University Review, Vol. 15, Issue 1, p. 14-19, 2021.

UNIVERSITY REVIEW

INVESTIGATION OF NANOMECHANICAL PROPERTIES OF MICRO-STRUCTURAL COMPONENTS OF SELECTED ALLOY TOOL STEEL

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ARTICLE INFO

***Article history*:**
Received: 15.3.2021
Received in revised form: 23.4.2021
Accepted:3.5.2021

Keywords:
Quasistatic nanoindentation
Berkovich test tip
Nanohardness
Reduced Young's modulus
Tool steels

Abstract:

The aim of this study is to measure and evaluate of mechanical properties of microstructural components of alloyed tool steel C120U according to STN Standard. The measurement was performed on an experimental device Hysitron TI 950 Triboindeneter, which is a part of CEDITEK Laboratories at FST TnUAD. The testing of alloyed tool steel C120U was performed due to the high demands on tool steels in industrial practice, such as high strength, toughness, fatigue and abrasive wear resistance, corrosion resistance, temperature stability and others. The Berkovich test tip type was used in the research process. Chapter 1 describes the research of foreign authors who focused on the mechanical properties of high-strength steels. Chapter 2 shows the results of own experiments such as chemical composition, mechanical properties, evaluation and description of microanalysis of alloy tool steel C120U by using light microscopy. The calculation of the Young's modulus of elasticity and the experimental method are also found in Chapter 2. Chapter 3 presents the measured mechanical properties of the components of the structure of the tested steel, the distribution of individual indent positions on SPM (Scanning Probe Microscopy) scans, the nanoindentation curve obtained from indents on SPM scans and a comparison of Young's modulus of elasticity Er and calculated Young's modulus of elasticity phase Es. The conclusion and evaluation of the measured data is given in Chapter 4.

1 Introduction

Quasi-static nanoindentation is a contact method which consists in mechanical contact of the test tip of the investigated material, 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 mechanical properties. Quasi-static nanoindentation differs from 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 conventional tests for measuring the hardness of materials, the contact area is calculated from direct

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measurements of the dimensions of the residual impression which remained on the sample surface after removal of the load [1,5]. When tested method by the quasi-static nanoindentation is the size of the residual impression in micrometers is too small to be measured directly. Therefore, it is common to determine the contact area by measuring the penetration depth of the test tip into the surface of the test specimen [1]. Nanoindentation techniques can also be used to calculate elastic modulus, deformation curing exponent, fracture toughness (for example for brittle materials) and viscoelastic properties. Data are obtained when the test tip is brought into contact with the flat surface of the sample with increasing load. 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] in his work performed nanoindentation tests of samples at room temperature on a NanoTest nanoindent supplied by company Micro Materials Ltd., Wrexham, UK, with using a three-sided Berkovich diamond tip with a nominal angle of 120 ° and a radius r = 100 nm [4]. Nanoindentation tests were performed at the same maximum load (F = 500 mN), with load speed of 50, 25, 16.67, 12.5, 10, 5 and 1 $mN.s^{-1}$ The test tip was then left to endurance at maximum load for t = 5 s. Then it followed by unloading with speed of 50 $mN.s^{-1}$ and for all tests. At least 10 indentation points were performed and for each load separately. The measurements results were subsequently averaged [4]. All hardness values measured during the nanoindentation process in the authors' study [4] are higher than the hardness values of the tested steel H13 [6,8]. The steel H13 was produced in the basic state, but without the use respectively participation SLM (Selective Laser Melting) obtained from the results of the Mencin process [3,4]. The results of this study are in agreement with the results of previous experimental reports on nanoindentation tests of H13 material [6,8]. The authors of the study [2] performed nanoindentation tests with samples at room temperature in order to evaluate the mechanical properties of SLM H13 steel. A three-sided diamond Berkovich test tip was used from company Micro Materials Ltd., Wrexham, UK. The maximum load was chosen with sufficient size to ensure the presence of indents at all stages of sample testing. Nanoindentation tests were performed at the same maximum load (500 mN) with the achieved load speed heights of 50, 25, 16.67, 12.5, 10.5 and $1mN.s^{-1}$. The test tip was then left to endurance at maximum load for t = 5 s behind which followed by unloading at a speed of 50 $mN.s^{-1}$ and for all tests. For each load there were at least ten indents and the results are then averaged [2]. The load stress (r), a representative load from the nanoindentation test, is defined as the instantaneous load (P) divided by the projected contact surface (Ac), which is also the definition of the indentation hardness (H) measured during [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 the depth and time data obtained for a given range of indentation depths [2].

