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NUMERICAL SIMULATION OF VICKERS MICRO-INDENTATION TEST TO ESTIMATE MICRO-HARDNESS

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Abstract: The paper aim is to investigate the experimental micro-hardness process by finite elements method (FEM) and validation of the numerical simulation by geometric evaluation of the imprint's parameters numerically obtained with the experimental results. In the first stage, micro-indentation experiments with a Vickers indenter were performed on two materials, aluminum 6060 and cooper C110. The average length of imprint diagonals and average value of Vickers micro-hardness of the materials were determined. In the second stage, the micro-indentation process for both materials were simulated by finite element method using a 3D model of a Vickers indenter for which the geometric and mechanical characteristics are known. For an accurate simulation the mechanical constants and material behavior in terms of strain –stress curves of the studied materials, has to be determined. Thus, the uniaxial tensile test was performed for both materials and their mechanical properties and the stress – strain curves were measured. The values of Vickers micro-hardness and the geometric parameters of imprints obtained from the finite element simulation were in very good agreement with the results obtained from micro-indentation experiments.

Key words: Vickers micro-hardness, micro-indentation, imprint parameters, numerical simulation.

1. INTRODUCTION

The micro-indentation technique is widely used for determine hardness and basic mechanical properties at nano and micro scale for different materials like metals, ceramics, composites, thin films, second phase particles and magnetic hard disk recording media [1-4]. This technique presents the advantage that the measurements can be performed on small amounts of material, with no restrictions regarding sample cross section, and the tests are realized without permanent destruction of the sample [5].

Micro-indentation consists in pressing against the material's sample with a force by an indenter with known geometry, causing local elastic and plastic deformations, resulting an imprint. The hardness of the sample is calculated taking account the applied force and the diagonals of the imprint optically measured. Most of the researchers have focused their investigations on estimation the elastic modulus

and hardness of the tested sample by using loading and unloading indentation curve ($P - h$) provided by instrumented micro-indentation technique [6-12]. However, indentation curves are not available for traditional hardness equipment, thus occurs the necessity to apply finite element methods (FEM) in order to characterize mechanical properties of the materials.

In the last two decades, FEM technique is highly used for modeling and solving many engineering problems. The advantage of using FEM is that can be performed several simulations in different conditions for the same sample for a better understanding of mechanical phenomena, thus saving time, material and financial resources.

In addition to experimental and analytical studies, have been performed numerical simulations of micro and macro-indentation process to determine the basic mechanical properties of different materials [13-18]. Thus, it was taken into account the influence of geometric modeling shape of indenter on load-

indentation depth curve for elasto-plastic materials, the mechanical and micro-structure behavior of the material, and the distribution of equivalent plastic deformations during nano and micro-indentation process [19-23].

In other studies were exhibited numerical analysis taking into account the influence of the coefficient of friction between the indenter and sample's surface on numerical estimation of elastic modulus and hardness of material [24]. Another aspect analyzed by numerical micro-indentation simulations is the sink-in and pile-up phenomenon, given by plastic deformation of the impression, and their influence in the estimation of Young's modulus and hardness of tested material [25]. Researchers have proposed numerical methods which allow assessment of indentation imprint and to estimate the hardness of bulk materials or metal matrix. They have compared numerical results obtained from FE simulation of nano-indentation process with experimental values in order to validate the FE simulation methods of indentation process [10, 26-29].

In this study, a 3D numerical simulation of experimental micro-indentation test using a Vickers indenter was performed to determine the geometrical parameters of imprint after elastic recovery and to estimate the Vickers micro-hardness of aluminum 6060 and cooper 110 samples. Uniaxial tensile test are made on both samples in order to define stress-strain curve required for numerical simulations. Also, Vickers micro-indentation experiments was made to compare experimental hardness with numerical results and to check the proposed FE method. For both samples, the Vickers micro-hardness, respectively the parameters which define the geometry of imprints determined by numerical simulations were in very good agreement with Vickers micro-hardness and imprints obtained from the micro-indentation experiment.

