EXPERIMENTAL RESEARCH AND EVALUATION OF MECHANICAL PROPERTIES OF MICROSTRUCTURAL COMPONENTS OF HIGH STRENGHT STEELS BY QUASISTATIC NANOINDENTATION

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Abstract:

The article solves the issue of evaluating the mechanical properties of the microstructural components of the C120U, 90MnCrV8 and X82WCrV9-5-2 tool steels. The reason for the choice of steels and their testing was the fact that on these tool steels are high demands and their properties using in industry, such as high strength, toughness, wear resistance, temperature stability, etc. Experimental measurements were performed on the device a Hysitron TI 950 Triboindenter with Triboscan evaluation software, which is part of CEDITEK Laboratories at Faculty of special technology. The Berkovich type was used as a test tip. Chapter 1 describes the research of foreign authors who in their studies dealt with the mechanical properties of selected types of high-strength steels. In chapter 2, the mechanical and chemical properties of the investigated materials, microstructural analysis and used methods are clearly presented. Based on the analysis, the methodologies of the performed experiments are described in sub-chapter 2.4. Achieved results of own experiments are presented in chapter 3. Realized experimental measurements and evaluation of measured data on selected locations of the investigated areas of tested steels using quasistatic nanoindentation are described in chapters 3 and 4.

1 Introduction

1.1 Manuscript preparation

Quasistatic nanoindentation is a contact testing method which consists in mechanical contact of the test tip with the investigated material and where the output measured quantities are reduced Young's modulus of elasticity E_r [GPa] and nanohardness H [GPa]. Their use is in areas where these quantities cannot be measured by conventional methods of measuring of mechanical properties. Conventional methods of hardness measuring such as Brinell, Knoop, Vickers and Rockwell differ in the geometry of the test specimens and indentation method of one material into the another. Quasistatic nanoindentation is a testing method for measuring the mechanical properties of materials, which differs from the basic methods in that nanometers (10^{-9} m) are used as a measure of penetration depth, in contrast to conventional methods where the units are micrometers (10^{-6} m) or millimeters (10^{-3} m) [1, 7]. In addition, used to the displacement scale is the characteristic distinguishing feature of most nanoindentation tests is the indirect measurement of the contact area and that is the area of contact between the testing tip and the specimen. In conventional tests for measuring the hardness of materials, the contact area is calculated from direct measurements of the dimensions of the residual impression left on the sample surface after removal of the load [1, 5|. When tested by the quasistatic nanoindentation method, the size of residual impression in the order of micrometers is too small to be measured

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directly. Therefore, it is common to determine the contact area by measuring the penetration depth of the test tip into the surface of the specimen [1]. In addition to the measurement of nanohardness H [GPa] and reduced Young's modulus of elasticity E_r [GPa] was by the quasistatic nanoindentation were performed by authors [8, 9]. Thus, these nanoindentation techniques can also be used to calculate elastic modulus, deformation-curing exponent, fracture toughness (e.g., for brittle materials) and viscoelastic properties. This type of data is obtained when the test tip is brought into contact with the flat surface of the sample with an ever-increasing load. The load and indentation depth are recorded with each load increment, which ultimately provides a measure of modulus and hardness as a function of depth below the surface [1]. Nanoindentation tests are commonly used to measure the hardness of materials, but diamond test tips such as Vickers, Berkovich and Knoop can also be used to investigate other mechanical properties of solids such as strength, fracture toughness and tensile / compressive residual stresses [1]. The authors [4] performed quasistatic nanoindentation tests of specimens at room temperature on a NanoTest device supplied by company Micro Materials Ltd., Wrexham, UK, using a three-sided Berkovich diamond test tip with a nominal angle of 120° and a radius of r = 100 nm [4]. All nanoindentation tests were performed at the same maximum load (F = 500 mN), with load speeds of 50, 25, 16.67, 12.5, 10, 5 and 1 mN.s^{-1} The test tip was then left on endurance at maximum load during t = 5 s. Then followed by unloading at a speed of 50 mN.s^{-1} for all tests. At least 10 indentation points were performed and for each load separately. The measurement results were subsequently averaged [4]. All hardness values measured during the nanoindentation process within the study of the authors [4] are higher than the hardness values on the tested steel H13 [6, 13]. Steel was produced in the basic state, but without the use of SLM and obtained from the results of the Mencin process [3, 4], The results of this study agree with the results of previous experimental reports on nanoindentation tests of H13 steel [6, 13]. The authors of the research [2] also performed nanoindentation with samples at room temperature in order to evaluate the mechanical properties of SLM H13 steel. The testing tip was also used by Micro Materials Ltd., Wrexham, UK and it was a three-sided diamond Berkovich testing tip. The maximum load was chosen with sufficient size to ensure the presence of imprints in all phases of tested sample. Specifically, all nanoindentation tests were performed at the same maximum load (F = 500 mN) with the achieved load rate heights of 50, 25, 16.67, 12.5, 10.5 and 1 mN.s^{-1}. The test tip was then left at maximum load t = 5 seconds, followed by unloading at speed 50 mN.s^{-1} for all tests. There were at least ten indents for each load and the results were then averaged [2]. A representative load from the quasistatic nanoindentation test is defined as the instantaneous load (P) divided by the projected contact surface (A_c), which is also the definition of the indentation hardness (H) during measurement [2]. In addition, during nanoindentation tests at a constant load speed, the degree of deformation is a non-linear function of time, which can be estimated from depth and time data obtained for a given range of indentation depths [2].