2 Materials and methods

2.1 Experimental method

Nanoindentation analysis was performed on a measuring device of the Hysitron Triboindenter TI 950 type (Fig. 1) and its evaluation software Triboscan (Fig. 2). Testing was performed at room temperature with the application of Berkovich's internal geometry in the laboratory of mechanical testing CEDITEK at the FST in Trenčín. Quasi-static nanoindentation measurement was realized on a metallographic sample (Fig. 3).

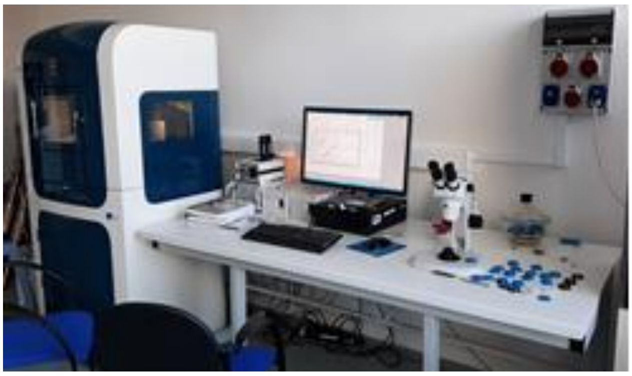

Fig. 1 Work equipment Hysitron TI 950 Triboindenter with accessories

During the nanoindentation measurement was recorded the load together with the displacement, when the Berkovich tip was pressed into the surface of the measured sample using standard *P-h* profiles. The quasi-static nanoindentation method was used at designated locations of the base material of the microstructure of the test sample (Fig. 3).

Fig. 2. Selection from the basic menu - selection of an appropriate measurement methodology.

The individual areas of research were determined with the help of an optical microscope as an built-in part of the device (Fig. 3).

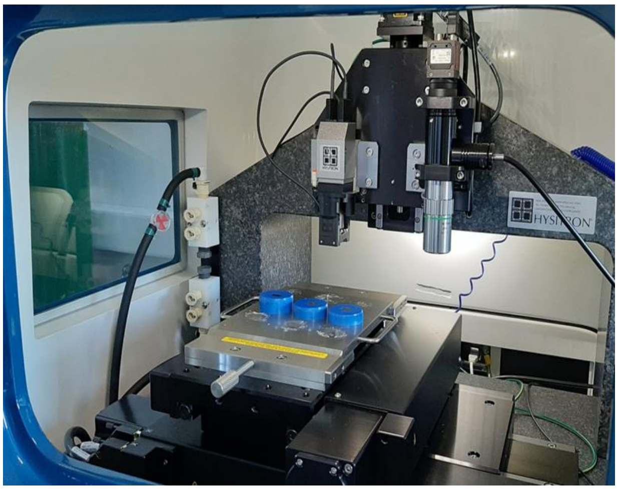

Fig. 3. Display of a metallographic sample

Subsequently, an SPM scan of a selected area with dimensions of 50x50 μm was performed (see Fig. 5). The selection of individual places for the implementation of indents for the selected material were defined by a mechanical form with a selected number of indents on the examined area. As a loading curve was used in the process experiment a standard trapezoid with a maximum at 8000 μN and with the total indentation time t = 2 s. The designations of the positions for the individual indents for the base material of the tested tool steel C120U are shown in Fig. 5. This way measured the values of nanoindentation hardness H [GPa] and reduced Young's modulus Er [GPa] in their individual positions were using of Triboscan software subsequently evaluated. At the end of the measurement process, *P-h* curves are generated for the individual indents shown in Fig. 6.

