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DETERMINATION OF MECHANICAL PROPERTIES OF NIOBIUM MICRO-ALLOYED STEEL USING INSTRUMENTED INDENTATION TEST

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Abstract

Niobium Nb micro-alloyed steels belong to a group of structural steels. They are alloyed with a small content of Nb (0.02-0.1 wt.%) which has a significant influence on many properties. Nb exhibits a strong affinity for nitrogen N and carbon C and causes the formation of niobium carbide NbC, niobium nitride NbN and niobium carbonitride Nb(C, N) in a structure which prevents grain growth and slows the recrystallization process and thus contributes to precipitation hardening. The mechanical properties of Nb micro-alloyed steel with Nb content of 0.035 wt.% were researched using instrumented indentation test. The indentation was carried out using different loads to determine the effect of the applied load on the measured properties and to research the applicability of the method for this group of materials. Based on the slope of the force-penetration depth curve during the loading and unloading, the Martens hardness, indentation hardness, indentation modulus, Vickers hardness, elastic/plastic work and creep characteristic were determined.

Keywords: *Nb micro-alloyed steel, instrumented indentation test, mechanical properties*

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INTRODUCTION

Niobium Nb micro-alloyed low carbon steel contains small amounts of Nb as an alloying element (0.02-0.1 wt%), which has a significant impact on many material properties [1]. Niobium shows a strong affinity for nitrogen and carbon and causes the formation of niobium carbide and niobium nitride within the structure of the steel which improve the grain refining, retardation of recrystallization, and precipitation hardening [1-6]. Those precipitates are dispersed in the form of small Nb (CN) precipitates, which are arranged in

fine lines [1,2,4]. Grain refinement is the only mechanism that simultaneously increases strength, toughness and ductility and makes Nb one of the most effective microalloying element, even if added in very small content [7]. Consequently, the toughness, hardness, yield point, ultimate tensile strength, formability, and weldability of the micro-alloyed steel is increased with a small decrease in elongation [1-6].
Today, there is a wide range of tests for the determination of the material's mechanical properties. Besides conventional tests such as tensile test, macro hardness measurement and Charpy impact test, instrumented indentation technique is widely used for the characterization. Instrumented indentation technique is relatively new technology developed in recent years which directly measures the mechanical properties from indentation load /displacement measurements [8]. It can measure accurately not only the hardness but also Young's modulus of each phase, accordingly, provides reliable data for the microstructure and performance design of material [8]. No visual observation of the indentation is required; eliminating the subjectivity of diagonal length measurements [9].
To research the applicability of instrumented indentation technique for this group of materials the research described in the paper is performed.

MATERIALS AND METHODS

The mechanical properties of Nb micro-alloyed low carbon steel using instrumented indentation test were researched in the paper. The chemical composition of low carbon Nb micro-alloyed steel is shown in Table 1.

Table 1. Chemical composition of the researched Nb micro alloyed steel, wt%

C	Mn	Si	P	S	Al	Nb	N
0.09	0.75	0.05	0.018	0.014	0.020	0.035	0.0081

Dynamic ultra-microhardness tester, type: DUH-211, manufactured by Shimatzu, Japan, was used for the instrumented indentation test. Triangular indentor with 115 ° tip angle (Berkovich indenter) was used. Measurements were performed using different forces; 5 measurements with the maximum forces F_{max} of 100 mN, 200 mN and 1961 mN were performed. The load of 1961 mN corresponds to measuring method HV0.2. Different forces were applied to investigate the influence of the test force on the measured properties and to investigate the applicability of the methods for this kind of materials.

Based on carried measurement, different mechanical properties were determined as follows [10]:

Martens hardness HM is determined from the values given by the force/indentation depth curve during the increasing of the test force (applied force), preferably after reaching the specified test force according to [10]:

$$HM = \frac{F}{26.43 \times h^2} \quad (1)$$

where is:
h – indentation depth, *F* – applied force.