2 Experimental investigation

2.1 Basic information

In the process of realized experimental research were used a total of three types of tool steels. These were the tool steels of C120U, 90MnCrV8 and X82WCrV9-5-2 types according to the ISO standard. These types are tool steels used in practice for the production of various tools, whether hand tools or cutting tools for machining. These steels are subject to high requirements, such as high strength, toughness, wear resistance, temperature stability and so on. It is the optimal combination of these above-mentioned mechanical properties that these selected tool steels acquire with a suitable chemical composition and heat treatment. The structure of these tool steels is usually formed by martensite, or a mixture of martensite and bainite, residual austenite and carbides after hardening [10, 12]. The machinability of these selected tool steels is improved by soft annealing [11]. The microstructure after soft annealing is subsequently formed by ferrite and carbides. The hardness of tool steels after hardening is usually given in HRC units (Rockwell hardness) and after soft annealing according to HB (Brinell hardness).

2.2 Mechanical and chemical properties

The C120U steel is an alloy tool steel with a higher carbon content of 1.1%. This steel was chosen as the first material tested to achieve a high hardness after hardening (min. 64 HRC) and which is tempered to 60±2 HRC. Steel is used for cutting, shearing, and forming tools, hand tools and gauges. The C120U steel has good core toughness, insensitivity to hardening cracks, more difficult hot formability, and good machinability in the annealed state. The 90MnCrV8 steel belongs to alloy tool steels according to the ISO standard. The most important additive elements of these steels are Cr, Mo, V, W. They are carbide-forming elements that increase the hardness and stability of the carbide phase and reduce the decrease in hardness during tempering. They also significantly increase wear resistance. As the additive elements increase hardenability, it is also possible to produce larger tools from these steels. The addition of Ni increases the toughness of this steel. According to the ISO standard, X82WCrV9-5-2 steel is classified as high-speed steel or HSS steel (High Speed Steel). The quality of this steel is determined by the presence of hard and abrasion-resistant carbides such as Cr, V, W. Their quantity in this type of steel is usually in the range of 15 to 22%. Into more powerful species is added Co (up to 10%), which increases the strength of the solid solution at higher temperatures. This type of tested steel is characterized by resistance against the decline hardness up to temperatures around 550 ºC. It is a high alloy ledeburitic steel with a carbon content of over 0.70%. The basic alloying element in HSS steels is W, which can be partially replaced by half the amount of Mo. The chemical composition and mechanical properties of the tested steel steels are shown in Tables 1, 2 and 3.

2.3 Mictrostructural analysis

The C120U is a supereutectoid tool steel with a cementitic-pearlitic structure (see in Fig. 1a). The steel is in the state after normalization annealing. The dark places are pearlite, which is an eutectoid mixture of ferrite and cementite. The white places represent secondary cementite. The 90MnCrV8 steel (see Fig. 1b) is a steel with a ferritic - pearlitic structure. The white places represent ferritic grains. The dark places are pearlite. The X82WCrV9-5-2 steel (see in Fig. 1c) is also a steel with a ferritic – pearlitic structure. The white places represent ferritic grains that are smaller than the previous steel. The dark areas are pearlite. After quenching of steel of the higher carbon or alloying elements at lower temperature M_s (Mn, Ni, Co) a certain amount of residual austenite remains in the steel. Residual austenite reduces the hardness of the steel, therefore the supereutectoid steel hardens from temperatures above A_1, where part of the carbon and alloying elements remain bound to carbides and their lower content in austenite leads to an increase in temperature M_s.

