microscope [3]. In micro- and nanoindentation this can be a considerable problem. Atomic force microscopy [4] or scanning electron microscopy [5] can be used for this task but it is time-consuming and requires very expensive equipment. To overcome this problem and to obtain as much information about material as possible from single indentation, the depth-sensing indentation was introduced. During such test, the position of indenter is measured while load increases from zero to specified maximum and to zero. The $P-h$ diagram is obtained which is the material response to increasing load. This diagram can be processed using various techniques to determine elastic and plastic characteristics of material.

Many methods for processing this diagram exist. The most used among them and standardized in many countries is Oliver-Pharr method that allows obtaining Young’s modulus of material [6]. It uses slope of unloading part of $P-h$ diagram knowing contact area from geometry relations. This method gives decent accuracy and easy to use but it cannot give any information about material’s plastic properties. Being based on Hertz contact problem’s solution [7] it cannot be of any use for characterization of plastic deformation during indentation but only for unloading which known to be only elastic. At the same time, the problem of plastic deformation of material during indentation remains unsolved due...
to presence of many variables such as yield stress, deformation hardness exponent, creep constants. Yet, it is possible to use an empiric method to make a solution of acceptable accuracy. In first approaches, researchers used results of many experiments for different materials, which have been later processed, and then correlation between indentation data and material properties have been established [8]. Practice shows that this relation can be found but this requires much resources for preparing many materials to undertake many indentations. In addition, mechanical properties can be different in various parts of material and also in different specimens. With increasing of computational power, real experiments are often substituted by finite element (FE) modeling [9]. Advantages of FE method can be of a great use for this field. Material behavior can be easily controlled to represent different materials and calculation can be automated for series of methods.

Purpose of this work was to create such method using results of FE modeling performed and to try it on material with known properties to check results. Work steps as followed:

- Build 3D FE model for material and Berkovich indenter (3-sided pyramid)
- Test model by comparing results of experimental indentation with results of indentation simulation for materials with same properties
- Once model is known to be well representing material’s behavior during indentation, perform calculations for materials with different mechanical properties
- Summarize results, find out relations and build dimensionless function to represent them.
- Try method for material with unknown properties and compare its results with same material’s compression test’s results

2. Modelling & experimental results

2.1. FE Model
In FE representation of deformed material or construction the object is split to a number of elements, each element’s behavior is controlled. These elements can be either three, two, one dimensional, or even dimensionless. In this study, where used three-dimensional 20-node (8 nodes in corners of element and 12 nodes on edges) elements to represent volume of indented material and two-dimensional 8-node (4 nodes in corners and 4 nodes on edges).

Elements’ response to stresses below yield stress is linear while for stress above yield stress it is multi linear so it can represent exponential hardening law. Element’s size is chosen according to gradients of stress and deformation. Higher gradient require smaller elements so under indenter, where element’s displacement is maximal it is smaller.

In case of geometrical symmetry, it is common to cut model by a symmetry plane [10]. In our case, we can cut the model to leave only 1/6 and account for remaining parts by involving symmetry boundary conditions on surfaces created by this cutting. Resulting model (Figure 1) can represent full model

![Figure 1. Resulting FE model. Regions with different element density have different color. Indenter is not shown for illustrational purpose.](image-url)
without loss of accuracy. This allows reducing number of elements to 17414 and nodes to 76851, so time required for process simulation on a computer is much less than for full model. Friction coefficient is chosen 0.2, which accords for dry friction between steel and diamond.

2.2. Model evaluation
Before using model for research purposes, it is required to ensure that it behaves correctly under load. Evaluation can be done by comparing experimental $P-h$ diagram to computational for same material. Microindentation experiment requires well prepared surface of specimen. Roughness of surface will lead to great spreading of indentation results [11]. To avoid this, the specimen was polished with sandpaper of different grain size, from bigger to smaller. Later, for removing disturbed metal layer, electrolytic etching was utilized on material surface. Despite of this procedure, experimental microindentation results still have noticeable variation because of different grain orientations and various inclusions in the material. Series of indentation tests performed to minimize this variation. Results of this series were averaged and compared to simulated $P-h$ diagram (Figure 2).

![Figure 2. Experimental P-h diagram versus simulated. Experimental line is averaged from series of 12 experiments of microindentation made. Simulated P-h diagram made with FE method for material properties obtained from compression test.](image)

In order to compare simulation with experiment, simulation should be made with same material properties as indented material. All needed constants, including yield stress, strain hardening exponent, Young modulus, can be obtained from standard compression test. This type of test, involving all specimen gives macroscopic properties of material, which is in some point of view equivalent to averaging indentation results.

