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APPLICATION OF QUASI-STATIC NANOINDENTATION METHOD FOR THE RESEARCH OF MECHANICAL PROPERTIES MICROSTRUCTURAL COMPONENTS OF TOOL STEEL

Henrieta Chochlíková[1] - Jana Escherová[2]

[1] Department of Engineering Technologies and Materials, Faculty of Special Technology, Alexander Dubcek University of Trencin, Ku kyselke 469, Trencin Slovakia,

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Abstract:

The material 90MnCrV8 was used for the valuation of mechanical properties of microstructural components of tool steel. Tool steel has to satisfy high demands in industrial practice such as high strength, toughness, wear resistance, temperature stability and so on. Experimental measurements were performed on a Hysitron TI 950 Tridoindenter with Triboscan evaluation software. The Berkovich type was used as a test tip.

The quasi- static nanoindentation is used as a methodology of measurement. The methodology and evaluation of microanalysis of tested tool steel by light microscopy is described in chapter 2. Chapter 3 contains own measurements and evaluation of measured data of tested tool steel using the method of quasi- static nanoindentation. .

1 Introduction

Using quasi-static nanoindentation we can measure mechanical properties such as modulus and hardness of materials in different shapes, sizes and scales. Thanks to this method we can measure mechanical properties from the hardness materials to soft biomaterials in a couple minutes. Quasi- static nanoindentation is used in research in different industrial fields in order to find out of nanomechanical properties of thin layers in electronics and packaging materials, coatings of cutting tools, coatings for thermal barriers, visco- elastic properties of polymeres, microhardness in industrial quality and control, resistance against scratching and wear and many more. Nano- hardness H [GPa] isn´t the only value that comes from quasi- static nanoindentation. This method can be used also for measurement and evaluation of reduced Young modulus of elasticity E_r [GPa]. Nanoindentation techniques can also be used for the calculation of elastic modulus, deformation-curing exponent, fracture toughness (for example for brittle materials) and visco- elastic properties.

Fundamentally it is relatively simple method of researching local mechanical properties, which is based on the indentation of an object with known dimensions and mechanical properties with a certain force into the studied material with a penetration depth in the nanometer scale. Load and depth of indentation are recorded at each load increment, which ultimately provides a measure of modulus hardness as a function of depth below the surface. The loading part of indentation cycle can consist of the initial elastic contact following with plastic deformation or loading of tested material at higher loads. The maximal depth of indentation for specific

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Corresponding author. Tel.: +421327400218
E-mail address: henrieta.chochlikova@tnuni.sk

loading together with inclination of indentation curve measured in tangent to the point considered at maximum load, leads to measuring the hardness and elastic modulus of sample material [1].

The main goal of quasi- static nanoindentation is to measure values of elastic modulus and nanohardness of the test material of the sample from the experimental values of the test tip of the load and penetration depth.

Nguyen et al. [6] aimed on measuring nanometric characteristics (microstructural characteristics and mechanical properties are investigated) of H13 material using scanning speed of 100 mm.s^{-1}. Measurements detected a relation between nanoindentational deformation and toughness. With an increase of speed of deformation (0,002 to 0,1 s^{-1}) the toughness is increased also in a range of 8,41 to 9,18 GPa. The work of these authors [8] presents a comparative study of several methods of nanoindentation which were applied on ferritic-martensitic steels of type T91 (9Cr-1Mo) and Eurofer 97 [7]. Measurements were realized with CSM method (Continuous Stiffness Measurement - CSM). Depth-controlled single cycle measurements atvarious indentation depths, force-controlled single cycle, force-controlled progressive multi-cycle measurements, ancontinuous stiffness measurements (CSM) using a Berkovich tip at room temperature have been combined to determine the indentation hardness and the elastic modulus, and to assess the robustness of the different methods in capturing the indentation size effects (ISE) of those steels [7]. Quasistatic methods for individual cycles with controlled depth and strength and progressive multi-cycle measurements show common accord, whereas continual measurements of toughness are depending on amplitude [8]. Studies [5] concerned with comparing curves *P-h* during maximal load, were used as a comparing curves *P-h* with results of exploring mechanical properties of microstructural parts of tool steel.