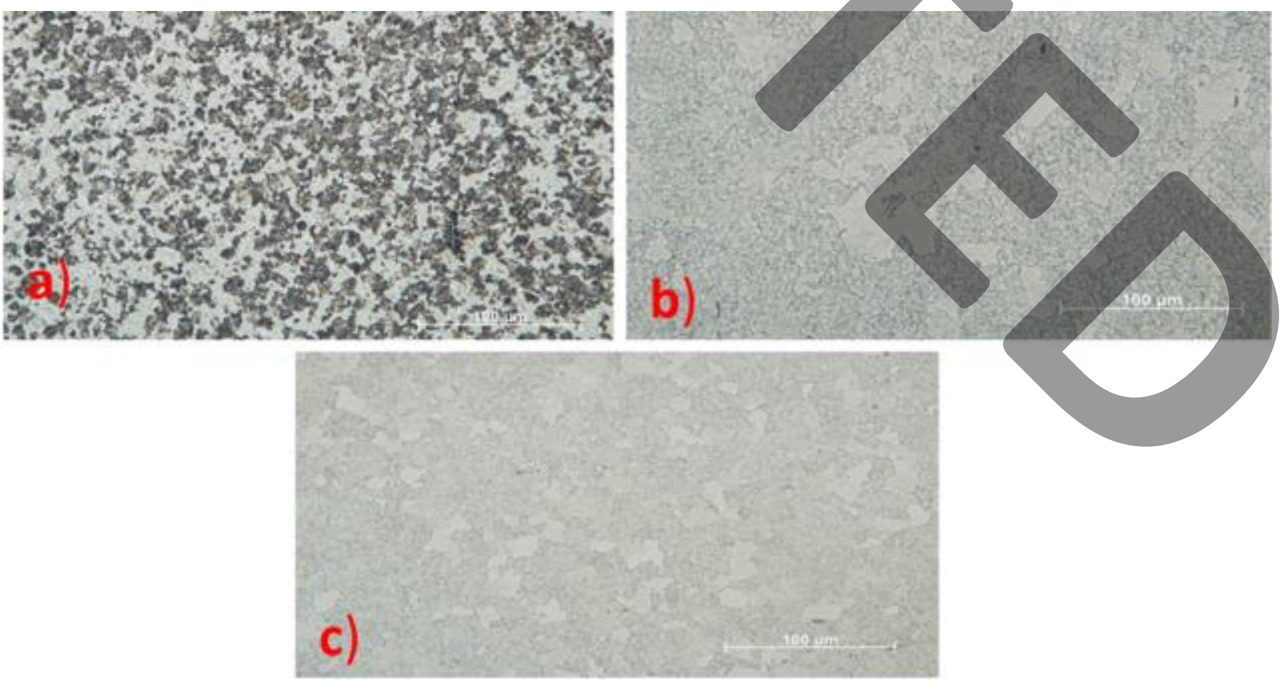

Figure 1. Microstructure of tested a) C120U steel; b) 90MnCrV8 steel; c) X82WCrV9-5-2 steel

Table 1. Chemical composition and basic mechanical properties of tested C120U tool steel

Chemical composition and mechanical properties of C120U tool steel								
Element	C	Mn	Si	P	S	Cr	Ni	Fe
weight [%]	1.10-1.24	0.20-0.35	0.15-0.30	max 0.025	max 0.30	max 0.15	max 0.20	balance
Hardness HRC	59 - 66 (h. t. 770 °C / water; t. t. 250 – 100 °C / 2h)							
Flexural strength R_{mo} [MPa]	~3 750 (at hardness HRC 60)							
Yield strength in pressure R_{et} [MPa]	~ 2 600 (at hardness HRC 60)							

Table 2. Chemical composition and basic mechanical properties of tested 90MnCrV8 tool steel