Martens hardness (HMs) is determined from the slope of the increasing force/indentation depth curve according to equation [10]:

$$HM_s = \frac{1}{26.43 \times m^2} \quad (2)$$

where is:
m – slope of the curve, determined by the linear regression of the increasing force / indentation depth curve

Indentation hardness H_{it} is defined as a measure of resistance to permanent deformation and was calculated according to [10]:

$$H_{it} = \frac{F_{max}}{A_p} \quad (3)$$

Where is:

A_p – projected area of contact between the indenter and the test piece determined, for triangular indentor with 115 ° tip angle (Berkovich indenter) A_p=23.96 · h_c^2;

h_c - the depth of the contact between the indenter and the sample determined, $h_c = h_{max} - ¾ (h_{max} - h_r)$;

h_r - derived from the force-displacement curve and is the intercept of the tangent to the unloading cycle at Fmax with the displacement axis as presented in Figure 1.

Indentation modulus E_{it} is calculated from the slope of the tangent for the calculation of indentation hardness H_{it} according to equation [10]:

$$\frac{1}{E_r} = \frac{1-V^2}{E_{it}} + \frac{1-V_i^2}{E_i} \quad (4)$$

$$S = \frac{dP}{dh} = \frac{2 \times E_r \times A_p^{0,5}}{\pi^2} \quad (5)$$

Where is:

E_r – reduced modulus of elasticity, E_i - modulus of indenter (1.14 ·10^6 N/mm^2), V_i - Poisson ration of indenter (0.07), V - Poisson ration of the sample, S – inclination when unloading.

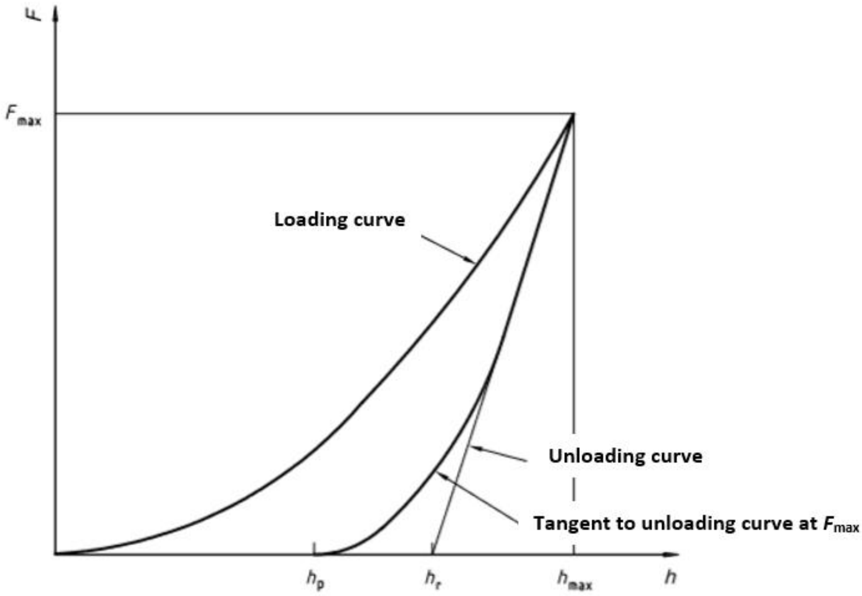


Figure 1. Force /indentation depth diagram [11]

Indentation creep C_{it} is defined as a relative change of indentation depth with respect to time of force application and is calculated according to [10]:

$$C_{it} = \frac{h_{2-h_1}}{h_1} \text{ , (\%)} \tag{6}$$

Where is:
h_1 – indentation depth at the time of maximum force, h_2 – indentation depth at the time of holding time.

Plastic and elastic parts of indentation work η_{it} is defined as a ratio of elastic work and total mechanical work W during application of the force and is calculated according to [10]:

$$\eta_{it} = \frac{W_{elast}}{W_{total}} \text{ , (\%)} \tag{7}$$

$$W_{total} = W_{elast} + W_{plast} \tag{8}$$

Based on the performed measurement, HV hardness is calculated from the equation [10]:

$$HV = 0.0924 \times H_{it} \tag{9}$$

RESULTS AND DISCUSSION

The results of measurements are presented in Tables 2 to 4 while the force-indentation depth graphs are presented in Figures 2 to 4.

