



Revue des Matériaux & Energies Renouvelable

Journal home : <https://www.univ-relizane.dz>

ISSN : 2507-7554

E- ISSN : 2661-7595



Validation of experimental tests of NANOINDENTATION with DIGITAL simulation

Open
Access

Djidel Salah Eddine¹ , Mahmoudi Noureddine² , Abel Hurtado-Macías³

¹Industrial engineering and sustainable development laboratory, Relizane university center, 48000 Relizane, Algeria

²Département de Génie Civil & Hydraulique, Université Tahar Moulay Saïda, 20000

³Metallurgy and structural design, Centro de Investigación en Materiales Avanzados S.C., and National Nanotechnology Laboratory, Miguel de Cervantes 120, Complejo Industrial Chihuahua, Chihuahua, C.P. 31109, Mexico

RESUME

Article history:

Received 30 December 2020

Received in revised form 31 December 2020

Accepted 03 July 2021

Keys word: mechanical characterization, nano indentation, finite element method, numerical simulation

In many fields of engineering, Copper and bronze and brass are used. In order to improve the improvement of their uses, it is important to have a reliable technique to determine their mechanical characteristics. The nano indentation technique is a versatile non-destructive test which is recently applied. This document presents a numerical simulation model of the nano indentation test using the commercial finite element code ABAQUS with a BERKOVITCH indenter. For extracting the mechanical characteristics of its materials, such as hardness, and modulus of elasticity, in order to understand the principle and operation of this new method.

Copyright© 2022 - All rights reserved

1. Introduction

In the field of mechanics, the nano indentation technique is rapidly gaining attention over the past twenty years as a useful experimental tool [1-3]. For the characterization of mechanical properties Nano indentation is mainly used in small local areas of the sample. This technique is applied to small parts without disturbing their structure, is because it requires samples of small volume that can be taken without destroying the constructive element studied, it is considered a non-destructive (NDT) and in allowing indentation depths of less than 200 nm (ISO 14577-1 [5]). Elastic mechanical parameters of materials can be determined. In the use of this technique especially in the part of unloading force-displacement data. However, for copper and bronze and brass, the elastic response is influenced by the elastic characteristics. Even at shallow depths, the results of the Nano indentation test on coated specimens. In most cases, it is accepted that to minimize the effect of stiffness on the measured load displacement curve, the thickness should be at least ten times greater than the penetration depth of the entry [4] , during the indentation program. The resulting indentation curve will continuously record the indentation load versus displacement, Usually representing the penetration depth of the indentation, the indentation program consists of the following steps: up to a certain load level prescribed loading, and the holding period at maximum load and unloading. By different experimental method The young's modulus and the indentation hardness can be estimated using the unloading part of the indentation curve from which the mechanical properties are obtained. To evaluate the nanoindentation data has its restriction to linear elastic housing The theory behind commonly applied methods and does not take into account the condition of the initial structure and residual stresses. To solve the corresponding mathematical problem recent

numerical simulations of the nano indentation experiment, which use the finite element method (FEM) to evaluate the mechanical properties have been used in elastic and plastic stress regions [7, 8] .

This numerical and experimental simulation gives the opportunity to obtain not only the hardness and the elastic modulus of the material studied, but also the distribution of the stress and strain fields under the tip of the input and to calculate the mechanical parameter of the model. .

2. Theoretical aspects

In the nanoindentation test, the indenter is pushed into the surface of the sample producing deformation of the material (Fig.1). the displacement *h* and the load *P* are monitored with great precision, in the nanoindentation machines shown schematically in (Fig 2).During the nanoindentation process, the indenter enters the sample up to the maximum load *P* max, where the corresponding penetration depth is *h* max. When the penetrator is removed from the sample, unloading displacement is also continuously monitored until the load reaches zero and a final or residual penetration depth *h*f is measured.

