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COMPARISON OF MECHANICAL PROPERTIES OF SURFACE LAYERS WITH USE OF NANOINDENTATION AND MICROINDENTATION TESTS

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Preliminary Note – Prethodno priopćenje

The objective of the paper is a mutual comparison of different methods for evaluation of mechanical properties of surface layers. Mechanical properties were tested with the use of nanoindentation and microindentation tests. Different loads and constant deformation speed were used in both cases. For the evaluation of mechanical properties, the AISI 304 type Chromium-Nickel steel commonly used in mechanical engineering industry was tested. Knowledge of relations and differences between nano and micromechanical properties is necessary for understanding of mechanical processes continuously occurring in surface layers during cutting processes.

Key words: nanohardness, microhardnesss, abrasive water jet, Chromium-Nickel steel

Usporedba mehaničkih svojstava površinskih slojeva pomoću testova nano i mikro utiskivanja. Cilj rada je međusobna usporedba različitih metoda za ocjenu mehaničkih svojstava površinskih slojeva. Mehanička svojstva su ispitivana pomoću testova nano i mikro utiskivanja. U oba slučaja su korištena različita opterećenja i konstantna brzina deformacije. Za ocjenjivanje mehaničkih svojstava je korišten krom nikl čelik (AISI 304 tip) koji se vrlo često primjenjuje u strojarstvu. Poznavanje međusobnih odnosa i razlika između nano i mikro mehaničkih svojstava je važno za razumijevanje procesa koji se događaju na površinskim slojevima tijekom procesa rezanja materijala.

Ključne riječi: nanotvrdoća, mikrotvrdoća, rezanje vodenim mlazom, krom-nikl čelik

INTRODUCTION

With development of modern measurement methods [1-5], there is a growing demand for research on mechanical and tribological properties of surface layers of materials.

The trend is given by growing requirements for quality in engineering applications (final quality of prepared surfaces, tribology, contact loading, wear resistance, corrosion resistance, etc.). Nanoindentation and microindentation tests represent an important tool for evaluation of mechanical properties of surface layers. These measurements techniques are based on the principle of immediate load recording during the penetration of an indentation tip into a material surface (Figure 1). Based on the known geometry of indentation tip, it

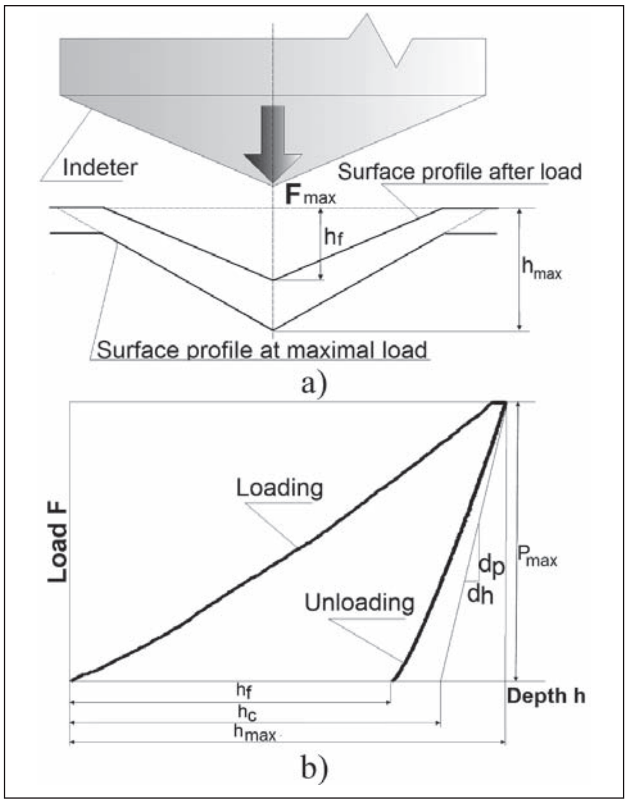

Figure 1 Principle of indentation test a) penetration of indentation tip into material surface, b) courses of loading and unloading curves

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is possible to determine the elasticity modulus and hardness of surface layers.

Berkovich and Vickers indenters are commonly used indenters for indentation experiments. The Berkovich indenter is a three-sided pyramid with the same depth ratio as the four-sided Vickers pyramid. Typical geometry of both indenters is shown in Figure 2. The geometric relationship between the indenters is given in Table 1.