Chemical composition and mechanical properties of 90MnCrV8 tool steel							
Element	C	Mn	Si	P	S	V	Fe
weight [%]	0.75-0.85	1.85-2.15	0.15-0.35	max 0.030	max 0.035	0.10-0.20	balance
Hardness HRC	55 - 62 (h. t. 780 °C / oil; t. t. 300 – 100 °C / 2h)						
Flexural strength R_{mo} [MPa]	~ 4 300 (at hardness HRC 60)						
Yield strength in pressure R_{et} [MPa]	~ 2 200 - 3000 (at hardness HRC 55 - 62)						

Table 3. Chemical composition and basic mechanical properties of tested X82WCrV9-5-2 tool steel

Chemical composition and mechanical properties of X82WCrV9-5-2 tool steel									
Element	C	Mn	Si	P	S	Cr	W	V	Fe
weight [%]	0.75-0.85	max 0.45	max 0.45	max 0.035	max 0.035	3.80-4.60	8.00-9.50	1.30-2.00	balance
Hardness HRC	~ 64 (h. t. 1230 °C / oil; t. t. 540 – 560 °C / 3 x 1h)								
Flexural strength R_{mo} [MPa]	~ 3 200 – 4 400 (at hardness HRC 64 - 65)								
Yield strength in pressure R_{et} [MPa]	~ 3 500 (at hardness HRC 65)								

Residual austenite has a positive effect against tool cracking during hardening. With increasing content of residual austenite, deformations during hardening are reduced (martensite has a larger volume than austenite) and the toughness of the steel increases. Residual austenite is transformed to martensite during the process of tempering. The carbides in tool steels increase hardness. They are harder than the matrix. Method of excluding carbides in the matrix have effect on plastic properties and toughness of tool steels. The exclusion of carbides can be changed by forming. During the forming, the carbides are crushed and are strength-oriented in the direction of forming.

2.4 Experimental methods

On the measuring device of type Hysitron Triboindenter TI 950 and its evaluation software Triboscan was performed nanoindentation analysis. All quasistatic nanoindentation tests were performed at room temperature with the application of Berkovich indentation tip geometry in the laboratory of mechanical testing CEDITEK at the Faculty of Special Technology in Trencin. All quasistatic nanoindentation measurements were performed on metallographic samples. Measurement by the quasi-static nanoindentation method requires the embossing the Berkovich testing tip into the sample under specified control load or displacement. The shift (*h*) is monitored as a function of the load (*P*) throughout the load-unload cycle, where the *P*-*h* dependence is known as the nanoindentation curve. The area under the load and unload curves is then equivalent to the energy of scattering. In the nanoindentation measurements, the load common with the displacement was recorded when the Berkovich testing tip was pressed into the surface of the measured sample using standard *P*-*h* profiles. This method of quasistatic nanoindentation was used at selected locations of the basic material of the microstructure of the test specimens. The individual areas of research were determined with the help of an optical microscope as an integrated part of the device (see Fig. 2). Subsequently was performed the SPM (Scanning Probe Microscopy) scan of a selected area with dimensions of 50x50 µm (see Fig. 3, 4, 5). The selection of individual places for the implementation of the indents themselves for the selected material were defined by a mechanical form with a selected number of indents on examined area. As a loading curve was used in the process of the performed experiment standard trapezoid with a maximum value of 8000 µN and a total holding time indentation of t = 2 s. Marking of positions for individual indents for the base material for all types of tested tool steels are shown on the above-mentioned SPM scans. The measured values of nanoindentation hardness H [GPa] and reduced Young's modulus of elasticity E_r [GPa] in their individual positions were subsequently evaluated using of Triboscan software. After completing the measurement process are generated the *P*-*h* curves for the individual indents are shown in Figs. 3, 4, 5.

Figure 2. Experimental setup and configuration of performed measurement

Calculations of the Young's modulus of elasticity phase E_s [GPa] for the investigated tool steels were performed according to equation (1):

$$E_s = (1-\upsilon_s^2) / (\frac{1}{E_r} - \frac{1-\upsilon_i^2}{E_i}) \tag{2}$$

where E_i is the modulus of the test tip and υ_s a υ_i are the Poisson constants for the sample and the Berkovich type test tip. Values E_i = 1141 GPa, υ_i = 0,07 a υ_s = 0,29 are used in all calculations.