2.2 Calculation of Young's modulus of elasticity of the phase

The calculation of the Young's modulus of elasticity of the phase E_s for the investigated alloyed tool steel C120U was realized according to the relation (1):

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

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. The values E_i = 1141 GPa, υ_i = 0,07 a υ_s = 0,29 are used in all calculations.
The value E_s for the cementite phase:
The calculation of the Young's modulus of elasticity of the phase E_s for the cementite phase for alloy tool steel type C120U it is calculated below. Where the calculated average value of the reduced Young's modulus of elasticity is E_r =212,06 GPa. After substituting the given values into the relation (1), the Young's modulus of elasticity of the phase is E_s = 238,30 GPa. The values of the reduced Young's modulus of elasticity E_r and the Young's modulus of elasticity of the phase E_s are given in Table 1.

Table 1 The Reduced modulus of elasticity and Young's modulus of elasticity of the phase

Steel	Phase			
	Cementite		Perlite (cementite component)	
	Es [GPa]	*Er* [GPa]	*Es* [GPa]	*Er* [GPa]
19221	238,30	212,06	201,84	184,85

The value E_s is for the perlite phase (cementite component):
The average value of the reduced Young's modulus of elasticity in this phase is E_r = 184,5 GPa. Other

values such as test tip modulus and Poisson constants for sample and test tip are the same as in the previous phase. The Young's modulus of elasticity of the phase for the perlite phase (cementite component) is E_s = 201,84 GPa.

2.3 Mechanical properties and chemical composition

The steel C120U is an alloyed tool steel with a higher carbon content of 1.1%. This steel was chosen from due to achievement a high hardness after hardening (min. 64 HRC) and which is tempered to 60 ± 2 HRC. This type of alloyed tool steel is used for cutting, shearing and forming tools, hand tools and gauges. The steel C120U has good toughness in core, insensitivity to hardening cracks, more difficult hot formability and good machinability in the annealed state. The chemical composition and mechanical properties of the tested alloyed tool steel C120U are shown in Table 2.

2.4 Microstructural analysis

The steel C120U is a supereutectoid steel with a cementitic-pearlitic structure (Fig. 4). The steel is in the state after normalization annealing. The dark places represent perlite, what is a eutectoid mixture of ferrite and cementite. The white areas represent secondary cementite.

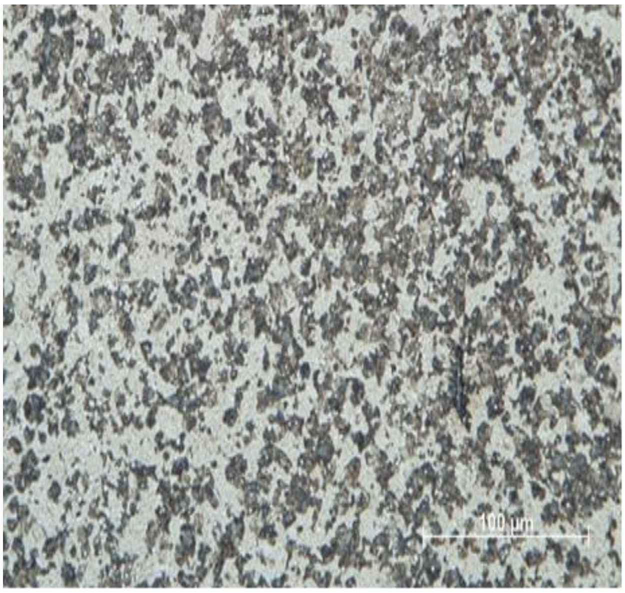

Fig. 4. Microstructure of alloyed tool steel C120U
Table 2 Chemical composition and basic mechanical properties of alloyed tool steel C120U

