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The Micro-hardness of Heat Treated Carbon Steel

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The aim of the submitted work is to study the influence of applied loads ranging from 0.09807 N to 0.9807 N on measured values of micro-hardness of heat treated carbon steel. The influence of applied load on measured value of micro-hardness was evaluated by Meyer's index *n*, PSR method and by Analysis of Variance (ANOVA). The influence of the load on the measured value of micro-hardness is statistically significant and the relationship between applied load and micro-hardness manifests the moderate reverse ISE. As far as the relationship between measured hardness and load independent "true hardness", the best fit was obtained between HV0.05 and "true hardness" calculated using index a_2.
Keywords: carbon steel, heat treatment, mechanical properties.

1. INTRODUCTION

Indentation hardness testing is a convenient mean of investigating the mechanical properties of a small volume of materials. The principle of Vickers micro-hardness method is identical to macro-hardness test, except for considerably smaller test loads [1].

When a very low load is used, the measured hardness is usually high; with an increase in test load, the measured hardness decreases. Such a phenomenon is referred to as "normal" indentation size effect (ISE). Using a load dependent hardness in material characterization may result in some unreliable conclusions [2].

The ISE may be caused by the testing equipment. The experimental error resulting from the measurement of indentation diagonals as a result of the limitations of the resolution of the objective lens and determination of the applied load belongs to this group [1 – 3]. Another source of ISE are intrinsic properties of the tested material (work hardening during indentation, load to initiate plastic deformation, indentation elastic recovery, elastic resistance of the materials) [1, 3, 4]. The effect of machining-induced residually stressed surface (grinding, polishing) of specimen and indenter/specimen friction are also explanations of the ISE [2, 3 – 5].

In contrast to "normal" ISE, a reverse (inverse, RISE) type of ISE, where the apparent micro-hardness increases with increasing test load, is also known. It essentially takes place in materials in which plastic deformation is predominant. Reverse ISE can be explained in terms of the existence of a distorted zone near the crystal-medium interface, effects of vibration and bluntness of indenter, the applied energy loss as a result of specimen chipping around the indentation and the generation of the cracks [4].

In the literature, there are many examples, which reveal that, the "normal" ISE occurs in brittle materials while the reverse ISE has been reported mainly for materials undergoing plastic deformation [1].

The purpose of this paper is to evaluate the influence of load on the values of micro-hardness of heat treated carbon steel.

2. EXPERIMENTAL DETAILS

The investigation has been carried out on the carbon steel (0.53 % C) STN 41 1600 (conform with DIN 17 000). The schedule of used treatment of the samples:
Annealing (temperature according to Table 1/20 min) → quenching (water 20 °C) → tempering (550 °C/60 min).

Table 1. The solution treatment temperature (T), HV10, HBW 2.5/187.5 and HV (average micro-hardness of 50 indentation in "cluster")

	T (°C)	HV10	HBW	HV
1	1000	276	299	272
2	990	275	305	261
3	960	280	307	243
4	930	298	310	270
5	900	275	313	247
6	870	296	312	276
7	840	284	298	261
8	810	285	299	253
9	780	300	302	272

The surface of samples for microstructure analysis and hardness measurement was wet ground on silicon carbide papers (the sequence 220, 240… and 3000 ANSI/CAMI grit), mechanically polished with water suspension of Al_2O_3 and etched with 2 % nital. As can be seen in Fig. 1 the microstructure of all samples was tempered Sorbite with small amount of ferrite in sample 9, areas with ferrite were omitted at micro-hardness measurement.

Micro-hardness was measured with tester Hanemann, type Mod D32 fitted to microscope Neophot-32 with a magnification 480×.

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A reference block – certified reference material (CRM) with specified hardness $H_c = 242$ HV0.05 with standard uncertainty $u = 5.4$ HV0.05 was used for calibration of the tester. Because of the repeatability $r_{rel} = 4.03$ %, error of tester $E_{rel} = 1.8$ % and relative expanded uncertainty of calibration $U_{rel} = 7.7$ % the tester meets the requirements of the standard [6].