2. METHODS AND MATERIALS

2.1 Methods

As previously defined, the conventional Vickers hardness is given by the maximum force divided by the four-sided pyramidal contact area, which is evaluated from the diagonals of the residual indentation marks after unloading.

In literature are given several formula to calculate the conventional Vickers hardness (HV), where either the imprint diagonals or projected area are taken into account [30, 31].

The base formula used to calculate the conventional Vickers micro-hardness (HV) is according to ASM Handbook. The HV value is depending on mean diagonals optically measured and the applied force is previously known [32, 33]:

$$HV = \frac{2000F \sin(\alpha/2)}{d^2} = \frac{1854.4F}{d^2} \quad (1)$$

where d is the mean diagonal of the residual impression [μm]; F is the applied force [gf] and α is the angle between opposite faces of the indenter and it is 136° for pyramidal indenter (Vickers).

In micro-indentation process local elastic and plastic deformations occurs when maximum force is applied and thus the residual imprint is generated. The elastic recovery during unloading happens mainly along loading direction and it is negligible in-plane direction (normally to loading direction).

If the recovery in the in-plane direction during unloading is negligible [34, 35], the diagonal evaluated from the contact depth at the corners in the loaded state should be the same as those of the residual imprint. The contact depth at the corner determines conventional Vickers hardness given by equation [11]:

$$HV = \frac{F_{max}}{26.43 \times (h_c)^2} \quad (2)$$

In equation (2) the denominator represents the projected contact area at the maximum indentation depth determined by F_{max} and can be calculated directly from the geometry of the residual imprint. The relationship between the diagonal and contact depth at the corner of the residual indent is given by:

$$h_c = \frac{d}{2\sqrt{2} \tan(\alpha/2)} \quad (3)$$

Equations (1) and (2) are used to determine Vickers micro-hardness when conventional indentation test are performed.

2.2 Materials

The investigated materials are aluminum extrusion alloy (Al 6060 T6) from the series 6 of the alloy and 110 copper known as Electrolytic Tough Pitch copper (ETP) or C11000. Aluminum alloy is widely used for architectural and building products, pipe and tubes, electrical components, profile products, heat sinks etc.

The Al 6060 T6 alloy offers good mechanical strength and good resistance corrosion, and the finished products are obtained with good surface quality. The chemical composition of aluminum alloy is given in standard EN-573-3:1994 and the mechanical properties in EN 755-2:2008 (Vickers hardness $HV = 0.785$ [GPa], elastic modulus, $E = 69$ [GPa]) [36, 37].

Copper 110 (C11000) is the most common copper type on the market and it has the highest electrical conductivity of any metal (except silver). Copper is used as an electrical component in transformers, switch gears and parts that require a high electrical conductivity. Mechanical properties of copper 110 is given in EN 1652 (or ASTM B152/B 152M – 06a): elastic modulus is 117 GPa and Vickers hardness varies between 90 and 117 GPa, depending on thermal treatments applied [38, 39].

The aluminum sample used for experimental measurements were cut from a commercial L shape profile with 1.25 mm wall thickness and the copper sample was cut from a sheet with 0.32 mm thickness. The finished surfaces of the aluminum profile were covered with a thin layer of corrosive protection (thickness of the micron size), which influence the micro-hardness of the material.

To obtain correct values of mechanical properties and micro-hardness by experimental measurements for both samples, it was necessary to (1) carefully polish the samples surface in order to remove the protection layer and (2) carefully remove the impurities of samples surface with dissolvent solution.

3. EXPERIMENTAL MEASUREMENTS

3.1 Micro-indentation test

In order to estimate the Vickers micro-hardness of the aluminum and copper samples by finite elements method (FEM) and to check

the proposed numerical model, it was necessary to determine the hardness of the aluminum Al 6060 T6 and copper samples by micro-indentation experimental tests. The Vickers micro-hardness test were performed according to ASTM E 384-99 and ISO 6057-1 [32, 40].