Chemical composition and mechanical properties of the tested steel C120U							
Chemical composition of steel C120U according to ISO Standard [wt. %]							
Element.	*C*	*Mn*	*Si*	*P*	*S*	*Cr*	*Ni*
wt. %	1,10-1,24	0,20-0,35	0,15 - 0,30	max 0,025	max 0,30	max 0,15	max 0,20
Chemical composition of steel C120U measured by spectral analysis [wt. %]							
Element	*C*	*Mn*	*Si*	*P*	*S*	*Cr*	*Ni*
wt. %	1,15-1,25	0,10-0,40	0,10 - 0,30	max 0,03	max 0,03	max 0,15	max 0,20
Mechanical properties of steel C120U							
Hardnes sHRC	59 - 66 (H. t. 770 °C / water; T. t. 250 – 100 °C / 2h)						
Flexural strength R_{mo} [MPa]	□ 3 750 (at HRC 60)						
Yield strength in pressure R_{et} [MPa]	□ 2 600 (at HRC 60)						

3 Results and discussion

As part of the nanoindentation test, measurements were performed consisting of six to seven indents at the selected place of the microstructure of the test area (Boundary). In the process experiment was measured area bounded by dimensions of 50x50 µm. As the loading curve was for realized measurement used standard trapezoid with a maximum at 8000 µN and an indentation time t = 2 s. The experimental device nanoindentor type Hysitron Triboindenter TI 950 was used as a test device. The measured positions of the individual indents are shown on the SPM (Scanning Probe Microscopy) scan of the evaluated area of the tested sample from C120U steel (Fig. 5). The measured values of nanoindentation hardness H [GPa] and reduced Young's modulus of elasticity E_r [GPa] in individual positions are given in Table 3. On Fig. 6 are shows the resulting shapes of the individual nanoindentation curves obtained from the indents on the SPM scan of the evaluated area of the test sample. The designation of the curves is identical with the designation of the measuring positions in Fig. 5 and in Table 3.

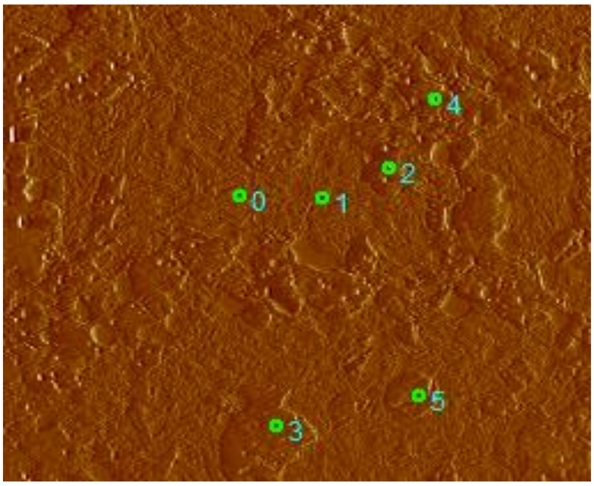

Fig. 5. Deployment of individual positions indents on SPM scan in the tested sample of alloyed tool steel C120U

Table 3 Measured mechanical properties of components structure alloyed tool steel C120U

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	Perlite (cem. c.)
3	6,03	170,65	Perlite (cem. c.)
4	6,14	189,91	Perlite (cem. c.)
5	6,53	200,23	Perlite (cem. c.)