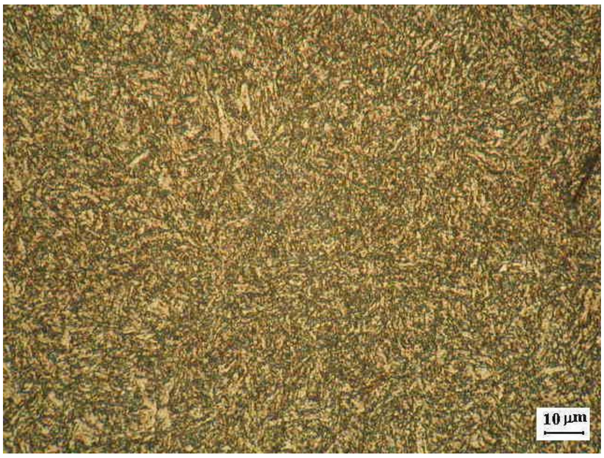

Fig. 1. The microstructure, annealing temperature 990 °C

Table 2. The micro-hardness $HV_{0.05}$, relative expanded uncertainty of HV0.05 and Meyer's index n and index A_{ln}

	HV0.05	U_{rel}	n	A_{ln}
1	286	7.7	2.646	9.66
2	283	7.8	2.791	10.38
3	273	9.0	2.667	9.79
4	290	7.1	2.595	9.65
5	243	9.2	2.471	9.04
6	290	8.1	2.664	9.95
7	274	7.6	2.604	9.64
8	256	9.2	2.602	–0.58
9	284	7.9	2.566	9.54

The micro-hardness was measured on the surface re-polished (after etching to visualize microstructure) to a mirror finish. The applied loads P were between 0.09807 N and 0.9807 N by 0.09807 N step. The load duration time was 15 seconds. The average indentation velocity of indenter in sample was 1.0 µm·s^{-1}. A researcher performed five indentations at each load. The result was a cluster of 50 indentations at one sample, the average value of micro-hardness of individual clusters HV are in Table 1. The micro-hardness HV0.05 and it's relative expanded uncertainty U_{rel} are in Table 2. The relationship between the temperature of annealing, load and micro-hardness are in Fig. 2. Grubbs' test (significance level $\alpha = 0.05$) was used for detection of statistical outliers. Their presence would indicate measurement process suffering from special disturbances and out of statistical control. The normality was determined by Freeware Process Capability Calculator software (Anderson-Darling test). The normality and the outliers were determined for files involving values of one "cluster" ($n = 50$ indentations). The values of micro-hardness of all "clusters" have other than normal distribution without occurrence of outliers. Absence of outliers suggests that the measurement process has avoided the gross errors.

The macro-hardness was finally measured with the tester HPO 250 by Vickers (HV10) and Brinell (HBW 2.5/187.5) methods. Measured values are in Table 1.

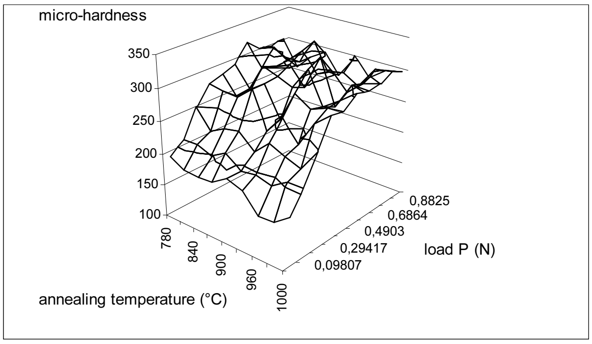

Fig. 2. The relationship between annealing temperature, load and micro-hardness

3. RESULTS

Meyer's Power Law and proportional specimen resistance (PSR) are two principal approaches to describe ISE quantitatively [3].

The simplest way to describe the ISE is Meyer's Law:

$$P = Ad^n . \tag{1}$$

The parameters n and A are determined by exponential curve fitting to indentation diagonal d (mm) versus applied load P (N) or n and A_{ln} from straight line graph of ln$d$ versus ln$P$. Meyer's index n or work hardening coefficient is the slope and coefficient A_{ln} is the y-intercept of straight line.

The index $n < 2$ for "normal" ISE, $n > 2$ for reverse ISE. When $n = 2$ the micro-hardness is independent of the load and is given by Kick's Law.

Measured values of Meyer's index n, as well as index A_{ln} are given in Table 2. All samples manifest reverse indentation size effect (RISE).

The proportional specimen resistance model of Li and Bradt (PSR) may be considered a modified form of the Hays/Kendall approach to the ISE [2]. Several authors [1 – 3, 7] have proposed that the ISE may be described by the (2):

$$P = a_1 d + a_2 d^2 . \tag{2}$$

Li and Bradt pointed out that the parameters a_1 (N·mm^{-1}) and a_2 (N·mm^{-2}) of (2) are related to the elastic and plastic properties of the material, respectively [5, 7, 8].