The micro-indentation tests were performed using a Wilson Hardness Tukon 1202 tester equipped with diamond square base indenter – Vickers indenter, in laboratory conditions at temperature $T = 25 \pm 1$ °C and stable humidity (53%). The Wilson Tukon 1202 is a conventional micro-hardness tester which perform static micro-indentations and the imprint on sample's surface is generated by the vertical motion (downward) of the Vickers indenter given by the action of a selected weight. Then, the Vickers micro-hardness value is automatic computed by WinControl software using the diagonals length of imprint optically measured.

Thus, on polished surface of the aluminum sample were made 15 indentations with a maximum load of 9.81 N (1000 gf), in order to check the repeatability of the measurements. The dwell time of the applied load was automated set by software at 10 s. The same procedure of experimental micro-indentation test it was achieved for copper sample as well. For example, one imprint is given in figure 1 for each sample.

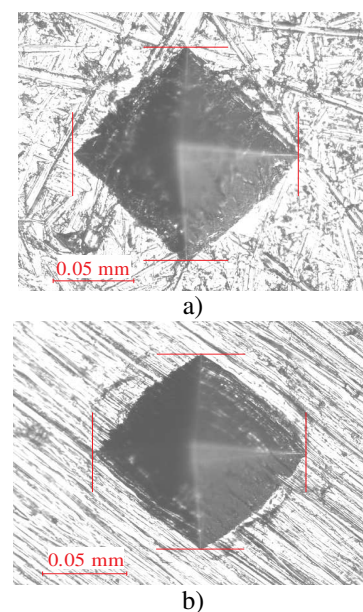


Fig. 1. Imprint of Al 6060 T6 (a) and copper (b) obtained by Vickers micro-indentation

The average value of both Vickers micro-hardness (HV_{exp}) and imprint diagonal (d_{exp}) of Al 6060 T6 and copper samples were obtained (table 1).

2.2 Tensile test

To perform the numerical simulations, the mechanical properties of aluminum Al 6060 T6 and copper 110 samples have been determined by tensile tests, according to ISO 6892-1 [41].

The mechanical properties and the stress-strain curve ($\sigma-\epsilon$) of the materials were

determined using Instron 3366 Dual Column Universal Testing Machine. For measurements of elastic strains (ϵ) with high accuracy a uniaxial extensometer was used.

The experimental stress-strain curve for Al 6060 and copper 110 are represented in figure 2 and the obtained mechanical properties are given in table 2

Table 1

Results obtained by experimental micro-indentation.

| Sample's material | Parameters of imprint | | Micro-hardness | |
|-------------------|-----------------------|----------------|----------------|------------------|
| | h_{exp} [mm] | d_{exp} [mm] | HV_{exp} | HV_{exp} [GPa] |
| Al 6060 T6 | 0.0292 | 0.145 | 88.03 | 0.864 |
| Cooper | 0.0274 | 0.136 | 99.08 | 0.97 |

