

THE INFLUENCE OF TILT ON BERKOVICH INDENTATION OF A STEEL SAMPLE USING FEM ANALYSIS AND NANOINDENTATION

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Abstract

The main shape deviations of a Berkovich indenter are the indenter bluntness and indenter tilt. The influence of the Berkovich indenter tilt on the results of the FEM modelling of steel nanoindentation is evaluated in this paper. The indenter tilt has an impact on the indentation curves and contact area. The X5CrNiCuNb 16-4 steel nanoindentation was modelled by FEM and the indentation curves were obtained. The Berkovich indenter was blunted and the tilt of the indenter was changed to determine its influence on the indentation curves. The results showed that growing tilt shifts the indentation curves to higher values of indentation forces and makes unloading curves steeper which is in agreement with a larger contact area. The calculated indentation curves were compared with the experimental indentation curves and the impact of the indenter tilt to the indentation curves was determined.

Keywords: *nanoindentation, indenter shape deviations, indenter tilt, indenter bluntness*

INTRODUCTION

Nanoindentation test

The nanoindentation test is often used to determine the Young modulus and hardness of the bulk materials or thin layers. At this test, the indenter is pressed into the specimen and the dependency of the indentation force on the indenter displacement (indentation curve) is measured. The Young modulus and hardness can be obtained from the indentation curve using the Oliver-Pharr analysis [1].

To perform the Oliver-Pharr analysis, the indentation curve and the indenter shape function have to be known. It is the dependency of the projected contact area (A_c) on the depth of the indentation without sink-in effect (h_c). The indenter shape function for the ideal indenter can be calculated by (1).

$$A_c = 24.56 \cdot h_c^2 \quad (1)$$

The indenter shape function determined by (1) is usually not enough accurate to describe the contact area when the experiment is done. The main reasons are the shape deviations of the indenter and the surface of the specimen and the pile-up effect [2].

This work was presented at the conference Local Mechanical Properties 2022 held in Kosice, Slovakia on May 11-13, 2022.

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At the nanoindentation, the surface near the indenter can bend under the indenter and then the projected contact area gets smaller, this is called the sink-in effect. The sink-in effect is included in the commonly used Oliver-Pharr analysis. If the plasticity of the specimen is huge, then the plastically deformed material accumulates under the indenter and creates a pile-up region. The pile-up makes the contact area bigger and it needs to be corrected to get correct results of Young's modulus or hardness [3].

FEM modelling of the nanoindentation test

The modelling with FEM can be used to study the results and the influences of the nanoindentation test without preparing the specimens and measurements. The FEM can be used to determine the contact pressure, the strain and stress fields under the indenter, and the border of the pile-up region. The FEM was used to calculate the correction factor β for the conical and Berkovich indenter [4] and can be inversely used to determine the parameters of the model of material from known indentation curves [5]. With the help of FEM, the influences of the indenter bluntness [6] and specimen tilt can be studied.

The impact of shape deviations

The shape deviations are crucial to the determination of the correct value of the projected contact area. The bluntness of the indenter has the highest impact on the results. The growing bluntness makes the contact area bigger and it needs to be included in the indenter shape functions, which is determined at the calibration [1]. When the FEM modelling is used with the ideal shape of the indenter, there is no calibration and then the indentation curves have different shapes but the determined values of the Young modulus are the same. The impact of the indenter bluntness on the indentation curves was determined in [6].

Another shape deviation is the mutual tilt of the indenter and specimen. Although the tilt is included in the shape function at calibration too, the measurement is done on another specimen, whose surface has a different tilt than the specimen with the reference material. This tilt influences the contact area [7] and it can influence the indentation curves too. The main aim of this work is the determination of the indenter tilt influence on the indentation curves. The influence of the indenter tilt in the plane of symmetry will be determined and then the plane of tilt will be rotated to check the influence of tilt in another axis. The effect of indenter tilt was determined for fused silica, which has sink-in behavior [7] but not for materials with pile-up as steel, which will be the main aim in this paper.