Table 2. Measurement results for the maximum force *F*max of 100 mN

	F_{max} [mN]	h_{max} [µm]	hp [µm]	h_r [µm]	HM_s [N/mm²]	H_{it} [N/mm²]	E_{it} [N/mm²]	C_{it} [%]	η_{it} [%]	HV*
1	101.52	1.4567	1.297	1.3775	2135.313	2170.154	1.76E+05	1.810	9.001	200.5
2	101.4	1.3285	1.193	1.2518	1547.631	2619.763	2.00E+05	1.243	8.517	242.1
3	101.43	1.2197	1.0531	1.1371	1888.213	3158.442	2.08E+05	2.601	9.122	291.8
4	101.4	1.4215	1.2736	1.3383	1967.441	2290.985	1.71E+05	1.710	9.581	211.7
5	101.45	1.267	1.0902	1.1867	1933.469	2907.497	2.05E+05	2.244	9.622	268.7
Aritm. mean	101.44	1.3387	1.1814	1.2583	1894.413	2629.368	1.92E+05	1.921	9.169	243.0
Std. Dev.	0.049	0.1	0.108	0.101	215.167	413.143	17109.432	0.521	0.455	38.174

Figure 2. Force – indentation depth graphs for the maximum force *F*max of 100 mN

Significant variations of measured hardness values and indentation modulus were obtained for the measurements with the maximum force *F*max of 100 mN, as presented in Table 2 and Fig. 2. The lowest calculated Vickers hardness amounts to 200.5 HV while the highest Vickers hardness amounts to 291.8 HV. The standard deviation with the value of 17109.4 N/mm^2 was determined for the measured indentation modulus E_{it}, the lowest E_{it} amounts to 171000 N/mm^2 while the highest E_{it} amounts to 208000 N/mm^2. Indentation modulus is comparable to Young modulus of elasticity of steel. The reason for variations of measured properties may be attributed to microstructural characteristics. The microstructure of the researched sample consists of ferrite and perlite with Nb precipitates in the interaction with dislocations. The size of precipitates was measured to be in the range of 4–10 nm [1-2] and cannot be observed by an optical microscope. Since the indentations are quite small they were most probably positioned in one phase regions, only ferritic or perlitic.
Indentation creep C_{it} amounts to 1.9 % with the standard deviation of 0.5 % meaning that no significant relative change of indentation depth with respect to time of force application occurred.
Elastic part of indentation work amounts to approximately 9 % with the standard deviation of 0.45 % meaning that mostly plastic deformation occurred as a consequence of the applied load.

Table 3. Measurement results for the maximum force *F*max of 200 mN

	F_{max} [mN]	h_{max} [µm]	*hp* [µm]	h_r [µm]	HM_s [N/mm^2]	H_{it} [N/mm^2]	E_{it} [N/mm^2]	C_{it} [%]	η_{it} [%]	HV*
1	208.47	2.0399	1.8808	1.9117	1696.448	2302.891	1.583e+005	3.377	8.999	212.8
2	211.15	2.1396	1.9705	2.0110	1405.255	2111.089	1.512e+005	4.077	8.464	195.6
3	211.16	1.9922	1.8312	1.8618	1877.274	2455.803	1.618e+005	3.631	9.422	226.9
4	210.55	1.8688	1.6965	1.7371	1477.437	2804.792	1.724e+005	4.092	9.157	259.2
5	211.15	2.1450	1.9649	2.0128	1657.001	2105.589	1.455e+005	4.496	9.714	194.6
Aritm. mean	210.50	2.0371	1.8688	1.9069	1622.683	2356.033	1.579e+005	3.935	9.151	217.7
Std. Dev.	1.165	0.115	0.113	0.115	186.954	290.208	10290.551	0.437	0.471	26.81

The differences of measured hardness values and indentation modulus were noted for the measurements performed with the maximum force *F*max of 200 mN as for 100 mN applied force. Still, standard deviations are smaller compared to standard deviations determined for the results obtained by 100 mN applied force. The lowest calculated Vickers hardness amounts to 194.6 HV while the highest Vickers hardness amounts to 259.2 HV. The standard deviation with the value of 10290.5 N/mm^2 was determined for the measured indentation modulus E_{it}, the lowest E_{it} amounts to 145500 N/mm^2 while the highest E_{it} amounts to 172400 N/mm^2. The hardness and indentation modulus are lower compared to hardness and indentation modulus determined by 100 mN test force indicating that the measured values are dependent of the applied load.