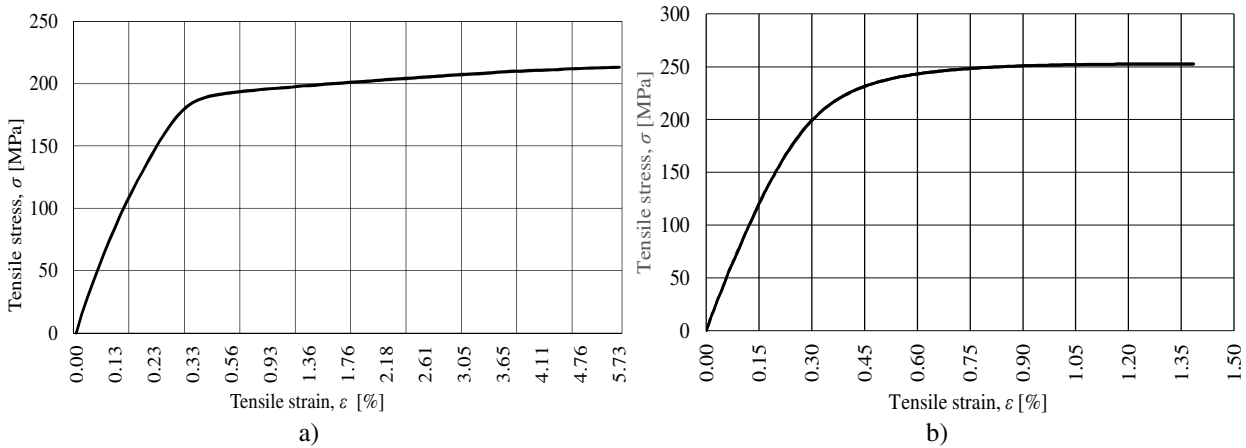


Fig. 2. Stress-strain curve obtained by tensile test of Al 6060 T6 (a) and Copper (b)

Table 2

Mechanical properties of Al 6060 T6 from tensile test.

| Sample's material | E modulus, $E_{tensile}$ [GPa] | Sample's thickness, t [mm] | Load at break [kN] | Yield stress, $R_{p0.2}$ [MPa] | Tensile strength, R_m [MPa] |
|-------------------|--------------------------------|------------------------------|--------------------|--------------------------------|-------------------------------|
| Al 6060 T6 | 64.010 | 1.25 | 3.328 | 191.78 | 213.01 |
| Copper 110 | 84.907 | 0.32 | 0.451 | 252.74 | 252.68 |

4. NUMERICAL SIMULATION

A three-dimensional model and ANSYS Workbench software is adopted to simulate the Vickers micro-indentation process in order to determine the geometrical parameters of the imprint, respectively the micro-hardness of the samples. The boundary condition was modeled as a fixed support at the lower surface of the

samples to prevent movements in any directions of the lower nodes of the modeled sample and the indenter was defined as traction-free on vertical direction. The size of the indented sample was taken to be large enough compared with the maximum contact radius, thus the boundary conditions have no effect on the computational results.

The contact conditions were imposed between inclined surfaces of the indenter and upper horizontal surface of the sample, without taking into account the friction between surfaces [25, 42]. The mesh of the numerical model were automatically generated with 3D tetrahedral finite elements (270.733 nodes and 191.644 finite elements) having a maximum element size of 0.11 mm. In order to improve the contact conditions a very fine mesh was defined in the contact area, mesh size increasing to larger elements as they get further away from indenter tip.

For this study, the material of the samples was considered isotropic and homogenous. The experimental tensile stress-strain curve has been employed to characterize the elasto-plastic behavior of the material and the large deformation formulation is included. The Poisson's ratio value of $\nu = 0.33$ for the aluminum alloy and copper was taken from the literature [18, 43, 44].

It was demonstrated that the tip imperfection of the indenter does not have a major influence on hardness [45-47]. In this context, for numerical simulations the Vickers indenter was modeled in the hypothesis of an ideal indenter and no corrections were made at the tip. The Vickers indenter was modeled as a 3D rigid body with the angle between its opposite faces $\alpha = 136^\circ$. The mechanical properties of the indenter were defined as the following: $E_i = 1140$ GPa, $\nu_i = 0.07$ [9].

The loading force was applied on the upper horizontal surface of the indenter, corresponding with the external applied force of the experimental Vickers micro-indentation.

The three distinct phases in the micro-indentation process, loading – dwell time – unloading, realized by the movements in the vertical direction of the indenter, were numerically simulated through defining the force according to the load steps shown in figure 3.