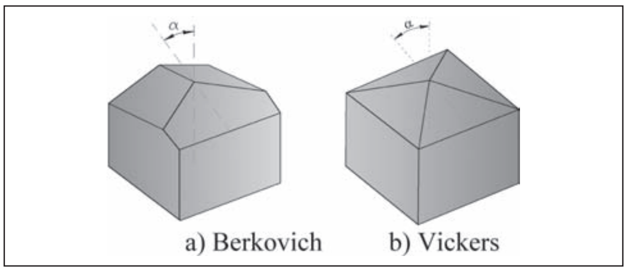

Figure 2 Geometry of indenters

Table 1 **Geometric relationship of indentors**

Indenter type	Projected area	Angle
Berkovich	$A= 24{,}56.h_c^2$	65,3°
Vickers	$A= 24{,}504.h_c^2$	68°

Oliver and Pharr's metodology was used for evaluation of indentation hardness and elasticity. The hardness can be calculated using following formula for a Berkovich indenter [1]:

$$H_{INT} = \frac{P_{\max}}{A(h_c)}, \tag{1}$$

where P_{max} is the maximum load, A is the projected indentation area, h_c represents the contact depth which is determined according to the Formula

$$h_c = h_{\max} - 0{,}75 \times \frac{P_{\max}}{S}, \tag{2}$$

where h_{max} is the maximum indentation depth and, S is the contact stiffness. The elastic modulus E of a material is calculated from E_r using the reduced modulus of an indenter according to the formulas 3 and 4.

$$\frac{1}{E_r} = \frac{1-v^2}{E^2} \cdot \frac{1-v_i^2}{E_i^2} \tag{3}$$

$$E_r = \frac{\sqrt{\pi}}{2\beta} \cdot \frac{S}{\sqrt{A}} \tag{4}$$

where v is the Poisson's ratio for the test material, and E_i and v_i are the elastic moduli and the Poisson's ratio of the indenter [1,3] .

CURRENT STATE OF PROBLEM

The mechanical preparation of surfaces created by conventional methods of cutting is always accompanied by heat load, which can change the mechanical properties in surface layers. The subject of our research was a comparison of mechanical properties of surface layers by nanoindentation a microindentation tests. We try to use abrasive water jet technology for primary cutting of samples because this technology represents cold, precise and computer-controlled shape cutting without any thermal strain. Minimal heat loading of material to be cut is the main advantage of this technology. Surface of samples prepared by AWJT was subsequently grinded and polished using abrasive paper and diamond suspension. The measurement accuracy of the mechanical properties of surface layers usually depends on the quality of surface preparation and wear of an indentation tip [1-5].

EXPERIMENTS

AISI 304 stainless steel was chosen as an initial material for the realization of experiments. This material is commonly used in various fields of industry and it is characterized by medium strength, good corrosion resistance, good maintenance and relatively low cost. The The chemical composition of the AISI 304 is given in Table 2.

Table 2 **Chemical composition of AISI 304 alloy, wt. / %**

Element	C	Mn	Si	P	S	Cr	Ni
AISI 304	0,08	2	0,75	0,04	0,03	18,0 20,0	8,00 11,00

An AISI sample with a square cross-section of 10 x 10 mm and height of 5 mm was made by abrasive waterjet cutting technology with the use of a PTV Ltd. company device (Figure 3). After cutting, the sample was grinded and polished using the Struers Tegra Pol 35 device (Figure 4). Sandpapers with grain sizes of 320, 800, 1 000 and 1 200 µm were used for grinding. The pressure force was set to 10N and rotation speed of 200 min^{-1} was used. Subsenquently the sample was polished using polishing diamond suspense with the grain size of 3µm and 1µm.

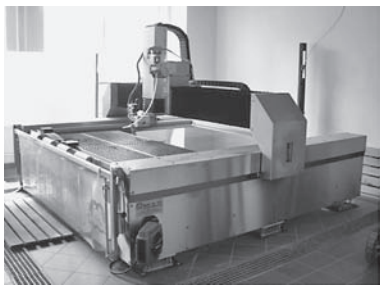

Figure 3 PTV CNC WJ2020 device for sample cutting

Figure 4 Struers Tegra Pol 35 device for sample grinding and polishing

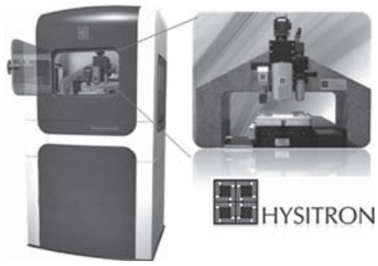

Figure 5 Hysitron Triboindenter TI 950 device used for measurements of nanomechanical properties

Figure 6 CSM Microhardness tester device used for measurements of micromechanical properties

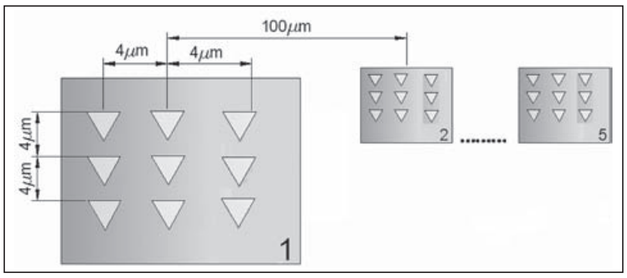

Figure 7 Schematic plan of micro- and nanoindentation on surface of tested sample

Indentation tests were performed on the Hysitron Triboindentor TI 950 (Figure 5) and on the CSM microhardness tester instruments (Figure 6).