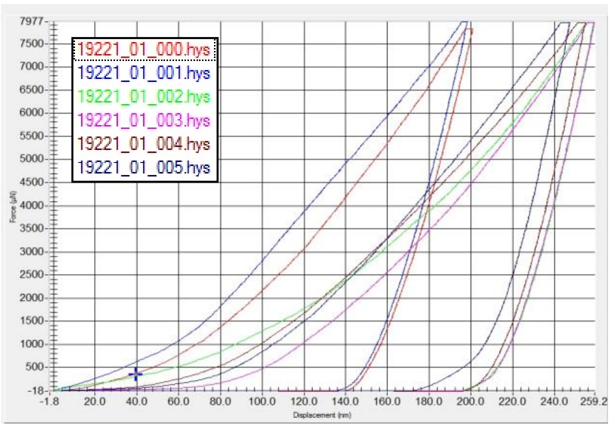

Fig. 6. Nanoindentation curves obtained from indents on SPM scans steel C120U

Load part of the indentation curve is used to evaluate nanohardness, where the unloading part is used to calculate reduced Young moduluas (see Fig. 6). Overall overview of the individual tested phases for the alloyed tool steel C120U and its nanohardness *H* and the reduced Young's modulus of elasticity E_r are shown in Table 3. A mutual comparison of the reduced Young's modulus of elasticity E_r and by relationship (1) the calculated Young's modulus of elasticity of phase E_s for the tested alloy tool steel C120U is shown on Fig. 7.

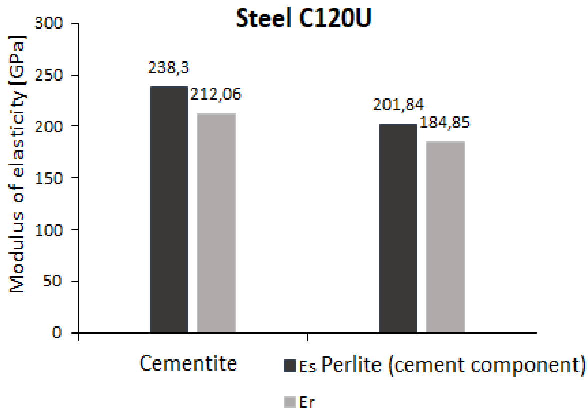

Fig. 7. Comparison of measured modulus Er and calculated modulus Es for alloyed tool steel C120U

Using the Hysitron TI 950 Triboindenter, the nanohardness values of the individual structural phase components were determined, as well as the reduced modulus of elasticity. The Berkovich type was used as a test tip. The reduced modulus of elasticity was used to calculate the modulus of elasticity of specific structural phase components. The results of the calculation are clearly marked in the graph on the Fig 7. It can be seen from the comparison that the values of the tensile modulus of elasticity of the individual phases are higher by 3% to 14% than their reduced modulus, assuming the above-mentioned values of the Berkovich indenter.

4 Conclusion

The aim of the performed experiment was to test the nanohardness of the basic structural components of the selected alloyed tool steel C120U with using the experimental method of quasi-static nanoindentation. The reason for the chosen alloy tool steel C120U was the fact that on this steel higher demands are placed in practice, such as high strength, wear resistance, toughness and other mechanical properties that can be review and evaluated on the basis of hardness. Using the test device Hysitron TI 950 Triboindenter, which is equipped with the evaluation software Triboscan were detected the nanohardness values through experiment by of the specific structural phase components as well as the reduced Young's modulus of elasticity. During testing was used indentation tip type Berkovich. The determined reduced Young's modulus of elasticity obtained by nanoindentation was used to calculate the Young's modulus of elasticity of the phase. The result of the calculation is clearly shown in Fig. 7. It is clear from the comparison that the values of the tensile modulus of elasticity of the individual phases are higher than their reduced modulus of elasticity, assuming the stated values of the Berkovich indenter.

5 Acknowledgement

This publication was created within the implementation of the project "Development and support of research - development activities of the Center for Quality Testing and Diagnostics of Materials in the field of specialization RIS3 SK", ITMS2014 +: 313011W442, supported by the Operational Program Integrated Infrastructure and European Regional Development Fund.

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