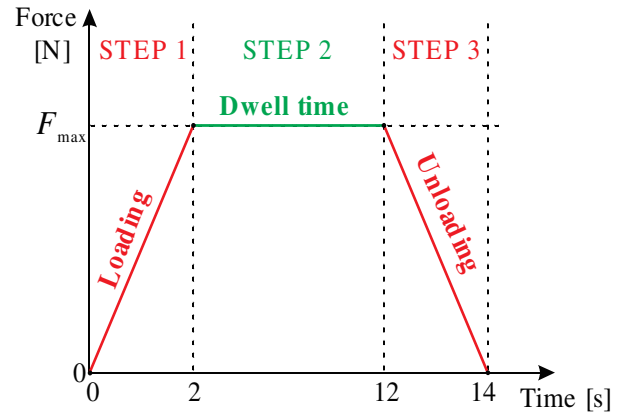


Fig. 3. The force-time history used in numerical simulations of indentation test

Thus, in the first step the force increases from 0 to F_{max} (9.81 N), generating the sample penetration (elastic-plastic deformations), the second step corresponds to the dwell time where the force is maintained constant resulting the maximum height of the imprint (h_{max}), respectively the imprint mark, and in the last step the force decreases to zero, corresponding to upward movement of the indenter and the elastic recovery of the material, resulting the final height (h_f) of imprint.

Also, it is specified that the computation time of the numerical micro-indentation, for each step described above, has been set as the experimental micro-indentation test: loading and unloading steps at 2 s and dwell time to 10 s.

From numerical imprint, were taken into consideration the parameters that define the height of the imprints (figure 4), for both samples, as follows: the maximum height (h_{max}) when is applied the maximum force and the imprint is generated, and final height (h_f) after unloading and the elastic recovery in loading direction of the residual imprint is produced.

Analyzing the simulation results shown in figure 5, it can be observed that the plastic deformations in loading direction are highlighted by the appearance of pile-up effect around the imprint by a residual area ($\Delta A+$), which influence the shape and size of the imprint [48, 49]. The pile-up effect can be identified in the numerical diagram from figure 5 by negative values, calculated from the undeformed surface of the sample [50].

Thus, the height of the numerical imprint (h_{num}) was calculated taking into account the final height (h_f) of imprint (after loading) and the

height of pile-up ($h_{pile-up}$), obtained from FE simulations:

$$h_{num} = h_f + h_{pile-up} \quad (4)$$

The numerical diagonals of imprint (diagonal 1 and 2 shown in figure 5), for

aluminum alloy and copper samples, were computed by geometrical relations knowing (h_f) and the angle (α) between opposite surfaces of the Vickers indenter.

The diagonal (d_{num}) given in table 3 was calculated as mean value of diagonals 1 and 2.

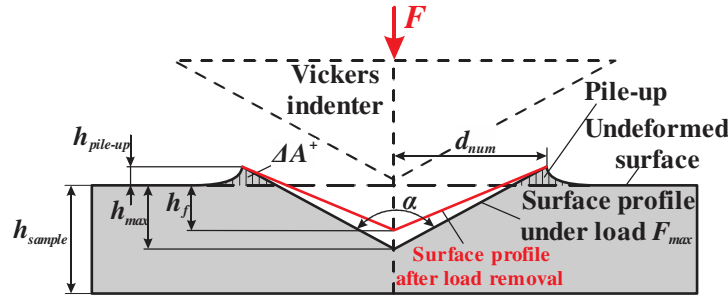


Fig. 4. Parameters defining the geometry of imprint

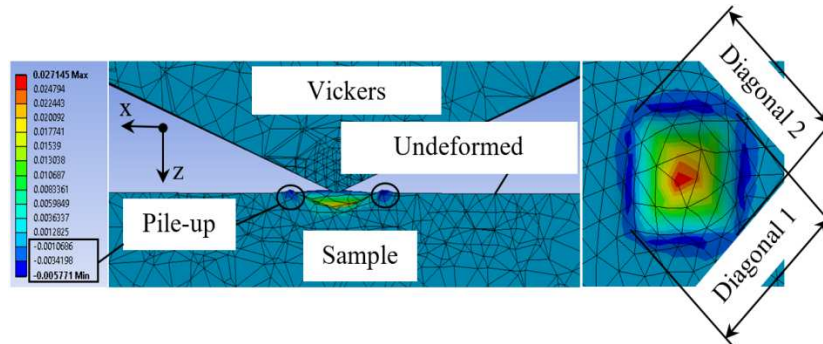


Fig. 5. Imprint obtained from numerical 3D simulation of the micro-indentation

Table 3

Numerical values for geometrical parameters of imprint and micro-hardness calculated with equation (2)