The nanoindentation tests were realized with a Berkovich tip and microindentation tests with a Vickers tip. The nanoindentation testing was performed with different forces of 1 000, 1 250, 1 500, 1 750 and 2 000 µN. Forces of 1 000, 1 250, 1 500, 1 750 and 2 000 mN were used in microindentation tests. Velocities of indenters penetration into the material surfaces were 400µN·min^{-1} and 40 µN·min^{-1}. The total number of 45 indents in regular distances of 4 µm were done in 5 areas of testing in 100 µm distances. A schematic plan of indentation is presented in Figure 7.

RESULTS

Calculations of the hardness and elasticity modulus were made using formulas (1-4) based on performed indentation tests, measured data and results of calibration. The analyses were made automatically in user's softwares which are parts of measurement devices. The calculation was carried out for selected indents, which were not influenced significantly by the quality of sur-

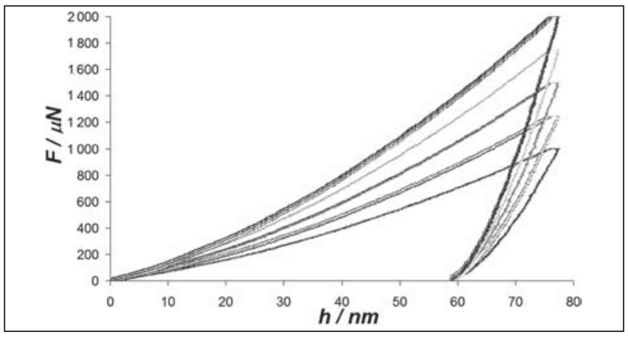

Figure 8 Courses of nanoindentation curves

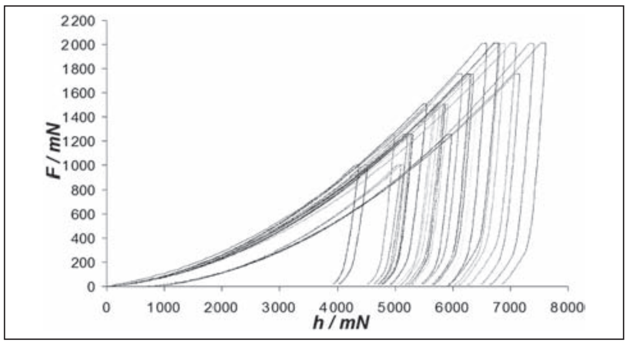

Figure 9 Courses of microindentation curves

face preparation. The resulting indentation curves are given in Figure 8 and Figure 9.

Good equality between loading and unloading curves by the same forces is illustrated in Figure 8 was achieved. Analysis of all forces was made according to the matrix consisting of 9 indentation imprints. The indentation depth differs from 58 to 60 nm. The courses of microindentation curves in Figure 9 show significant differences. They were caused by plastic deformation and size of applied forces (they were 1 000 x higher in comparison with nanoindentation tests). Possible reasons of these differences are various mechanical properties occuring in the middle and on the border of a grain with metallic structure. The indentation depth ranges from about 4 000 to 7 000 nm. Figure 10 illustrates the scatter plot relating elasticity modulus and hardness from both methods of measurements. Similar dispersions of the elasticity modulus values are shown in the graph. The final value varies statistically from 165 to 175 GPa.

Figure 11 represents the comparison of mechanical properties for selected loading forces. As can be seen in