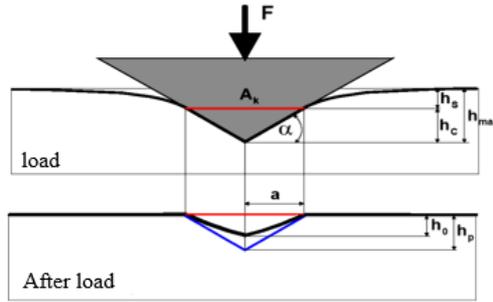


Fig 1 Elasto-plastic deformation at the maximum applied load *P* max and plastic deformation after releasing the load.

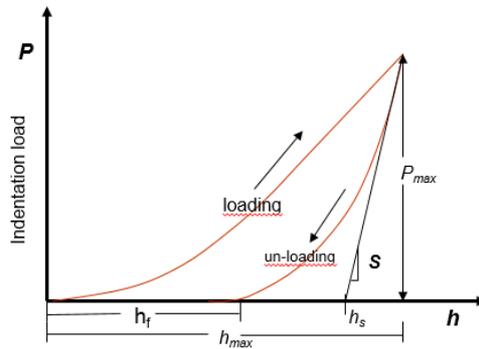


Fig 2 Force-displacement curve indentation test

to accurately calculate *H* and *E* from the indentation charge displacement data, Oliver and Pharr developed a method in the 1990s without the need to measure deformed area under the microscope .

the contact stiffness *S* by differentiating the unloading curve adjustment, and evaluate the result at the maximum depth of penetration:

$$S = \left(\frac{dP}{dh} \right)_{h_{max}}$$

As mentioned above, depth-sensing indentation measurements are used to determine the hardness and the Young’s modulus. The hardness, *HIT*, is evaluated by (e.g., Oliver and Pharr, 1992)

$$H_{IT} = \frac{P}{A_{cp}}$$

where *P* is the maximum applied load and *A_{cp}* is the contact area of the indentation, at the maximum load. The reduced Young’s modulus, *E_r*, is determined from (e.g., Sneddon, 1965; Oliver and Pharr, 1992)

The contact depth is estimated according to:

The method of Oliver and Pharr [sink-in] (Eq. (1)), and the method of Loubet et al. (pil-up) (Eq. (2))

$$h_c = h_{max} - \varepsilon \frac{P}{S}$$

$$h_c = 1,2 \left(h_{max} - \varepsilon \frac{P}{S} \right)$$

where ε is a constant which depends on the geometry of the penetrator . the value $\varepsilon = 0.75$ become the value used for the analysis According to an empirical observation with Berkovich and cube corner indenters. h_{max} is the maximum depth P is the maximum applied load and S is stiffness.

an ideal Berkovich indenter, the projected area can be calculated as:

$$A_{cp} = \frac{P}{24,5h_c^2}$$

The reduced Young's modulus, E_r , is determined from (e.g., Sneddon, 1965; Oliver and Pharr, 1992)

$$S = \frac{2}{\beta\sqrt{\pi}} E_r \sqrt{A_{cp}}$$

where β is a geometrical factor depending on the indenter (berkovitch 1,034) . E_r is the reduced elastic modulus of the contact defined as:

$$\frac{1}{E_r} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i}$$

with E and ν are the elastic modulus and the Poisson ratio of the sample and E_i and ν_i the elastic and Poisson's modulus report of the indenter. during nanoindentation For penetrators such as the Berkovich pyramid, for considering that the sample and the indenter have an elastic deformation The module reduced to Eq. (17) is used. To indent it in diamond, the values are frequently used $E_i = 1140\text{GPa}$ and $\nu = 0.07$. Equation (18) requires knowing the sample report which is generally unknown. One possibility is to use a value $\nu = 0.33$ which produces in most materials on an uncertainty of 5% in the calculated value of E.

3. numerical simulation

The commercial finite element code ABAQUS is used for these digital examples. the contact pair model is first defined, In ABAQUS. The body of the master (indenter) and the slave (deformable sample) is identified; the neglect of friction between the body of the master and the slave. It is also assumed that the tip of the input is initially in contact (point contact) with the sample (slave body). The simulations are carried out in a state of great tension.