| Numerical results | | | | | | | | |
|-------------------|-----------------------|---------------|-----------------------|-------------------|-------------------|----------------|------------|---------------------|
| Sample's material | Parameters of imprint | | | | | Micro-hardness | | |
| | h_{max} [mm] | h_f [mm] | $h_{pile-up}$ [mm] | h_{num} [mm] | d_{num} [mm] | h_c [mm] | HV_{num} | HV_{num} [GPa] |
| Al 6060 T6 | 0.0295 | 0.0271 | 0.00203 | 0.02913 | 0.14418 | 0.02059 | 89.2 | 0.875 |
| Copper | 0.0268 | 0.0247 | 0.00168 | 0.02642 | 0.13075 | 0.01876 | 108.48 | 1.064 |

The Vickers micro-hardness given in table 3 was computed with equation (2) where h_c (equation 3) was calculated according to the average value of the diagonal (d_{num}) determined by numerical simulations. Also, the Vickers micro-hardness obtained for both specimens were checked with the equation (1) defined by the standard.

3. RESULTS AND DISCUSSION

Numerical simulation of indentations allows determination of geometrical parameters of imprints and calculation of sample's Vickers

micro-hardness of 0.875 GPa (for aluminum) and, respectively 1.064 GPa (for copper).

It can be observed that the values of Vickers micro-hardness of the tested materials obtained by experimental micro-indentations of 0.864 GPa for aluminum and 0.97 for copper are in good agreement with the numerical micro-hardness, relative deviations being under 10% (see table 4).

Based on experimental Vickers micro-hardness measurements the average length of diagonals of imprint for both materials have also a closed values to those determined by Vickers micro-hardness (table 4).

In this study the validation of inverse method for extracting Vickers micro-hardness from non-instrumented indentation tests has been proposed in terms of dimensional analysis and finite element simulations. Both the uniaxial

tensile test and the micro-indentation experiment were performed on the same materials in order to compare and validate the values of micro-hardness obtained from numerical simulations.

Table 4

Numerical and experimental values of impression parameters and micro-hardness

| Sample's material | Numerical micro-indentation | | | Experimental micro-indentation | | | Relative deviation |
|-------------------|-----------------------------|----------------|------------------|--------------------------------|----------------|------------------|--------------------|
| | h_{num} [mm] | d_{num} [mm] | HV_{num} [GPa] | h_{exp} [mm] | d_{exp} [mm] | HV_{exp} [GPa] | HV [%] |
| Al 6060 T6 | 0.02913 | 0.14418 | 0.875 | 0.0292 | 0.145 | 0.864 | 1.27 |
| Copper | 0.02642 | 0.13075 | 1.064 | 0.0274 | 0.136 | 0.97 | 9.69 |