METHODS

Model of material

The indentation of the X5CrNiCuNb 16-4 steel was experimentally measured and the same material was expected for the FEM modelling. To get a precise description of the behavior of this material, the tensile test was done (Fig. 1) and then it was modelled by FEM to calibrate the model of the material.

The stress-strain curve of the steel (Fig. 2) is composed of three parts. The first part is the linear-elastic model of the material. Young's modulus was used from the experimental measurement ($E_s = 202$ GPa). The Poisson's ratio was not measured but a commonly used value ($\nu = 0.3$) was used.

The second part of the steel stress-strain curve starts with the first plastic deformation. The multilinear elasto-plastic model with isotropic hardening was used to describe the stress-strain curve by eleven points on its and connected by lines (Fig. 2). To get the most precise results, the first point of the multilinear elasto-plastic model was

placed at a point, where the start of deviation from the line in the experimentally measured stress-strain curve was apparent. At this part, the few points of the multilinear model were fitted to experimentally measure the stress-strain curve. This curve can be used until the reach of the ultimate strength. At this point, the experimentally measured stress-strain curve is not valid anymore due to the start of necking. When necking occurs, the plastic deformation starts to localize in the small area in the specimen center and the measured stress-strain curve cannot be used for the model of material for FEM anymore.

To describe the part of the stress-strain curve after necking, the FEM model of the tensile test was done. The small imperfection was added to the FEM model to get the neck in the center of the specimen. With this model, the FEM simulation of the tensile test with necking was calculated. A few other points were added to the stress-strain curve and their values were iteratively changed to get the same force-displacement curve as was measured by the tensile test. The necking was included in the FEM model and then it was comparable with the experimental measurement (Fig. 1). The calculated curves gave great conformity with the experimental force-displacement curve from the tensile test and then determined points could be added to the model of material (Fig. 2).

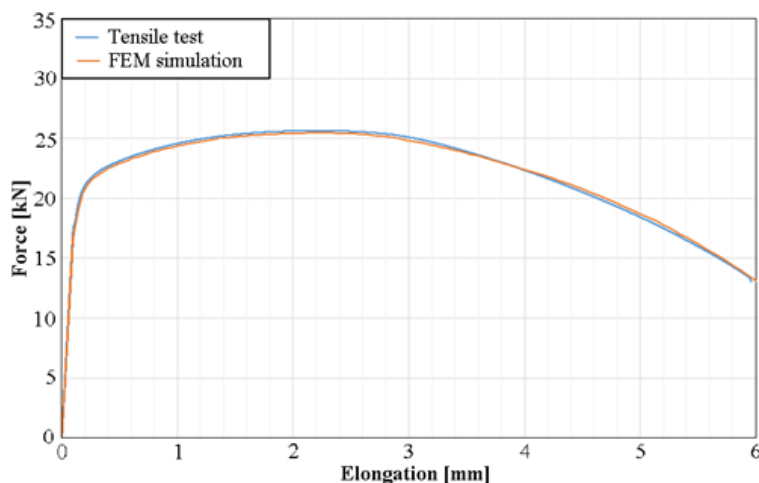


Fig. 1. Comparison of the tensile test results and the force-elongation curve calculated by FEM.