3.1. Indenter and specimen contact

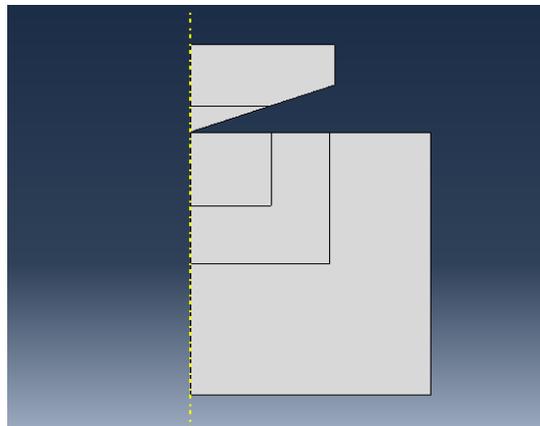


Fig. 3. Indenter et un modèle de contact de spécimen dans Abaqus

3.2. Abaqus

In this model, an indenter is pushed through three deformable tests by applying a load to the focal point of to examine the elastic plastic deformation of the materials. The indenter material is harder than the test material. Young's modulus penetration size = 1000 GPa, poisson coefficient = 0.07, angle of $\nu 70.3^\circ$. Copper Modulus of elasticity 100 GPa, Poisson ratio 0.33.

3.3. Limit conditions

The 2D axisymmetric problem in the Oxy coordinate system is considered with the axis of symmetry along the y direction in our study. The contact between the indenter and the sample is frictionless.

Along the axis of symmetry, no lateral displacement is allowed to preserve the axisymmetric formulation So the displacement in the x direction is set to zero. in both directions in fixed Displacements on the bottom of the model. The load applied to the indentation can be defined by the displacement or by the force following the classic indentation program the load is applied, During the simulation, which means that we have active loading and unloading steps which are mounted according to the particular loading program used in the nano indentation tests.

Résulte and discussion

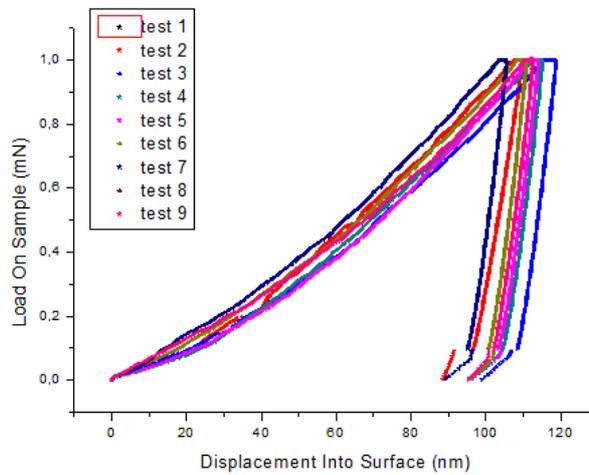


Fig .4

Table a presents the experimental results obtained from copper

Test	E GPa	H Gpa	Displacement nm	Load mN	Stiffness N/m
1	116,7	2,64	109,8	0,998	81610
2	193,4	2,36	113	0,994	133654
3	145,8	2,21	118,8	0,995	108442
4	150,6	2,34	115,1	0,998	108543
5	149	2,38	114,1	0,995	106563
6	174,1	2,47	110,9	0,995	119635
7	175,7	2,7	105,5	0,995	115270
8	165	2,41	112,7	0,998	115649
9	161,4	2,52	110,1	0,998	111015
Mean	159,1	2,45	112,2	0,996	111154
Std. Dev.	22	0,15	3,7	0,002	13800
% COV	13,82	6,25	3,33	0,16	12,41
Minimum	116,7	2,21	105,5	0,994	81610
Maximum	193,4	2,7	118,8	0,998	133654

Hardness of copper

The hardness is varying between 2,71GPa <math> < H_{op} < 2,52 \text{ GPa}</math>