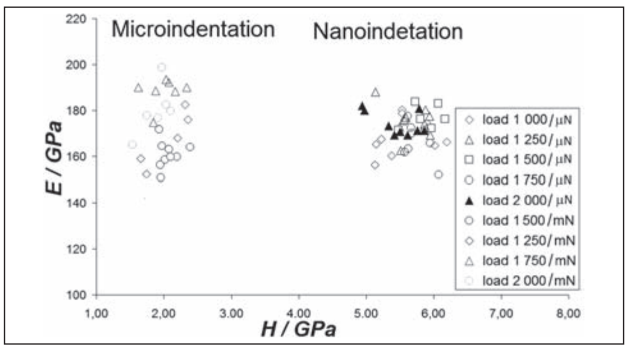

Figure 10. Ratio between elasticity modulus and hardness

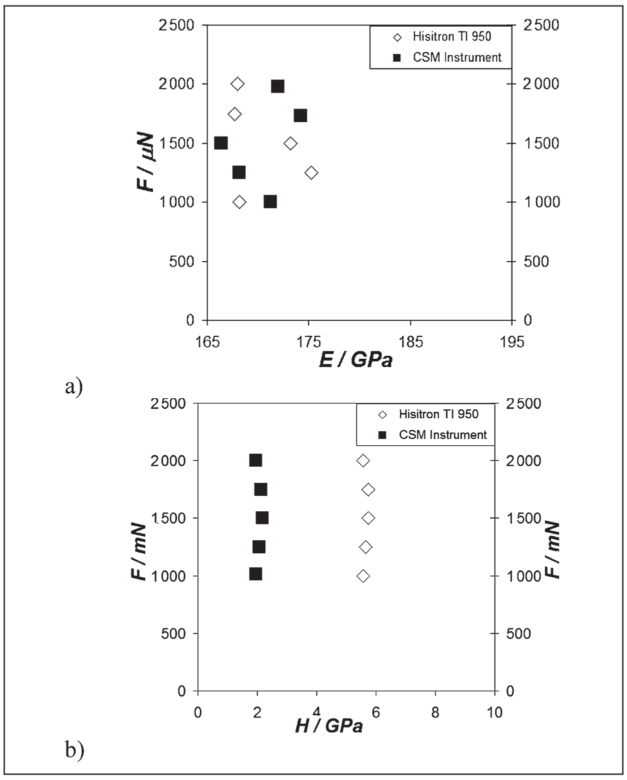

Figure 11 Comparison of mechanical properties for selected forces a) elasticity modulus b) hardness

Table 3 **Results of mechanical properties**

F/µN	Nano-hardness	SD	F/mN	Micro-hardness	SD
1 000	5,55	0,13	1 000	2,10	0,20
1 250	5,65	0,08	1 250	2,07	0,07
1 500	5,74	0,24	1 500	2,17	0,16
1 750	5,73	0,19	1 750	2,12	0,16
2 000	5,56	0,16	2 000	1,95	028
F/µN	Elasticity modulus	SD	F/mN	Elasticity modulus	SD
1 000	168,17	0,27	1 000	171,26	0,35
1 250	175,25	0,10	1 250	168,12	0,24
1 500	173,22	0,22	1 500	166,36	0,17
1 750	167,73	0,15	1 750	173,82	0,37
2 000	167,95	0,31	2 000	172,18	0,19

the graphs, the value of applied loading force does not influence final values of elasticity modulus and hardness.

A very interesting fact was the presence of two ranges of the hardness value in areas of about 2 GPa and 6 GPa, respectively. Lower hardness values were obtained with the use of microindentation tests (see Figure 11 b). This result can be influenced by higher mechanical deformation in nanolayer occurring during interaction of abrasive particles with the basic plane. An indentation tip penetrates during the microindentation deeper into the surface layer witch is not influenced by mechanical load as the nanolayer. All results obtained from both measurements are summarized in Table 3.

CONCLUSION

The paper presents results acquired during the evaluation of mechanical properties of surface layers using the AISI 304 type Chromium-Nickel alloy in the nanoindentation and microindentation tests. To eliminate heat load during sample preparation, the abrasive water jet technology was used to prepare sample. Subsequently, the sample was grinded and polished. To evaluate the properties, different forces were used as described in results. The nanonindentation measurement was accompanied by creation of elastic deformation, and microindentation measurement was accompanied by creation of plastic deformations. The results from both measurements are given in the Table 3. Comparing the elasticity modulus, good equality of both measurements was achieved. According to the Figure 11 b, the values of hardness different. The hardness in microindentation tests is more than 3x lower in comparison with nanohardness. It proves higher mechanical hardness in nanolayers. One of possible applications of these measurement methods is the evaluation of mechanical properties of steel layers during interaction with pulsed water jet. It would be possible to influence (control) the quality of mechanical properties in surface layers on the basis of known technological parameters of pulsed water jet.

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REFERENCES

[1] Oliver, W.C., Pharr, G.M., An Improved Technique for Determining Hardness and Elastic Modulus Using Load and Displacement Sensing Indentation Experiments, Journal of Materials Research 7 (1992), 1564-1583.

[2] Zelenak, M., Valicek, J.,Brumek, J., et al. Measurement and analysis of the hardness of aluminium surface layers by the nanoindentation and scratch tests. Chemicke listy.105 (2011) 17, 680-683.

[3] Hajkova P., Louda P., Spatenka P.Comparison of evaluating methods of thin films nanohardnees; Czechoslovak journals of physics 56 (2006), 1162-1168.

[4] Hlavacek P., Valicek J.,Brumek J.et al. Use of the Indentation tests for the evaluating of machinability of materials during abrasive water jet cutting. Chemicke listy, 105 (2011), 735–737.

[5] Foldyna, J. Sitek, L. Ščučka, J. Martinec, P. Valíček, J. Paleníková, K. Effects of pulsating water jet impact on aluminium surface. Journal of material processing technology 209 (2009), 20, 617-618.

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