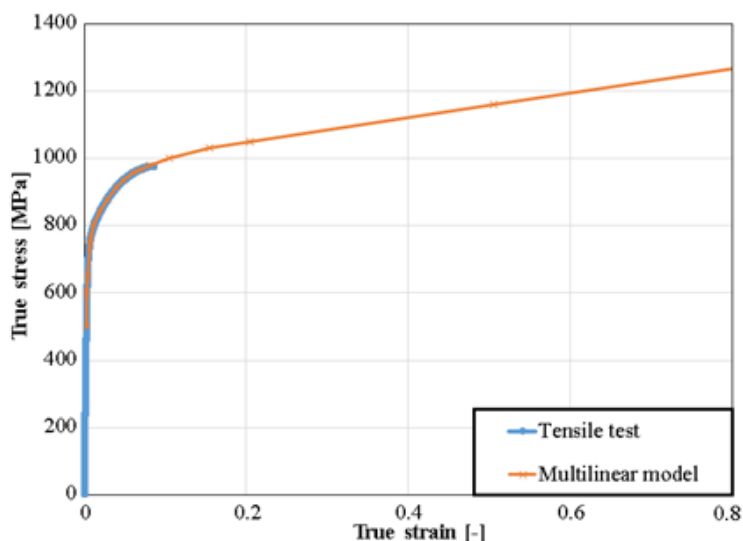


Fig. 2. The calculated parameters of the model of material, subsequently used for the modelling of the nanoindentation test.

FEM model of indentation with blunted indenter

Model of geometry – The geometry of the specimen and modified Berkovich indenter (referred to as Berkovich indenter) was created. The dimensions (Fig. 3) were chosen to be at least 100 times bigger than the depth of indentation, to prevent the influence of the results by the edge of the specimen. The Berkovich indenter was expected as blunted. The bluntness of 400 nm was applied to its tip and edges. This bluntness was chosen to be slightly lower than is allowed by ISO 14577-1. [8], where the bluntness of 500 nm is allowed for the used depths of indentation. The bluntness is shifting the indentation curves to the higher values of forces and can have a significant impact on the results [6]. The blunted Berkovich indenter has three planes of symmetry and due to the symmetry of the geometry, material, boundary conditions, and expected symmetry of the results, only one sixth of the indenter and specimen was modelled.

Boundary conditions – Due to the symmetry, the displacement perpendicular to the planes of symmetry was fixed on the areas in the planes of symmetry. The displacement in the way of z axis was fixed on the lower area of the specimen (Fig. 3). The specimen was loaded by the displacement of the Berkovich indenter. The loading was divided into two load steps. In the first load step, the displacement of 500 nm plus the gap between the specimen and the blunted indenter, caused by the bluntness, was applied on the upper area of the indenter. In the second load step, the indenter was moved back to separate the indenter and specimen. The large displacements were expected and the loading and unloading phase were divided into more than 100 substeps.

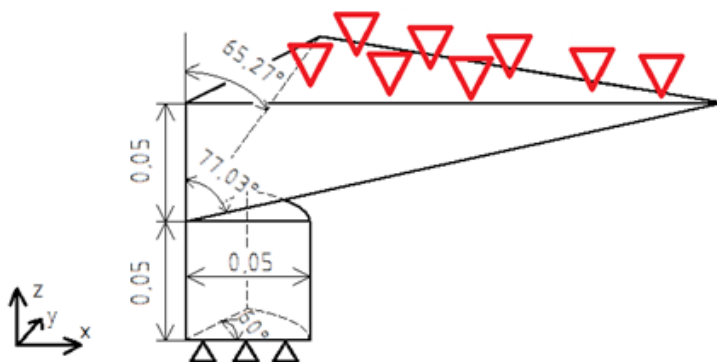


Fig. 3. The geometry and boundary conditions used for FEM.

Mesh – The mesh (Fig. 4) was created from the volume elements with quadratic base functions (SOLID 186). The mesh was from hexaedrical coarse elements far away from the contact region and very fine elements under the indenter. The transitional volume from tetraedrical elements was created between these two volumes. The indenter was meshed with elements with tetraedrical shape, which were fine on the tip of the indenter and coarse in the far volume. The contact region was created between the specimen and indenter and meshed with the CONTA 174 and TARGE 170 elements. The surface of the specimen was chosen as a contact area and the surface of the indenter as a target. The factor of penetration tolerance was lowered to get the penetration lower than 1 nm. The steel has a pile-up behavior and then the coefficient of friction needs to be used between the indenter and specimen. The value of $\mu = 0.1$ was assumed [9].