2 Material and methodology of measurement

2.1 Properties and microstructure of tool steel 90MnCrV8

Steel 19 312, 90MnCrV8 belongs to alloyed tool steels according to the STN standard. The most important alloying elements of these steels are Cr, Mo, V, and W. These elements are carbide- forming and they increase the hardness and stability of the carbide phase and reduce a decrease of hardness during tempering. They also increase resistance against wear in a large extent. As these alloying elements increase depth of hardening, it is possible to produce tools of bigger proportions. Besides, we can increase the toughness by adding Ni. Chemical composition of steel 90MnCrV8 is shown in table 1 [4]. Steel 90MnCrV8 is suitable for manufacturing cutting tools for non-metals (knives on paper), tools for cutting in cold conditions (different shaping dies), tools for pulling sheets, moderately stressed forms for pressing metalling and non- metalling powders, whose shape is more complicated, for processing plastics requiring good stability of dimensions after thermic processing, production of gauges [2],[3].

Table 1 Chemical composition of steel 90MnCrV8 [average wt. %] [4]

Chem. element	C	Mn	Si
Average wt. %	0,75-0,85	1,85-2,15	0,15-0,35
Chem. element	P	S	V
Average wt. %	Max 0,030	Max 0,035	0,10-0,20

Tested material 90MnCrV8 is steel with ferritic-pearlitic structure. The white areas represent ferritic grains, and the dark areas represent pearlitic grains, e.g. in Fig. 1.

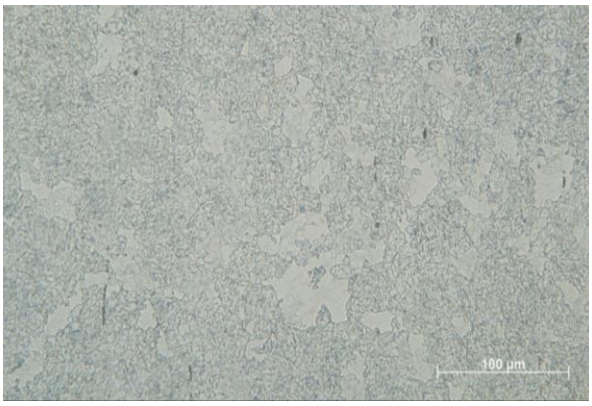


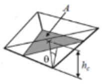
Fig. 1 Microstructure of testing steel 90MnCrV8

2.2 Methodology of measurement

Nanoindentation analysis was performed on a measuring device of the Hysitron TI 950 Tridoindenter with Triboscan evaluation software. The nanoindentation test was performed at room temperature with application of internal geometry the Berkovich in laboratory of mechanical tests CEDITEK on Faculty of special technology in Trenčín.

Quasi-static nanoindentation measurements were released on metallographic sample of tool steel 90MnCrV8 (equivalent 1.2842; STN 19 312). Measurement by the quasi-static nanoindentation method requires the indentation of test tip Berkovich geometry into the sample under specified load control or displacement. The displacement (*h*) is monitored as a function of the load (*P*) during the whole cycle of loading- unloading, where dependence *P-h* is known as nanoindentation curve. The area under curves of loading and unloading is then equivalent to energy of dispersion. In all the performed nanoindentation measurement, the load together with the displacement was recorded when the Berk test tip was pressed into the surface of the measured sample using standard *P-h* profiles. Parameters of tip are mentioned in table 2. The method of quasi-static nanoindentation was used on chosen spots of basic material of microstructural testing sample (Fig. 1). The area of research was specified using optic microscope as an inbuilt part of device. Subsequently was realized a so-called SPM scan of a selected area with dimensions 50x50 μm.

Table 2 Geometry of testing tip a its projection [1]

Testing tip	Projection surface	Top peak angle θ (deg)	Effective conic angle α (deg)	Intercept factor $^{a}\varepsilon$	Geometric correction factor β	Projection
Berkovich	$A=3\sqrt{3}h_c^2\tan^2\theta$	65,26°	70,3°	0,75	1,034	

The selection of particular spots for realization of individual indents for chosen material were defined by mechanical form with selected number of indents on explored surface. The standard trapezoid with a maximum at 8000 μN and a total indentation endurance time $t = 2s$ was used as a loading curve in the process of the performed experiment. Measured values of nanoindentation hardness *H* [GPa] and reduced Young modulus of elasticity E_r [GPa] were then evaluated in particular positions using Triboscan software.

The realization of measurement using loading cycle and the development of indent curve formation (on the left) is seen in the Fig. 2. Results of measurement were generated to xls table of hardness values *H* (GPa) and reduced Young modulus of elasticity E_r (GPa) (table 3).

After the termination of measurement *P-h* curves for particular indents are generated, e. g. in Fig 4.