Indentation creep C_{it} amounts to 3.9 % with the standard deviation of 0.4 % which is higher compared to 100 mN results while the elastic part of indentation work amounts to approximately 9 % with the standard deviation of 0.45 % and corresponds to the elastic part of indentation work of 100 mN results.

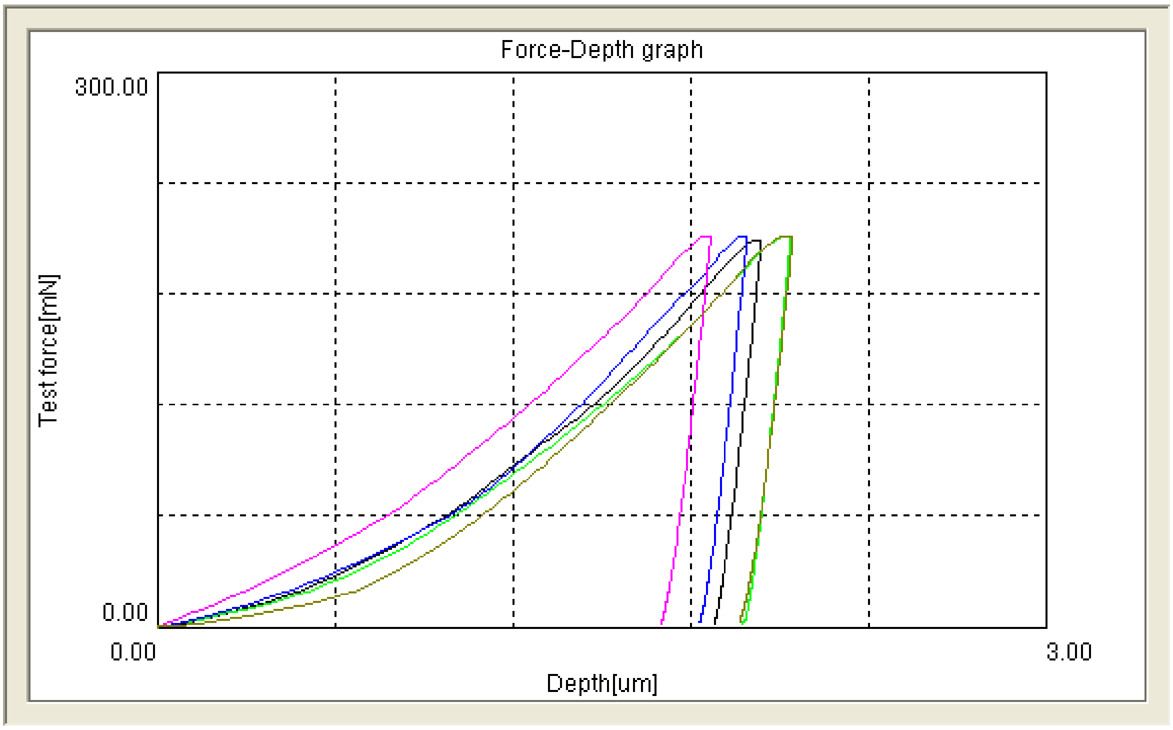
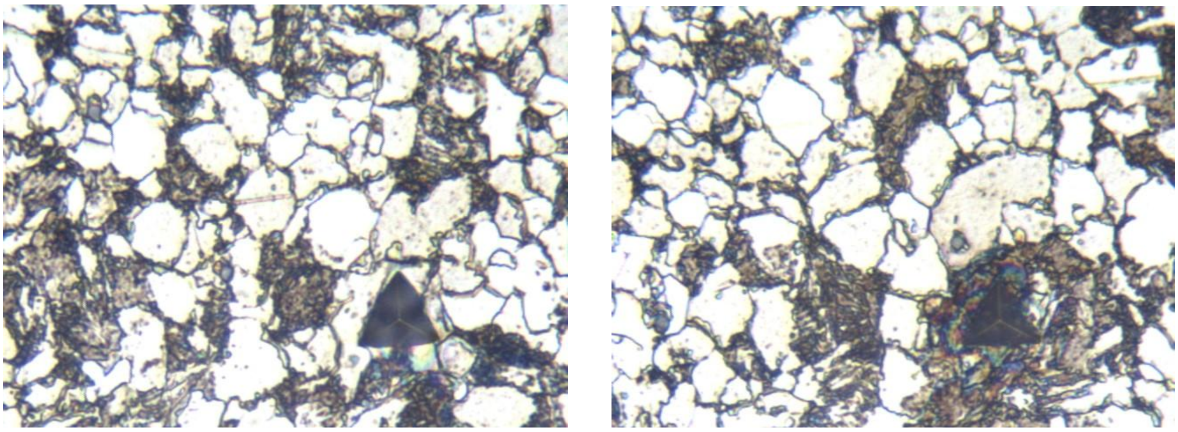