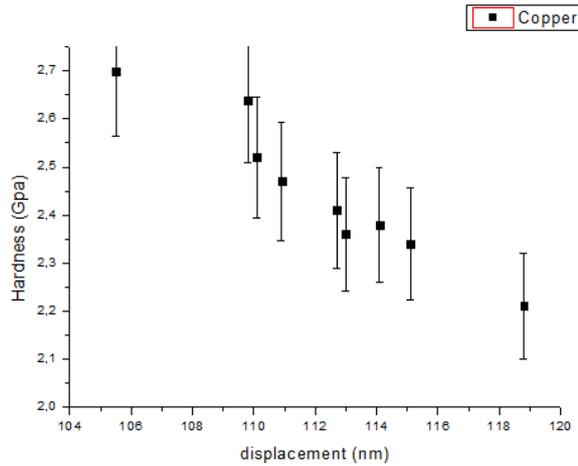


Fig.5. shows hardness vs maximum displacement

we note that the small displacement h [105,5475] nm with the maximum load 0.9950 mn has the greatest hardness [175.7] GPa.is then we note that the greatest displacement h [118.7633] nm with the maximum load 0,995 mn has the smallest hardness obtained [2,21] GPa. This study presents that the hardness has a relation with the displacement.

Young’s modulus

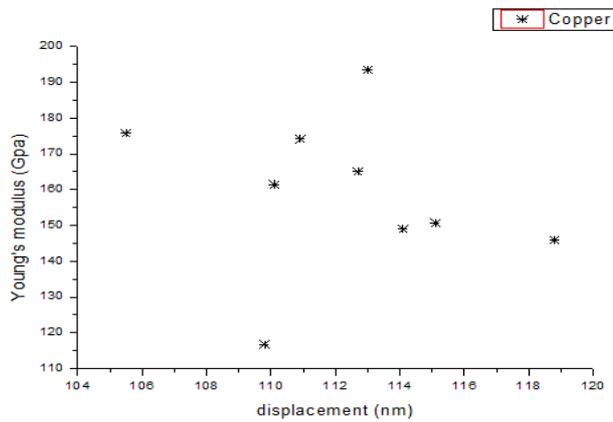


Fig.6. Young’s modulus vs maximum displacement

we note that the small displacement h [105,5475] nm with the maximum load 0.9950 mn has the greatest hardness [2,71] GPa.is then we note that the greatest displacement h [118.7633] nm with the maximum load 0.9949 mn has the smallest hardness obtained [145.8] GPa. This study presents that the hardness has a relation with the displacement.

In the calculation, the elastic deformation occurs in the beginning of the process. The Mises yield criterion is applied in the occurrence of the plastic deformation. The Mises stress equation is given by the expression

$$\sigma_{Mises} = \sqrt{\frac{(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2}{2}}$$

where σ_1 , σ_2 and σ_3 are the three principle stresses. When the σ_{Mises} reaches the yield strength (σ_0), the specimen starts to deform plastically. There is no strain hardening behavior of the specimen considered in the model.

Discussion

the copper nanoindentation loading-unloading process was simulated, with the performance of finite element analysis (FE),

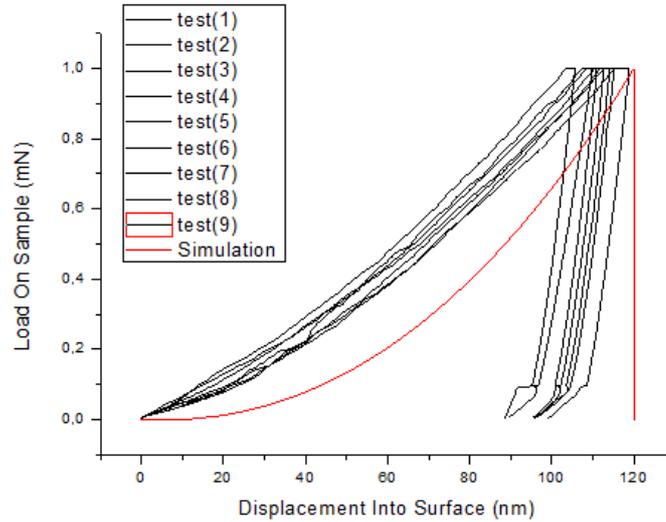


Figure.7.the load displacement curves

Figure 7 shows the load displacement curves resulting from with the prediction of the simulation modeling, and the input data from Table 1. Since the experimental data was used as the basis of the simulation, such as the limit of elasticity and Poisson's ratio, it is not surprising that there is good agreement between the simulation and the experimental results.