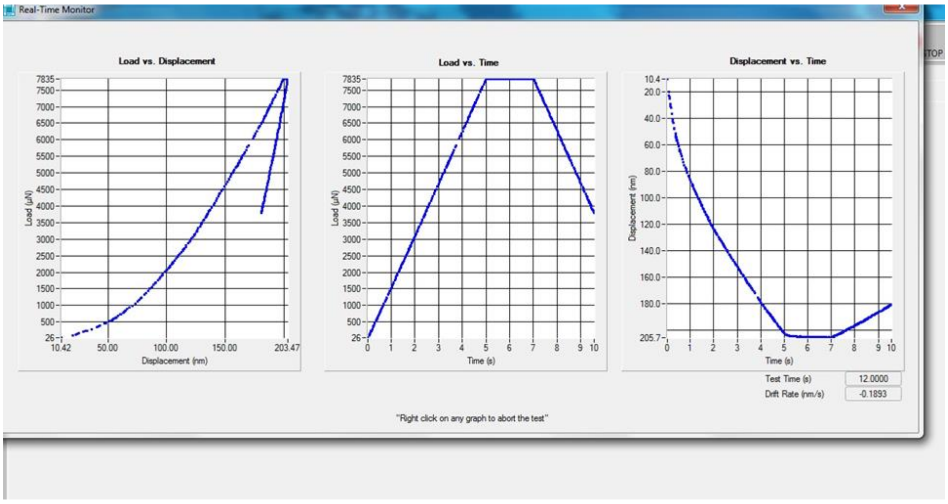
3 Material Measurement of nanoindentation hardness *H* and reduced Young modulus of elasticity E_r on microstructural particles of steel 90MnCrV8.

Measurement were composed of six or seven indents on chosen spots in microstructural tested area (Boundary). Dimensions of measured areas were limited on 50x50 μm. In the research the standard trapezoid with maximum in 8000 μN and a total indentation endurance time $t = 2$s was used as a loading curve in the process of the performed experiment. A Hysitron Triboindenter TI950 was used as a test device.

The placement of indents of valued area of steel 90MnCrV8 sample on SPM scan are seen in the Fig. 3. Measured values of nanoindentation hardness *H* [GPa] and reduced Young modulus of elasticity E_r [GPa] are visible in table 3.

The shapes of the nanoindentation curves obtained from the indents on the SPM scan of the evaluated area of the test sample of 90MnCrV8 steel are shown in Fig. 3. There could be an overlap to the ferrite field during the evaluation of perlitic cementite. Therefore, lower values of nanohardness *H* [GPa] could be measured.

The summary of phases of observed tool steel and their nanohardness H and reduced Young elasticity modulus E_r is shown in table 4.



Test Time (s) 12.0000
Drift Rate (nm/s) -0.1893

"Right click on any graph to abort the test"

Fig. 2 The course of the measurement by the load cycle and the course of the curve formation.

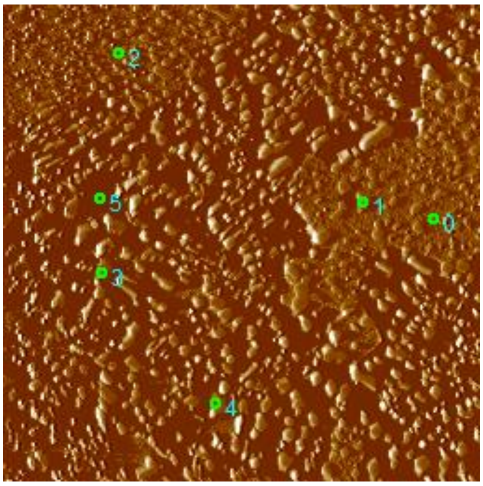


Fig. 3 Placement of indents on SPM scan steel 90MnCrV8

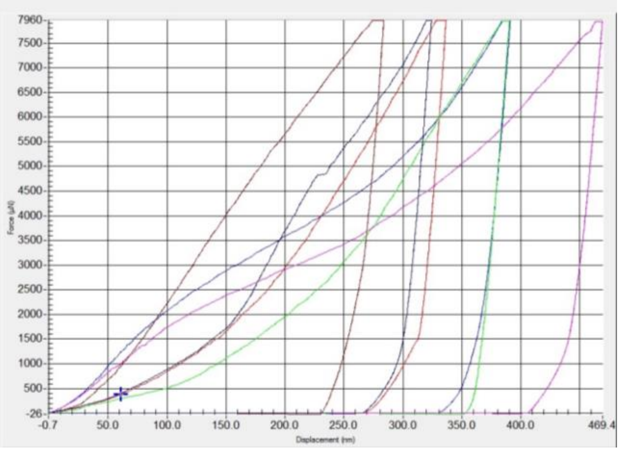


Fig. 4 Particular curves obtained from indents on SPM scan of steel 90MnCrV8.

