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

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, microhardness, abrasive water jet, Chromium-Nickel steel

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](http://hdl.handle.net/10084/90514)

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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.

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

\[ H_{\text{INT}} = \frac{P_{\text{max}}}{A(h_c)} \]  

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

\[ h_c = h_{\text{max}} - 0.75 \times \frac{P_{\text{max}}}{S} \]  

where \( h_{\text{max}} \) 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.

\[ E_r = \frac{1}{\frac{1 - \nu^2}{E_i^2} - \frac{1 - \nu_i^2}{E_i^2}} \]  
\[ E_r = \frac{\sqrt{\nu_i}}{2\nu} \times \frac{S}{\sqrt{A}} \]

where \( \nu \) is the Poisson’s ratio for the test material, and \( E_i \) and \( \nu_i \) 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 and 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 ground 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 chemical composition of the AISI 304 is given in Table 2.

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 gridding. The pressure force was set to 10N and rotation speed of 200 min⁻¹ was used. Subsequently the sample was polished using polishing diamond suspension with the grain size of 3µm and 1µm.
Indentation tests were performed on the Hysitron Triboindenter 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 400 µN 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 surface 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 occurring 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...
influence final values of elasticity modulus and hardness. 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


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

Table 3 Results of mechanical properties

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<tr>
<th>F/N</th>
<th>Nano-hardness SD</th>
<th>F/N</th>
<th>Micro-hardness SD</th>
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<table>
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<th>F/N</th>
<th>Elasticity modulus SD</th>
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