Figure 3. Force – indentation depth graphs for the maximum force *F*max of 200 mN

As already stated in the text, the variations of measured hardness and indentation modulus may be attributed to microstructural characteristics. The indentations on the etched samples surface were analysed on optical measurement system which is integral part of the machine. The microstructure with indentation is presented in Figure 4.



a) b)

Figure 4. Indentations applied by 200 mN force
a) ferite region; b) perlite region

The microstructure of the researched sample consists of a ferrite and perlite with Nb precipitates.

Indentation presented in Figure 4. a) placed in the ferrite region is smaller compared to indentation placed in the perlite region (Figure 4. b) which explains the differences in measured hardness and other properties. Differences are not desirable when determining the mechanical properties of the material in general, but it is desirable when the mechanical properties of the individual phases need to be determined, which is also one of the advantages of the instrumented indentation technique. For determination of sample's mechanical properties in general, the force of 1969 mN was applied which corresponds to measuring method HV0.2 (Table 4 and Figure 5).

Table 4. Measurement results for the maximum force *F*max of 1969 mN

	F_{max} [mN]	h_{max} [µm]	hp [µm]	h_r [µm]	HM_s [N/mm²]	H_{it} [N/mm²]	E_{it} [N/mm²]	C_{it} [%]	η_{it} [%]	HV*
1	1969.53	6.2181	5.3752	5.6733	1861.861	2435.563	1.13E+05	1.077	13.231	225.1
2	1968.69	6.3136	5.471	5.7678	1729.125	2357.0	1.10E+05	1.094	13.115	217.8
3	1968.5	6.1885	5.4082	5.6349	1682.922	2464.885	1.12E+05	1.09	13.254	227.8
4	1968.28	6.2727	5.3419	5.7291	1844.821	2388.136	1.12E+05	1.136	13.005	220.7
5	1968.27	6.5354	5.6419	6.0000	1560.165	2183.37	1.08E+05	1.065	12.339	201.7
Aritm. mean	1968.65	6.3057	5.4477	5.761	1735.779	2365.791	1.11E+05	1.092	12.989	218.6
Std. Dev.	0.52	0.137	0.119	0.143	123.961	110.161	1818.187	0.027	0.377	10.179