Table 3 Local mechanical characteristics of steel 90MnCrV8 structure components.

Position	Nanohardness H [GPa]	Reduced modulus of elasticity E_r [GPa]	Phase (expected)
0	3,17	204,08	Ferrite
1	2,32	168,21	Ferrite
2	2,34	161,59	Ferrite
3	1,90	129,09	Perlite (Ferrite com.)
4	4,53	243,95	Perlite (cem. com.)
5	4,42	218,66	Perlite (cem. com.)

3.1 Calculation of Young modulus elasticity of the phase

Modulus of elasticity in overall talks about the ability of the material to resist elastic deformation under the influence of tension and is defined as a fraction of tension and deformation. From the analysis of indentation data, it is possible to obtain the modulus of elasticity from an angle of tangent the same way as using the determination of indentation toughness according to Oliver and Pharr [9] using the following relation (1).

The calculation of Young modulus of phase elasticity E_s for researched tool steel 90MnCrV8 was realized according to the equation 1.

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

where E_i is modulus of testing tip and υ_s and υ_i are Poisson constants for the sample and testing tip Berkovich. Values are E_i = 1141 GPa, υ_i = 0,07 and υ_s = 0,29.

Values of reduced Young modulus of elasticity E_r and Young modulus of phase elasticity E_s of tool steel 90MnCrV8 are written in table 5 and the comparision is shown in Fig. 5.

Tab. 4 Phases of steel and their nanohardness H [GPa] and reduced Young modulus of elasticity E_r [GPa]

Steel	Phase					
	Ferrite		Perlite (component of cementite)		Perlite (component of ferrite)	
	H	E_r	*H*	E_r	*H*	E_r
90MnCrV8	2,61	177,96	4,48	231,31	1,9	129,09

Tab. 5 Reduced Young modulus of elasticity E_r [GPa] and Young modulus of phase elasticity E_s [GPa].

Steel	Phase					
	Ferrite		Perlite (component of cementite)		Perlite (component of ferrite)	
	E_s	E_r	E_s	E_r	E_s	E_r
90MnCrV8	192,94	177,96	265,39	231,31	133,23	129,09

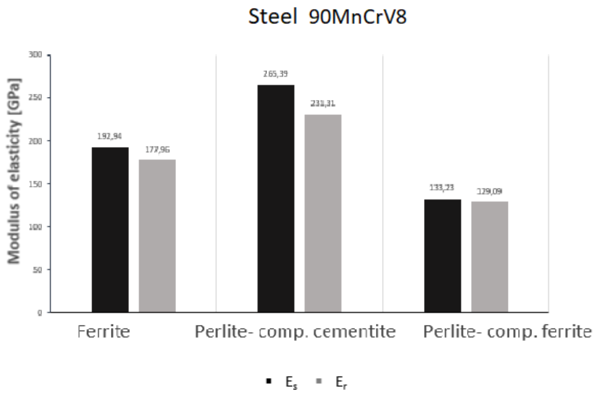


Fig. 5 Comparison of modules of elasticity phases of steel 90MnCrV8

4 Conclusion

The main goal of realized experiment was to test nanohardness of basic structural components ferrite and perlite of tool steel 90MnCrV8 using the method of quasi-static nanoindentation. The reason for choosing this particular kind of steel was the fact, that there are high demands on the material in industry, such as high strength, toughness, wear resistance, temperature stability and other mechanical qualities based on hardness.

The values of nanohardness of particular structural phases and reduced Young modulus of elasticity were detected using the testing device named Hysitron TI 950 Triboindenter, with Triboscan evaluation software. The Berkovich type was used as a test tip. Reduced modulus of elasticity was then used for the calculation of the modulus of elasticity of specific structural phase components according to the equation (1). Results of calculations are tabularly seen in the graph n. 1. From the mutual comparison it is visible that values of elasticity modulus in particular phases are higher by 4% up to 13% than their reduced elasticity modulus assuming the above-mentioned values of the Berkovich geometry test indentation tip used in the experimental process.

Using the experimental method of quasi-static nanoindentation and using the selection of test positions which correspond to particular phases, it is possible to determine mechanical characteristics of particular phases in explored areas on the nanolevel. This fact subsequently gives room 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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