Another study was carried out in order to extract the mechanical properties such as hardness using the developed FE model. From the load displacement curves, the unloading curves were used to derive the hardness values by the analytical technique developed by Oliver and Pharr. (11) the comparison of hardness FEM is 91,83% between experiment. The calculated hardness is approximately 2.25 GPa, which is in good agreement with experience. It should be noted that FEM is able to extract the mechanical properties of the material such as hardness. In order to better understand the deformation behavior of the material when the indentation depth is increased. The simulation of the development of plastic deformation in copper has been studied

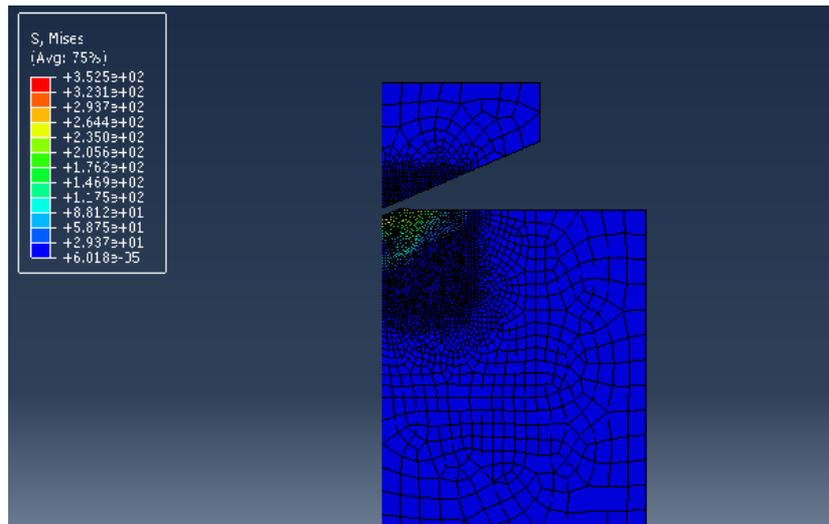


Figure.8. the propagation of the plastic deformation zone in the copper

Figure 8 shows the propagation of the plastic deformation zone in the copper. At the start, the plastic deformation of the sample at the interface (between the indenter and the sample) is initiated and then propagated. At small indentation depths, plastic deformation takes place around the tip region of the indenter and propagates both vertically and laterally as a round

shape At greater indentation depths, plastic deformation also propagates vertically and laterally. Further increase in depth results in further lateral propagation of the plastic zone in the material, as shown in Figure 8. In order to better understand the developed plastic deformation, figure 9 shows the propagation of the plastic deformation zone in the three-dimensional (2-D) model.