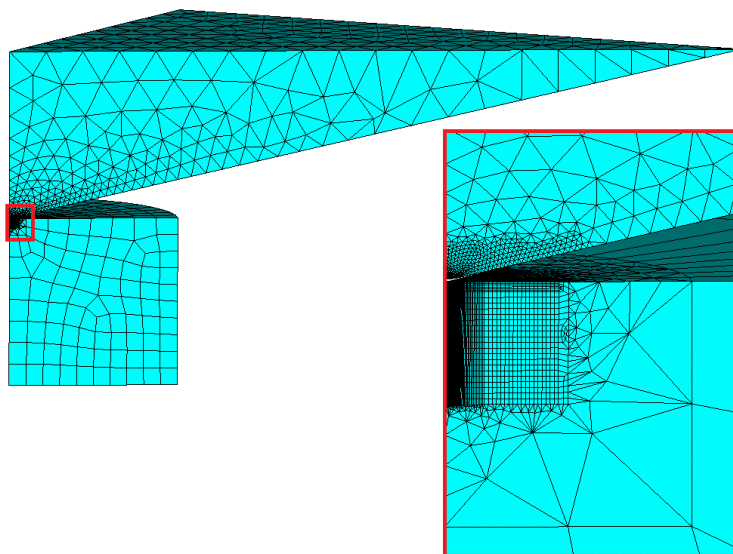


Fig. 4. Mesh used for FEM calculations.

FEM model of indentation with blunted indenter and tilted specimen

To calculate indentation curves for the tilted specimen, the FEM model was modified. The specimen was tilted in the plane, which includes the edge of the Berkovich indenter (Fig. 5). The tilt was chosen from 1° to 5° with the step of 1° . Due to the tilt of the specimen, there are not three planes of symmetry anymore. Only one plane of symmetry, which is the same as the plane of the tilt, left; therefore, half of the model was modelled. The displacements perpendicular to this plane were fixed on it and the displacement in the x-axis needed to be fixed on the lower area of the specimen and the upper area of the indenter. It was verified that these changes do not influence the results of the calculations.

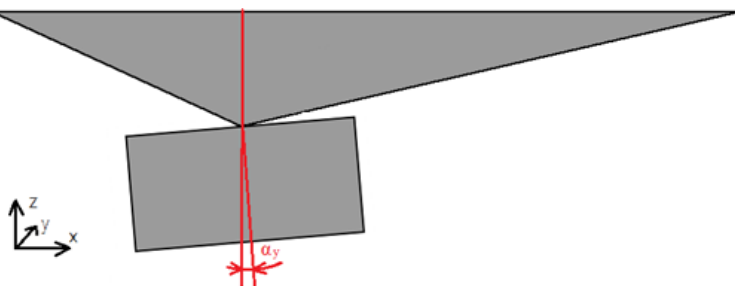


Fig. 5. Indentation with tilted specimen.

FEM model of indentation with rotated plane of tilt

The specimen tilt is often general; therefore, the tilt plane does not have to be the plane with the edge of the indenter. To check the influence of the indenter tilt plane, the plane of the tilt was rotated from 0° to 60° with the step of 20° , and the FEM calculations were done. The rotation of about 60° was calculated with the same model as was used for the tilting in the plane with the edge of the indenter, only the tilt was done in the opposite direction. To calculate two other cases (rotation of the tilting plane about 20° and 40°), the full model needed to be done. The displacement in all directions was fixed on the lower area of the specimen and displacement in the x and y axes were fixed on the upper areas of the indenter. In this case, the computational time grew up a lot. To make the task be calculated in less than 5 days, the mesh had to be coarser. It could slightly influence the accuracy of the calculated contact pressures, which were not evaluated, but the displacements and forces, which are less sensitive to mesh quality should be accurate enough, so the change of mesh should not significantly influence the calculated indentation curves.