3 Results and discussion

As part of the iplementation of nanoindentation tests, measurements consisting of six to seven indents were performed at selected places of the microstructure of the test area (Boundary). In the process of performed experiments, all three measured areas were bounded by dimensions of 50x50 µm. As a loading curve for all performed measurements was used standard trapezoid with a maximum value of F = 8000 µN and a total holding time indentation t = 2 s. As a test device was used nanoindenter of type Hysitron Triboindenter TI950. The measured positions of the individual indents are shown on SPM (Scanning Probe Microscopy) scans of the evaluated areas in all tested steel samples of the C120U, 90MnCrV8, and X82WCrV9-5-2. The measured values of nanoindentation hardness H [GPa] and reduced Young's modulus of elasticity E_r [GPa] in individual positions are shown in Tab. 4, 5, 6. In the Figs. 3, 4, 5 are the resulting shapes of the individual nanoindentation curves obtained from the indents on the SPM scans of the evaluated areas of the tested samples. The marking of the curves is identical with the marking of the positions of individual performed measurements.

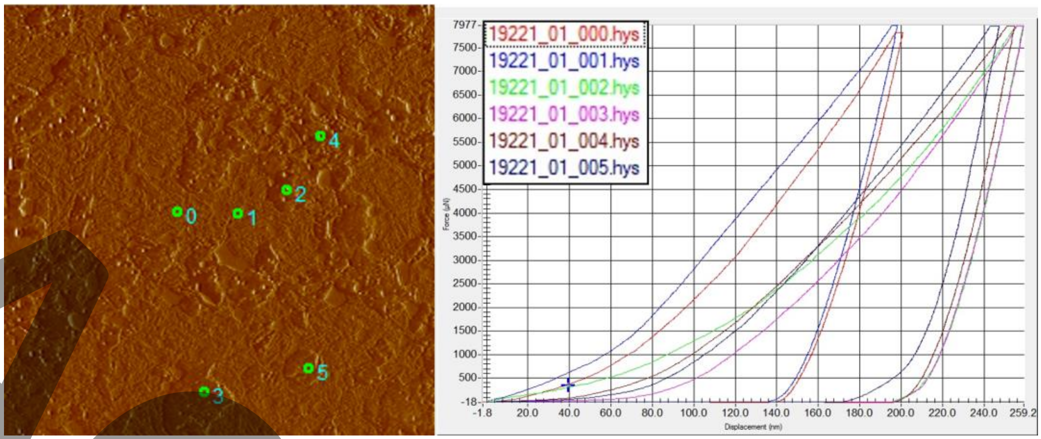

Figure 3. Distribution of individual indent positions on SPM scans of the tested C120U tool steel sample (Left) and Nanoindentation curves obtained from indents on SPM scans of C120U tool steel (Right)

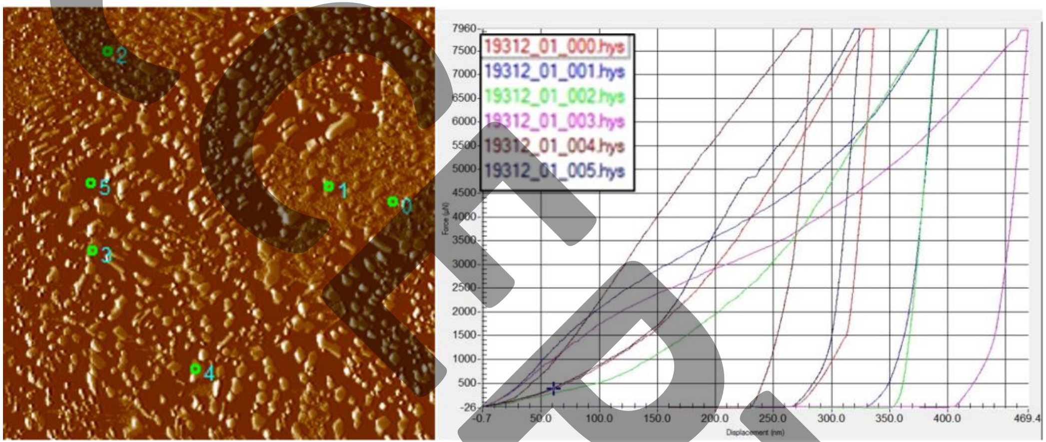

Figure 4. Distribution of individual indent positions on SPM scans of the tested 90MnCrV8 tool steel sample (Left) and Nanoindentation curves obtained from indents on SPM scans of 90MnCrV8 steel (Right)