2.3. Simulations
To construct dimensionless functions connecting material’s constants and indentation results, series of experiments was made. Using model described in section 2.1 with following parameters: Young modulus 200 GPa, hardening exponent 0.15, yield stress from 150 MPa to 500 MPa with 50 MPa step. Each simulation’s $P-h$ diagram was processed and graphs for necessary constants were plotted (Figure 3). Usually loading line follows Kick’s Law (1), so $C$ is taken as one of the constants to link $P-h$ diagram with material properties:

$$P = Ch^2,$$

where $P$ - force on indenter, $h$ – displacement of indenter, $C$ – material constant.
Other constant $S$ is a slope of unloading curve during starting moment of unloading. Variables $h_{\text{max}}$ and $h_r$ are the maximum indentation depth and indentation depth after full unloading consequently.

Ratio of elastic to the total energy $W_e/W_t$ is calculated as area ratio under lines of $P-h$ diagram. Plastic energy can be calculated as the difference between area under loading curve and elastic energy, which is area under unloading curve.

**Figure 3.** Relation between $C$ constant in Kick’s law and yield stress of material (left). Relation between part of dimensionless equation with work ratio (right). Both graphs represent results of FE simulations. Each circle is different material with varied yield stress.

### 2.4. Method for material properties determination

Using relations given by FE simulation we can construct the method suitable to evaluate material properties with result of FE indentation. It should include following steps.

1. Process experimental $P-h$ diagram to obtain useful values, such as $C$, $S$, $h_{\text{max}}$, $h_r$, $W_e/W_t$.
2. Using Oliver-Pharr or equivalent method to obtain Young modulus with $S$ constant.
3. Obtain relation between yield stress and characteristic stress (stress at 29% deformation during uniaxial loading) from equation (2)

$$\frac{\sigma_{0.29} - \sigma_s}{0.29E^*} + 11\frac{\sigma_T}{E^*} = 1.152 \left( \frac{W_p}{W_t} \right)^2 - 2.795 \left( \frac{W_p}{W_t} \right) + 1.643 \quad (2)$$

where $E^*$ - Young modulus corrected by elastic deformation of indenter.

4. Substitute this relation to equation (3) and solve for yield stress. This equation is used in many previous works by other authors [12] [13].

$$C = M_1 \sigma_{0.29} \left( 1 + \frac{\sigma_T}{\sigma_{0.29}} \right) M_2 + \ln \left( \frac{E^*}{\sigma_T} \right) \quad (3)$$

where $M_1 = 6.02$ and $M_2 = -0.875$ for Berkovich indenter.

5. From exponential hardening law calculate strain hardening exponent

$$n = \frac{\ln \left( \frac{\sigma_m}{\sigma_{0.29}} \right)}{5.01} \quad (4)$$

Method was tested using indentation results for AISI 1020 steel. Set of material properties estimates using method above was compared to compression standard test results. Yield stress value lies within 10% error interval, which is good for indentation methods. For calculation of Young modulus Oliver-Pharr method was utilized. Results of comparison shown in table 1.
Table 1. Results of material’s properties estimations using standard compression method and indentation.

| Method     | Young modulus (GPa) | Yield stress (MPa) | Strain hardening exponent |
|------------|---------------------|-------------------|---------------------------|
| Compression| 215                 | 340               | 0.13                      |
| Indentation| 213                 | 375               | 0.16                      |
| Error      | 0.9%                | 10%               | 23%                       |

3. Conclusion
Finite element model for simulation of indentation using Berkovich indenter was build. Model was test by comparing its results to experiment of indentation and good accuracy was observed. Method for estimation of mechanical properties was made using simulations conducted with FE model. Method tested on AISI 1020 steel and 10% error for estimation of yield stress was found comparing to standard compression test.

Acknowledgements
This work was performed within the framework of the Center of Nuclear Systems and Materials supported by MEPhI Academic Excellence Project (contract № 02.a03.21.0005, 27.08.2013).

References
[1] Johnson K L 1970 J. Mech. and Phys. of Solids 18 2 115
[2] Harvey S 1993 J. of mat. res. 8 1291-99
[3] Giannakopoulos A E, Larsson P-L, and Vestergaard R 1994 J. of solids and str. 31 2679-708
[4] Petzold M, Landgraf J, Füting M and Olaf J M 1995 Thin Solid Films 264 153
[5] Byong-Taek L, Nishiyama A and Hiraga K 1993 JIM 34 682
[6] Oliver W and Pharr G 1992 J. of mat. res. 7 1564-83
[7] Spence D A 1975 J. of elast. 5 297
[8] Doerner M F, Gardner D S and Nix W D 1986 J. of Mater. Res. 106 845
[9] Laursen T A and Simo J C 1992 J. of Mater. Res. 703 618
[10] Chandrupatla T R Belegundu A D, Ramesh T and Ray C 1997 Introduction to finite elements in engineering (Upper Saddle River: Prentice Hall) pp. 279-300
[11] Gilbert J L, Cumber J and Butterfield A 2002 J. of biomed. mater. res. 612 270
[12] Giannakopoulos A E and Suresh S 1999 Determination of elastoplastic properties by instrumented sharp indentation. Scripta mater. 40 1191-98
[13] Venkatesh T, Van Vliet K J, Giannakopoulos A E and Suresh S 2000 Scripta mater 42 833