RESULTS AND DISCUSSION

The influence of the indenter tilt

The first calculations were made with the blunted Berkovich indenter without the tilt of the specimen. The calculated indentation curves (Fig. 6) showed lower values of the indentation forces than experimental data. This can be caused by either higher bluntness than was expected in the calculation [6] or by the tilt of the indenter.

The results of FEM calculations show higher values of indentation forces with growing tilt. The difference in the curves caused by the 1° tilt is getting bigger with the growing specimen tilt. At the tilt of 5° , the calculated loading curves show great correlation with the experimental data. The unloading curves fit experimental data well in the first half of unloading. There is a discrepancy in the last phase of unloading, which can be caused by

the more complex plastic deformation caused by the residual stresses. This discrepancy should not influence the commonly used Oliver-Pharr analysis, because, the only first part of the unloading curve is usually used.

The growing values of the maximal force and slightly growing unloading stiffness are in agreement with [7]. In this paper, the indenter shape function for the ideal tilted indenter was analyzed with the result that the contact area is growing with the tilted specimen. To get the correct value of Young's modulus, the higher values of the indenter shape function are related to the steeper unloading curve.

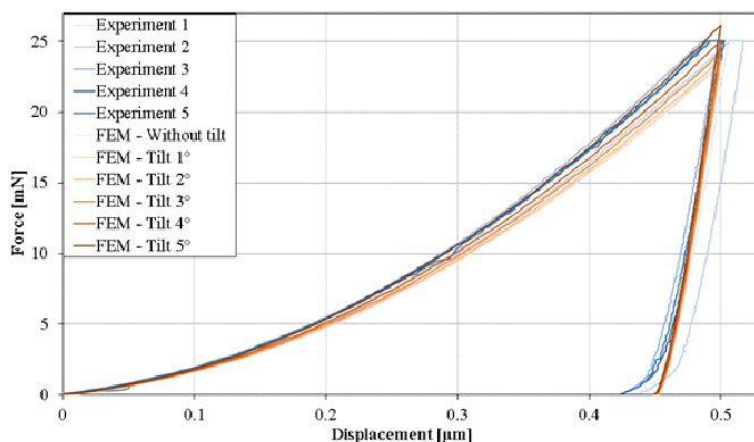


Fig. 6. Comparison of the indentation curves calculated for the different indenter tilts with the experimentally determined indentation curves.

The influence of the rotation of the tilt plane

After the determination of the influence of the specimen tilt in the plane of the edge of the specimen, the calculations with the rotated plane of the specimen tilt were calculated and the influence of the direction of the tilt was determined. The calculated indentation curves for the tilt of 5° in the plane rotated about 0° , 20° , 40° , and 60° were compared (Fig. 7). Due to the geometry of the indenter and the specimen, higher angles of the rotation of the tilt plane are symmetrical with calculated results.

The indentation curves show that the rotating of the specimen tilt plane influences results only slightly. When the tilt is in the plane of the indenter edge or rotated by 10° , the indentation curves have nearly the same results. With the growing rotation of the tilt plane, the maximal values of the forces slightly decrease. The biggest difference is for the tilt plane in the center of the indenter side surface (opposite tilt to the indenter edge). The relative difference to the tilt in the 0° plane is 3 %. The results show that the rotation of the plane of indenter tilt has a smaller influence on the results opposite to the angle of indenter tilt or indenter bluntness [6].