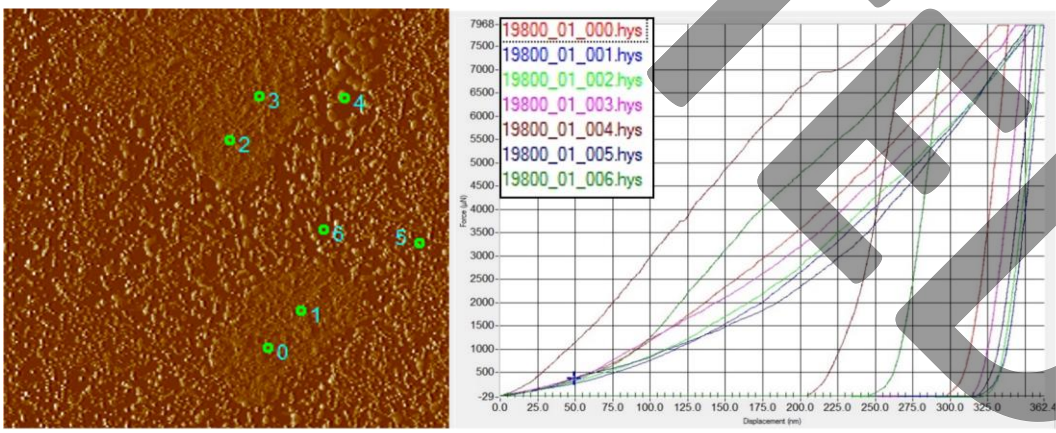

Figure 5. Distribution of individual indent positions on SPM scans of the tested X82WCrV9-5-2 tool steel sample (Left) and Nanoindentation curves obtained from indents on SPM scans of X82WCrV9-5-2 (Right)

A general overview of the individual tested phases for all types of tool steels and their nanohardness *H* and the reduced Young's modulus of elasticity E_r are shown in Tab. 4, 5, 6.

Table 4. Measured local mechanical properties components structure of C120U tool steel

Position	Nanohardness H [GPa]	Reduced modulus of elasticity E_r [GPa]	Phase (estimated)
0	10.88	207.69	Cementite
1	11.38	216.43	Cementite
2	5.98	178.60	Pearlite (cem. c.)
3	6.03	170.65	Pearlite (cem. c.)
4	6.14	189.91	Pearlite (cem. c.)
5	6.53	200.23	Pearlite (cem. c.)

Table 5. Measured local mechanical properties components structure of 90MnCrV8 tool steel

Position	Nanohardness H [GPa]	Reduced modulus of elasticity E_r [GPa]	Phase (estimated)
0	3.17	204.08	Ferrite
1	2.32	168.21	Ferrite
2	2.34	161.59	Ferrite
3	1.90	129.09	Pearlite (fer. c.)
4	4.53	243.95	Pearlite (cem. c.)
5	4.42	218.66	Pearlite (cem. c.)

Table 6. Measured local mechanical properties components structure of X82WCrV9-5-2

Position	Nanohardness H [GPa]	Reduced modulus of elasticity E_r [GPa]	Phase (estimated)
0	3.14	192.47	Ferrite
1	2.72	180.83	Ferrite
2	2.75	187.59	Ferrite
3	2.94	181.87	Ferrite
4	5.38	180.00	Carbide
5	2.80	193.33	Pearlite (fer. c.)

The mutual comparison of the reduced Young's modulus of elasticity E_r and according to the Equation (1) of the calculated Young's modulus of elasticity of the phase E_s for the individual types of tested tool steels are shown in Fig. 6, 7, 8.

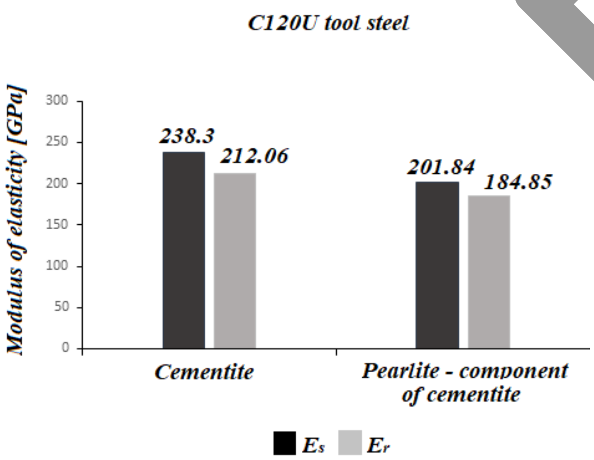

Figure 6. Comparison of measured modulus E_r and calculated modulus E_s for C120U tool steel