The parameter a_1 characterizes the load dependence of micro-hardness and describes the ISE in the PSR model. It consists of two components: the elastic resistance of the test sample and the friction resistance developed at the indenter facet/sample interface [1, 3]. The harder materials generally have higher a_1 values [8]. The parameter a_2 is directly related to load-independent micro-hardness sometimes referred to as "true hardness" H_{PSR} [2, 9].

$$H_{PSR}\, a_2 = 0.1891 \cdot a_2 . \tag{3}$$

Equation (2) may be rearranged in the form:

$$\frac{P}{d} = a_1 + a_2 d . \tag{4}$$

The parameters a_1 and a_2 of (4) may be obtained from the plots of P/d (N·mm^{-1}) against d (mm). Equation (5) can be regarded as a modified form of the PSR model.

$$P = c_0 + c_1 d + c_2 d^2 . \quad (5)$$

The parameters c_0 (N), c_1 (N·mm^{-1}) and c_2 (N·mm^{-2}) of (5) may be obtained from the quadratic regressions of P (N) against d (mm). Parameter c_0 is associated with residual surface stresses in the sample and parameters $c_1 \approx a_1$ and $c_2 \approx a_2$ are related, respectively with the elastic and plastic properties of the sample [1, 2]. The ratio c_1/c_2 is a measure of the residual stresses due to machining and polishing of the sample while c_0 denotes the residual stresses in the sample. Therefore a relationship between c_0 and c_1/c_2 is expected [1], this fact confirms Fig. 3. The numerical values of c_0, proportional to residual stresses in the sample increases with increasing of both HV and HV0.05, Fig. 4. The realionship between HV10 and c_0 is is similar, but interpretation of the relation HBW – c_0 is linked with ambiguity and requires extra work.

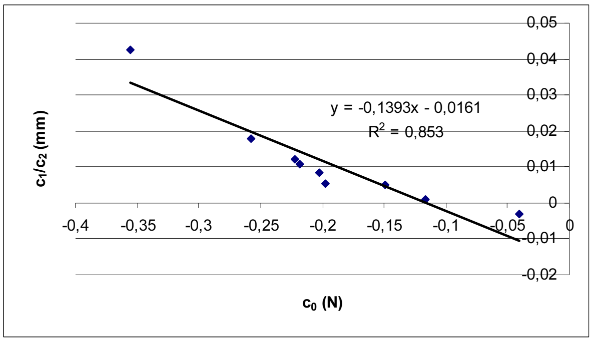

Fig. 3. The relationship between c_0 and c_1/c_2

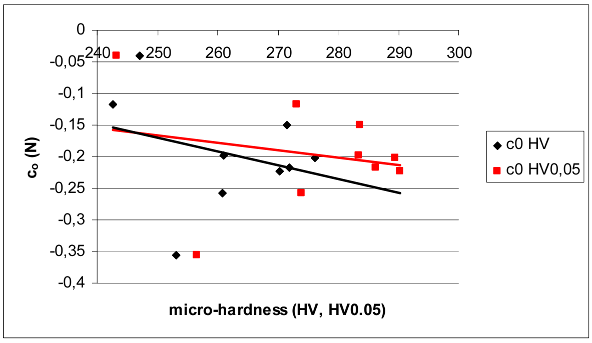

Fig. 4. The relationship between micro-hardness and c_0

Meyer's index n increases with increasing micro-hardness, as it can be seen in Fig. 5. Identical relation between measured micro-hardness and n was observed for CRMs (certified reference materials – standard blocks) made of iron or heat treated steel with micro-hardness between 194 HV0.05 and 519 HV0.05 [10], heat – treated aluminum alloy EN 6082 or technically pure metals (Al, Zn, Cu, Fe, Ni, Co, %wt. of metal > 99.5 %) [11], all with reverse indentation size effect (RISE).

The "true hardness" by analogy to a_2 can be calculated as $H_{PSR}\,c_2$ using c_2 in equation (3), therefore:

$$H_{PSR}\,c_2 = 0.1891 \cdot c_2 . \quad (6)$$

Hays and Kendall proposed existence of minimum test load W (N) necessary to initiate plastic deformation. Below it only elastic deformation occurs.