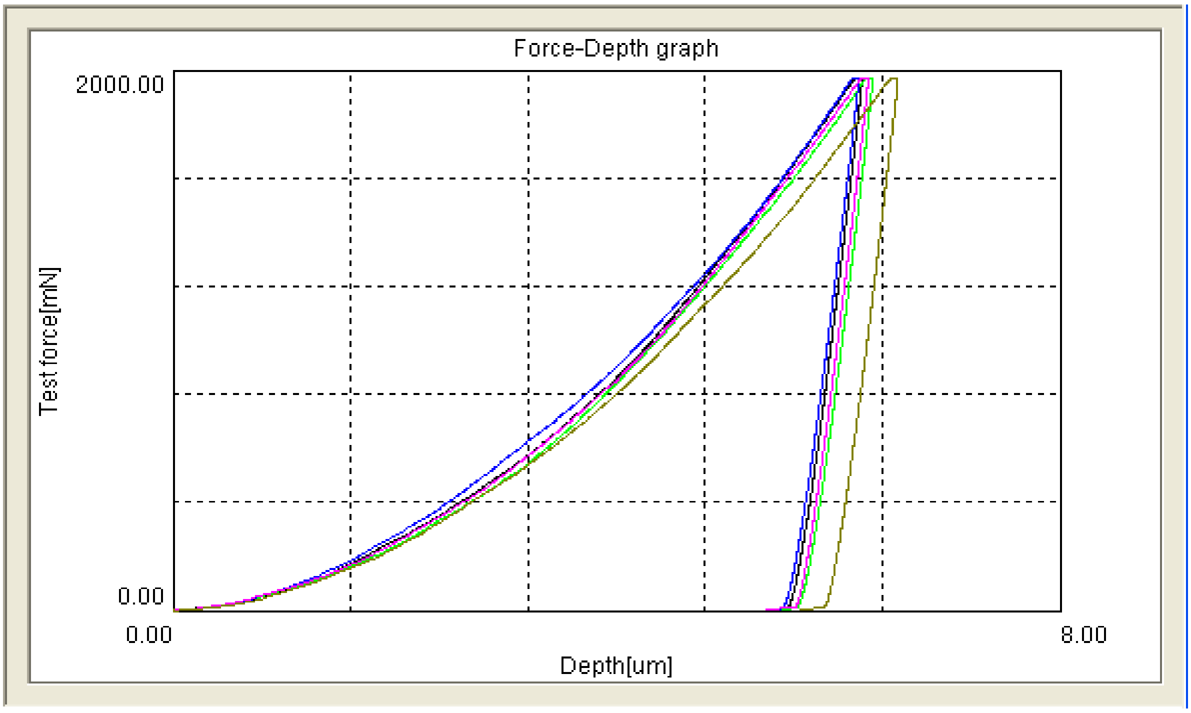


Figure 5. Force – indentation depth graphs for the maximum force Fmax of 1969 mN

The variations of measured hardness values and indentation modulus are not significant since both ferrite and perlite grains are covered by indentions as presented in Figure 6. Accordingly, standard deviations are significantly smaller compared to standard deviations determined by lower forces (100 mN and 200 mN). The lowest calculated Vickers hardness amounts to 201.7 HV while the highest Vickers hardness amounts to 225.1 HV. The standard deviation with the value of 1818.2 N/mm^2 was determined for the measured indentation modulus E_{it} whose arithmetical mean amounts to 111000 N/mm^2. The indentation modulus is much lower compared to indentation modulus determined by smaller loads and compared to Young modulus of elasticity which will be the topic of future research.

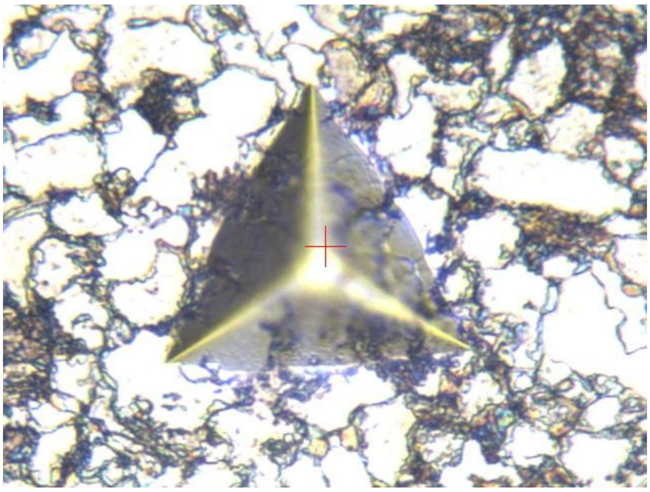


Figure 6. Indentation applied by 1969 mN force in two phase regions

CONCLUSIONS

From the conducted research can be concluded as follows:

• The maximum applied force *F*max significantly influenced the measured mechanical properties of low carbon Nb micro-alloyed steel; higher values of hardness and indentation modulus and bigger standard deviations of measuring results were obtained for smaller forces. Smaller forces are not recommended for determination of sample's mechanical properties in general.

• Deviation of the measured properties may be attributed to microstructural characteristics since the low carbon Nb micro-alloyed steel consists of ferrite and perlite phases with Nb precipitates. The load of 100 mN and lower could be applied for the determination of mechanical properties of the individual phases.

• The maximum applied force didn't significantly affect the indentation creep values and the ratio of elastic and overall work.

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