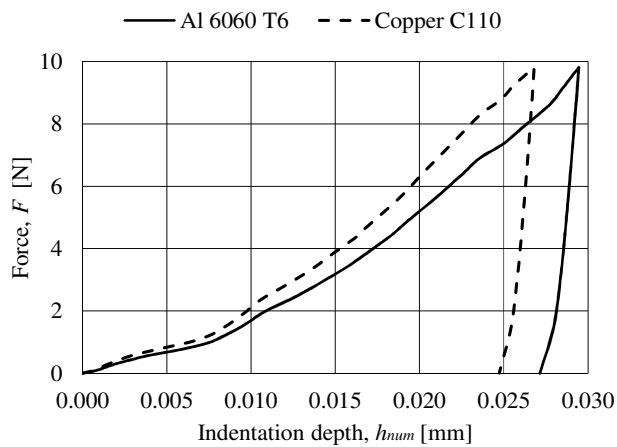


Fig. 6. Force vs. indentation depth curve obtained by numerical simulation

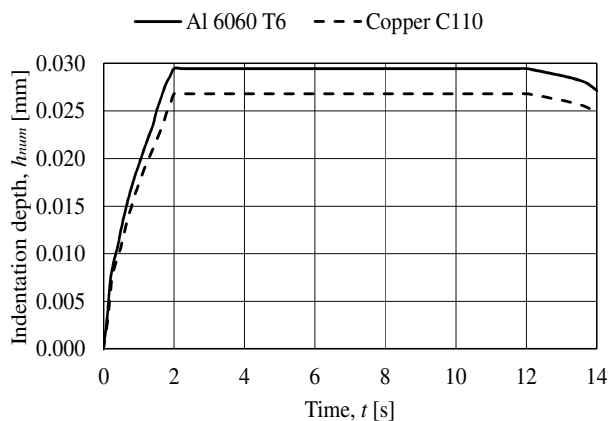


Fig. 7. Time vs. indentation depth during micro-indentation simulations

The experimental micro-indentation tests have been validated by 3D finite element simulation. The result was the geometry of the imprint (diagonals size, maximum height during loading, final height after unloading and the pile-

up effect around imprint) of the Al 6060 T6 and copper 110 samples and its Vickers micro-hardness, respectively.

Figure 6 gives the variation of indentation depth during load steps defined in the numerical simulations, for both studied materials, and represents a typical force–displacement curve for an instrumented indentation test. The plot corresponds to the test segments identified in figure 3, data in step 2 is plotted, but is not visible as a function of displacement.

The force versus indentation depth unloading curve helps to estimate the contact stiffness and the contact area, in order to be able to evaluate the elastic modulus and micro-hardness by applying the Oliver and Pharr approach.

Figure 7 represent an indentation depth – time history curve, obtained also by numerical simulations for aluminum alloy and copper samples.

From these figures it can be observed that, the material of the samples exhibits a nonlinear behavior during a loading-unloading micro-indentation cycle, as expected from theoretical curve presented in [6], and respectively highlights the elastic recovery in loading direction of the material after indentation is performed.

Good agreement was found between the values of Vickers micro-hardness obtained from the two methods (experimental and numerical simulations) and the results validation being achieved by good agreement between the geometric parameters of imprint obtained from

the finite element simulation and those obtained from the micro-indentation experiment.

Moreover, the measured Vickers micro-hardness, and respectively the numerical micro-hardness are close to those reported in literature [11, 13, 37, 39].

The finite elements technique is a useful tool to provide information that would have been difficult to obtain by experimental tests and also offer a better understanding of the mechanical behavior of the sample during micro-indentation measurements.

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SIMULAREA NUMERICĂ A PROCESULUI DE MICROINDENTARE VICKERS PENTRU ESTIMAREA MICRODURITĂȚII

Rezumat: Scopul lucrării este de a investiga procesul experimental de microduritate prin metoda elementelor finite (MEF) și validarea simulării numerice prin evaluarea geometrică a parametrilor amprentei obținuți numeric cu amprenta experimentală obținută prin microindentare. În prima etapă s-au efectuat măsurători de microindentare cu indenter Vickers pe două materiale, aluminiu 6060 și cupru C110. S-au determinat lungimea medie a diagonalelor amprentei și valoarea medie a microdurității Vickers a materialelor. În cea de-a doua etapă, s-a efectuat simularea numerică a procesului de microindentare cu MEF utilizând un indenter 3D Vickers pentru care sunt cunoscute caracteristicile geometrice și mecanice. Pentru simularea fidelă a comportamentului mecanic al materialelor studiate cu MEF este necesar să se cunoască în prealabil curba de tensiuni - deformații specifice ($\sigma - \epsilon$). Astfel, s-a efectuat testul de tracțiune uniaxială pentru determinarea curbei ($\sigma - \epsilon$) și a proprietăților mecanice pentru cele două materiale. Valorile microdurității Vickers și parametrii geometrici ai amprentelor obținute prin simularea cu MEF au fost în concordanță foarte bună cu rezultatele obținute experimental, prin microindentare Vickers.

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