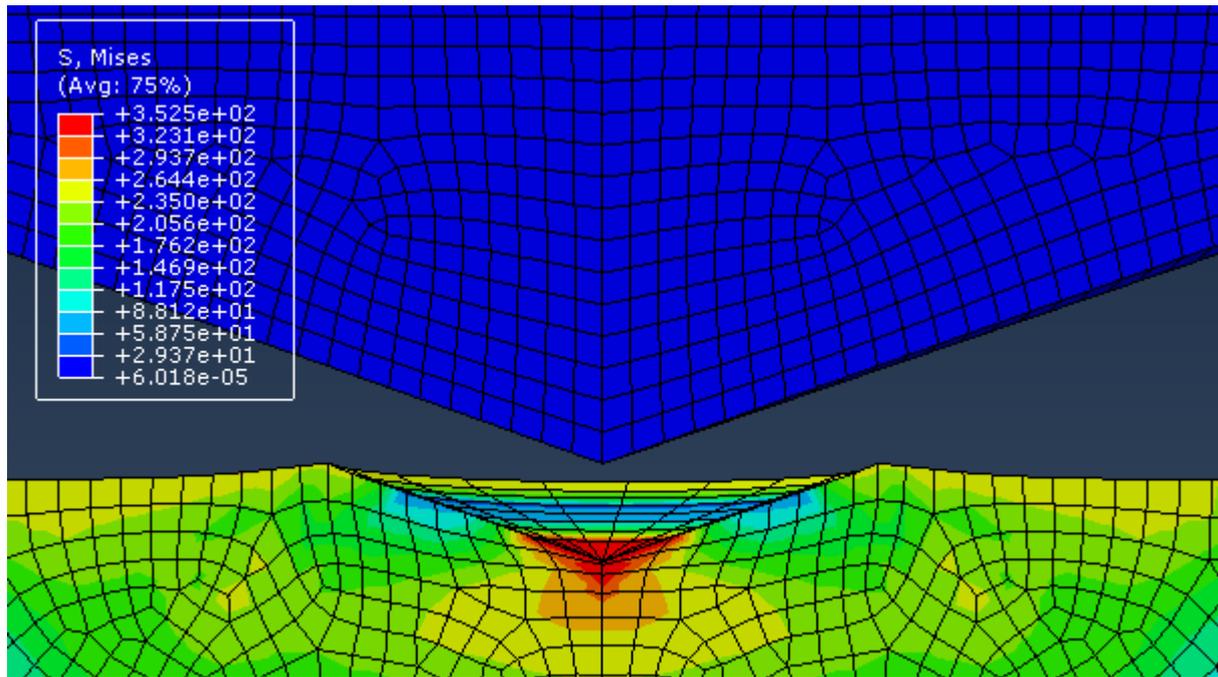


figure .9.the plastic deformation zone in the three-dimensional (2-D) model

CONCLUSIONS

The instrumented indentation test is a technique used to estimate the mechanical properties of materials such as hardness and Young's modulus. This indentation study was performed on a solid metallic material which is copper. The FE model was developed to simulate the nanoindentation response of copper. The model is able to simulate the loading and unloading stages of plastic deformation behavior during the indentation process. The applicability of the model was investigated by copper nanoindentation. The results of the simulation are in good agreement with the experimental results.

Références

1. Nix, W. D. Propriétés mécaniques de couches minces. Metallurgical and Materials Transactions A 20 (11), 2217-2245, 1989.
2. Pharr, G. M. D. L. Callahan, S. D. McAdams, T. Y. Tsui, S. Anders, A. Anders, I. J. W. Ager, I. G. Brown, C. S. Bhatia et S. R. P. Silva. La dureté, le module élastique et la structure des films de carbone très durs produits par le dépôt cathodique-arc avec biais d'impulsion de substrat. Lettres de physique appliquée 68(6), 779-781, 1996.
3. Nix, W. D. Propriétés élastiques et plastiques des couches minces sur les substrats : techniques de nanoindentation. Science et génie des matériaux A 234-236, 37-44, 1997.
4. Bunshah, R. F. Manuel de revêtements durs : Technologies de dépôt, propriétés et Applications. Noyes Publications, 2001
5. BS EN ISO 14577 Parties 1, 2 et 3 : Matériaux métalliques - Test d'indentation instrumentée pour la dureté et les paramètres des matériaux, 2002.
6. Oliver, W.C. et G. M. Pharr. Une technique améliorée pour déterminer la dureté et le module élastique à l'aide d'expériences d'indentation de détection de charge et de déplacement. J. Mater. Rés. 7(6), 1564-1583, 1992.
7. Datcheva, M., Cherneva, S., Stoycheva, M., Iankov, R., et Stoychev, D. (2011). Détermination des caractéristiques des matériaux d'aluminium anodisés au moyen de mesures de nano indentation. Sciences et applications des matériaux, 2(10), 1452.
8. Iankov, R., Cherneva, S., et Stoychev, D. (2008). Recherche des propriétés matérielles des films de cuivre minces par la modélisation finie d'élément du test de micro-indentation. Applied Surface Science, 254(17), 5460-5469.