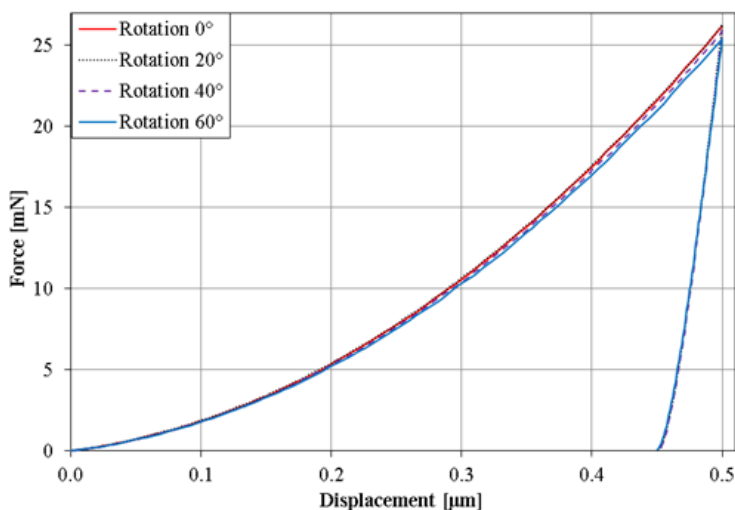


Fig. 7. Calculated indentation curves with the 5° tilt of specimen and rotated tilt plane.

The Young's modulus and hardness

To determine the Young's modulus and hardness, the Oliver-Pharr analysis [1] was used. Due to the pile-up, the experimental results were distorted, due to the inability to measure the correct value of the projection of the contact area. For the results obtained by FEM, the contact area was determined from the surface of the elements in contact projected to the plane of the specimen surface. The correction factor for the influence of radial displacements $\beta = 1.06$ [10, 11] was used. The calculated value of reduced modulus for the indentation without tilt was 191 GPa, which was the same as for the indenter with 5° tilt. When the rotation of the tilt plane at about 60° occurred, the determined reduced modulus dropped to 186 GPa. Similar results were obtained for hardness, which was 3.0 GPa for the indenter without tilt, 3.1 GPa for 5° tilt, and 3.1 for the rotated plane of the tilt. The calculated reduced modulus 191 GPa matches with the specimen Young's modulus 204 GPa, which is nearly the same as the value measured from the tensile test and used for FEM calculations which was 202 GPa. All calculated results show that the indenter tilt does not have any significant impact on the determined values of Young's modulus and hardness if the contact area is correctly determined.

CONCLUSIONS

The influence of the indenter tilt on the indentation curves was analyzed in this paper. The model of material X5CrNiCuNb 16-4 steel was fitted to experimental data and verified by FEM and then used for the FEM calculation of the nanoindentation with the blunted and tilted indenter. The growing indenter tilt makes higher indentation forces. This is in agreement with [7] which shows higher contact surfaces caused by the indenter tilt. At the nanoindentation test, the specimen is tilted in a random direction. To analyze this influence, the full model of the indenter and specimen was done, and the tilt plane was rotated from 0° to 60° with step 20°. The higher angles of tilt plane rotation are symmetrical with the calculated results. The results show that there is only a small difference in the indentation curves with the rotating tilt plane. In our previous paper [6], the influence of the indenter bluntness on the indentation curves was analyzed. The

comparison of all influences indicates that the most important influence on nanoindentation is caused by the indenter bluntness. The tilt could have a significant impact on the indentation curves but if the contact area is corrected, the calculated Young's modulus and hardness are not influenced. The influence of the tilt plane rotation is not so much important and usually can be omitted from another FEM calculation. This is important because the symmetry can be used to reduce the model and save computational time.

ACKNOWLEDGMENT

The tensile test was measured by the Institute of Material Sciences and Engineering of the Faculty of Mechanical Engineering, Brno University of Technology.

This study was realized with the support by the grant FSI-S-17-4386 and with the institutional support RVO: 61388998.

Computational resources were supplied by the project "e-Infrastruktura CZ" (e-INFRA CZ LM2018140) supported by the Ministry of Education, Youth and Sports of the Czech Republic.

The authors also gratefully acknowledge the support by the Operational Program Research, Development and Education, projects No. CZ.02.1.01/0.0/0.0/17_049/0008422 and No. CZ.02.1.01/0.0/0.0/16_019/0000754 of the Ministry of Education, Youth and Sports of the Czech Republic. R.C. and J.T. also thank the project No. TN01000038 of the Technology Agency of the Czech Republic for support of their workplace.

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