Then the load dependence of hardness is expressed:

$$P = W + A_1 d^2 , \quad (7)$$

where A_1 (N·mm^{-2}) is a constant independent of load. The values of W and A_1 may be obtained from the regressions of P (N) against d^2 (mm) [1]. The "true hardness" by analogy to a_2 can be calculated as $H_{PSR}\,A_1$ using A_1 in equation (3).

$$H_{PSR}\,A_1 = 0.1891 \cdot A_1 . \quad (8)$$

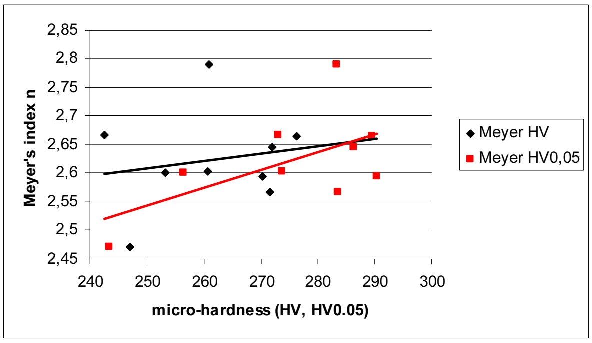

Fig. 5. The relationship between micro-hardness and Meyer's index n

The relationships between "true hardness" calculated with the aid of the indices a_2, c_2 and A_1 and measured macro- and micro-hardness can be seen in Figs. 6 and 7 as well as in Table 3.

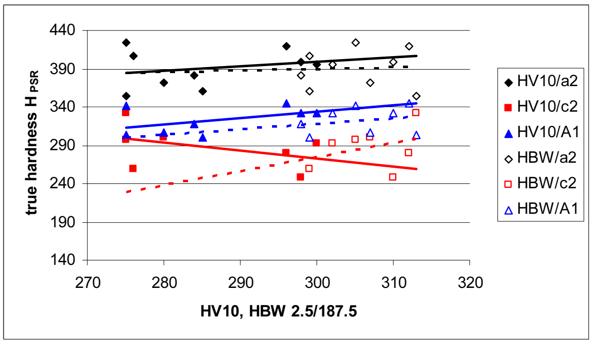

Fig. 6. The relationship between macro-hardness and "true hardness"

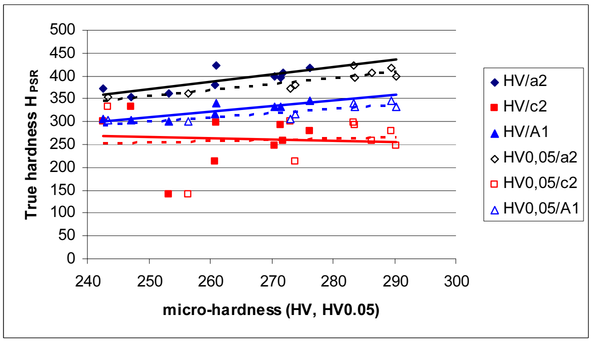

Fig. 7. The relationship between micro-hardness and "true hardness"

As far as the realtionship between HV10 and "true hardness", the best results were obtained if index c_2 was used for calculation. Using paired t-test comparing the means of two groups, by conventional criteria, this difference is considered to be not statistically significant ($p = 0.3045$), but the correlation is small. The index A_1 is the best for HBW. The index c_2 appears as uncertain for calculation the relationship between micro-hardness and "true hardness". The best fit was found between HV0.05 and "true hardness" calculated using index a_2:

$$H_{\mathrm{PSR}} a_2 = 1.3361 \cdot \mathrm{HV0.05} + 21.965\,. \qquad (9)$$

Table 3. The value of the coefficient of correlation r^2 between measured macro- or micro-hardness and "true hardness"

	$H_{\mathrm{PSR}}\,a_2$	$H_{\mathrm{PSR}}\,c_2$	$H_{\mathrm{PSR}}\,A_1$
HV10	0.0603	0.1887	0.0366
HBW 2.5/187.5	0.0019	0.1151	0.4983
HV	0.5961	0.0022	0.5222
HV0.05	0.7639	0.1115	0.5396

According to two way analysis of variance (ANOVA, significance level $\alpha = 0.05$) without replication the load ($p = 3.11\text{E-}40$) and the temperature of the solution treatment ($p = 2.88\text{E-}14$) both have statistically significant influence on the measured values of the micro-hardness HV.

4. CONCLUSIONS

1. The influence of the load on the measured value of micro-hardness is statistically significant.
2. The relationship between applied load and micro-hardness manifests reverse ISE for all annealing temperatures. This relationship is typical for materials in which plastic deformation is predominant.
3. The reverse character of ISE increases with increasing of hardness.
4. The best fit was obtained between HV0.05 and "true hardness" calculated using index a_2.

Acknowledgments

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