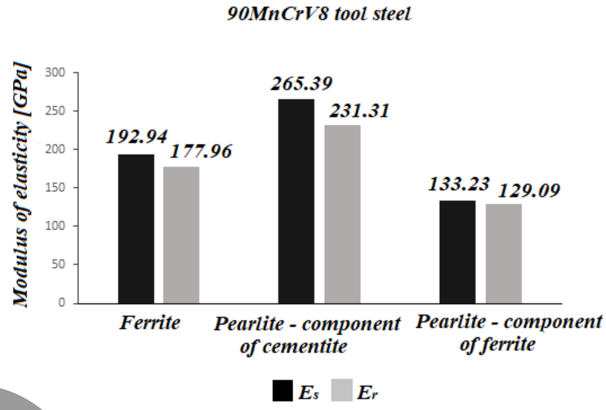

Figure 7. Comparison of measured modulus E_r and calculated modulus E_s for 90MnCrV8 tool steel

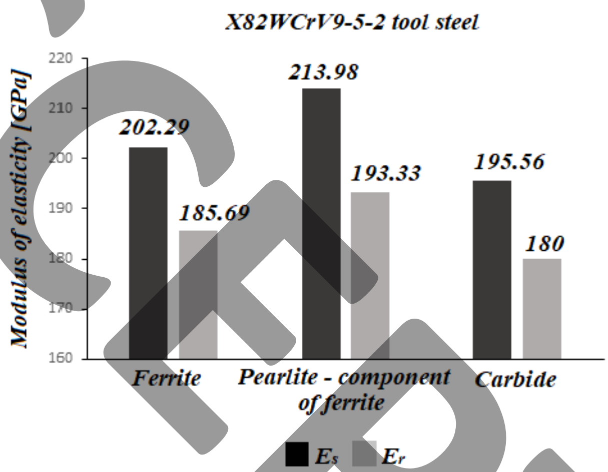

Figure 8. Comparison of measured modulus E_r and calculated modulus E_s for X82WCrV9-5-2 tool steel

When applying the technique of so-called “The depth of indentation” which is used in the quasistatic nanoindentation, the modulus of elasticity of the sample was determined from the slope of the relief curve as a reaction after loading (Displacement). The modulus measured in this way is formally called the "Indentation modulus" of the EIT sample material. Ideally, the indentation modulus has exactly the same, meaning as the term "Elastic modulus" or "Young's modulus", but this is not the case for some materials. The value of the indentation module can therefore be influenced by the behaviour of the test material (e.g., by the accumulation of dislocations), which is not considered, when analysing the load transfer data.

4 Conclusion

The main goal of all experiments performed by the authors, which are described in detail in the previous chapters of the article, was to test the nanohardness of the basic structural components of individual C120U, 90MnCrV8 and X82WCrV9-5-2 tool steels using the method of quasistatic nanoindentation. The reason for choosing these types of steels was the fact that these steels are subject to high requirements in industrial practice, such as high strength, toughness, wear resistance and other mechanical properties that can be evaluated on the basis of hardness. Used measuring device was the Hysitron TI 950 Triboindenter with Triboscan evaluation software, and nanohardness values of the individual structural phase components and the reduced Young's modulus of elasticity were determined during the experimental process. The Berkovich testing geometry was used as a test indentation tip. The reduced modulus of elasticity was used to calculate the modulus of elasticity of the structural phase components according to Equation (1). The results of the performed calculations are clearly shown in Fig. 6, 7 and 8. From the comparison, it can be seen that the values of the modulus of elasticity in tension of individual phases are higher by 3% to 14% than their reduced modulus, assuming the above values of the Berkovich indenter. By means of the quasistatic nanoindentation in the form of selection of test positions corresponding to individual phases, it is possible to determine the mechanical properties of individual phases in the investigated areas at the nano level. This fact subsequently gives an area for further research of the basic material, for example by creating matrices of individual indents, where it is possible to determine the percentage of individual phases in the investigated microstructures.

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